



Trinity College Dublin
Coláiste na Tríonóide, Baile Átha Cliath
The University of Dublin

VALIDATION OF NUMERICAL METHODS IN
COLD SPRAY BY DIRECT
NOZZLE-INTERNAL MEASUREMENTS OF
NON-DILUTE PARTICLE MOTION

MORTEN-CHRISTIAN LOUIS MEYER

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Doctor of Philosophy

Declaration

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Abstract

This work presents the research conducted during the development of a new optical particle velocity measurement in the field of Cold Spray. Cold Spray is a process for surface coating formation and additive manufacturing, in which powder particles are accelerated by an expanding process gas in a converging-diverging supersonic nozzle and deposited via high-speed impact onto a substrate. Temperatures below the melting point are characteristic for this process, which leads to unique benefits, such as low oxide contents and residual stresses, as well as sprayability of progressive material combinations and temperature-sensitive powders. The core feature of this process is the gas-particle nozzle and jet flow, defining the required high velocities upon impact. Although some aspects can be simulated reasonably well using computational models, the general validity of such is case-dependent and coverage of advanced features, such as the particle loading effect, is not yet established. As a result, application challenges like nozzle clogging and the prediction of the deposition efficiency cannot be assessed with these methods. In order to overcome this limitation and to provide means for validation of a new generation of computational models, a new measurement technique is developed in this study.

The approach combines the Cold Spray process with a Particle Tracking Velocimetry system and introduces the design of a transparent quartz glass nozzle for internal particle motion measurements. Particle behaviour during powder injection, nozzle-immanent distribution, acceleration as well as particle dispersion within the jet were observed at various pressures and particle loadings. The injector position was varied to study the influence of the particle injection plume and materials were altered to identify differences between distinct Stokes number ranges. The character of the particle phase motion was analysed and particle-particle interactions found to play an important role. Firstly, acceleration of particles in the nozzle were experimentally quantified and the statistical distributions for

particle speed and direction evaluated, highlighting the importance of randomised motion. This information was used to advance the understanding of nozzle clogging and particle heating residence time.

Commercially available software (*Ansys FLUENTv16.0*) was used to construct a computational model with advanced methods, including full phase coupling, particle collisions and a tailored particle phase boundary condition. The value of the presented experimental set-up for advanced model validation was demonstrated by comparing the measurements against the simulation results. Limits of such computational models were identified in this respect: it is necessary to employ an advanced, fully coupled model to obtain adequate velocity predictions, while dispersion characteristics remain challenging also for detailed models. This results in very material-dependent opportunities for computational clogging and heating time analysis. The validation was used to identify ways to manipulate the models for better representation of the nozzle-internal flow physics.

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Meinem Vater

*“So ungefähr stell ich mir die
Quintessenz von Dialektik der
Aufklärung vor.” NMZS*

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Nomenclature

Roman symbols

A	Cross-sectional area [m ²]
b	Collision random parameter [-]
c	Speed of sound [m/s]
C_D	Particle drag coefficient [-]
c_p	Specific heat [J/(kgK)]
d_{inj}	Diameter [m]
E	Specific energy [J/kg]
$\vec{F}$	External body forces coupling term [N/m ³]
F	Particle-gas volume ratio [-]
$\vec{g}$	Gravitational acceleration vector [m/s ²]
$Gr_{F,n}$	Normalised velocity gradient with volume ratio [-]
$Gr_{Z,n}$	Normalised velocity gradient with mass loading [-]
h	Height [m]
h_j	Specific enthalpy [J/kg]
h_q	Heat transfer coefficient [W/(m ² K)]
I	Unit identity tensor
$\vec{J}$	Diffusion flux
k	Specific turbulent kinetic energy [J/kg]
k_{eff}	Effective conductivity [W/(mK)]

l	Length [m]
$l_{influence}$	Length of influence of wall collision in converging section [m]
l_{turb}	Integral turbulent length scale [m]
Ma	Mach number [-]
$\dot{m}$	Mass flow rate [kg/s]
$\bar{n}$	Average number of particle collisions [-]
N_{coll}/N	Percentage of particles with positive wall normal velocity [%]
Nu	Nusselt number [-]
p	Pressure [Pa]
Pr	Prandtl number [-]
R	Specific gas constant [J/(kgK)]
r	Radius [m] (geometry), Main line/feeder flow ratio [-] (gas variables)
Re	Reynolds number [-]
S_h	External heat source coupling term [J/(m ³ s)]
Sk	Stokes number [-]
T	Local gas temperature [K]
u_i	Velocity component in i-th dimension [m/s]
V	Volume of mesh element [m ³]
$\vec{v}$	Velocity vector [m/s]
v	Velocity magnitude [m/s]
w	Nozzle width in z-direction [m]
x	Longitudinal location [m]
y^+	Dimensionless wall adjacent mesh cell size [-]
Z	Particle mass loading [-]

Greek symbols

α	Trajectory angle [°]
α_p	Particle volume fraction [-]
γ	Gas heat capacity ratio [-]
$\delta_{influence}$	Width of influence of wall collision in diverging section [m]
ε	Turbulent dissipation rate [J/(kgs)]
ζ	Turbulence random parameter [-]

η	Coating quality parameter [-]
μ	Dynamic molecular viscosity of gas phase [Pas]
Π_{mom}	Momentum coupling parameter [-]
ρ	Density [kg/m ³]
σ_u	Yield stress [N/m ²]
$\overline{\overline{\tau}}_{eff}$	Effective deformation tensor [J/m ³]
τ	Characteristic time scale [s]
Φ_s	Sphericity of particle [-]

Subscripts

0	Gas stagnation value
1, 2	Collision of particle parcels 1 and 2
*	Critical (sonic) condition
c	Nozzle converging section (geometry), continuous phase (gas variables)
c, e	Centreline at nozzle exit
cr	Critical impact condition
d	Nozzle diverging section
e	Nozzle exit
i	Nozzle inlet
inj	Injector needle
m	Melting point
n, w	Normal to nozzle wall
p	Particle
p, i	Particle impact condition
r	Relaxation of particle
tr	Nozzle throat contour
t, w	Tangential to nozzle wall
V	Velocity response of particle
w, i	Impact onto nozzle wall

Abbreviations

1D,2D,3D	One-, two-, three-dimensional
BC	Boundary condition

CFD	Cold Spray
CS	Cold Spray
DE	Deposition Efficiency
DPM	Discrete Phase Model
DPV	Doppler Picture Velocimetry
DRW	Discrete Random Walk model
ER	Expansion Ratio
FFT	Fast Fourier Transform
FoV	Field of View
HVOF	High-Velocity-Oxy-Fuel
L2F	Laser-two-focus
LES	Large Eddy Simulation
MoC	Methods of Characteristics
NSE	Navier-Stokes equations
NZ1	Cold Spray Nozzle 1
NZTR	Transparent Cold Spray Nozzle
pdf	Probability density function
PIV	Particle Image Velocimetry
PTP	Particle Temperature Parameter
PTU	Programmable Timing Unit
PTV	Particle Tracking Velocimetry
PVP	Particle Velocity Parameter
RANS	Reynolds-Averaged Navier-Stokes equations
rms	Root mean square
RSM	Reynolds Stress Model
SEM	Scanning Electron Microscope
SoD	Stand-off distance
UDF	User-defined function
VMD	Volumetric Mean Diameter

Chapter 1

Introduction

There is something fascinating about flows. Humans are amazed by creeks and rivers, waterfalls, and the waves of oceans. By nature, our intuition is tightly connected to fluids - gases and liquids. Yet, it is astonishing how mysteriously this un-holdable matter behaves to us, after thousands of years of study. From the antique times of Archimedes and Julius Frontinus, through the Islamicate physicists in the middle ages and the disciples of the renaissance, by Newton, Bernoulli and Euler, to the big names of Helmholtz, Kelvin, Stokes, Rayleigh, Prandtl and countless more: studies on fluid mechanics were never suspended.

It is true, that one grows along with one's tasks. Maybe our fascination for fluid flows contributed to the exponential growth of complexity and detail of studies. Along with it, a vast amount of applications were born from technological advancements. It appears that no relevant process nowadays functions without fluid mechanics. In fact, flowing media are often core elements in modern automotive, transport and aviation sectors, in construction, agriculture and food industry, even in biological and biotechnological sciences, as well as health and medicine.

Technology development in these domains is impossible without manufacturing, the process of converting raw materials into required shapes and combining their properties in such a way, that the part can fulfil its function in the bigger picture. Through the innovation process, new advanced ways of manufacturing are developed, that increasingly combine the achievements in fluid dynamics with the functionality of manufacturing. Man-

ufacturing is the means by which technological benefits for society are realised. In fact, economic growth by virtue of improved industrial infrastructure, as well as research and development of technical solutions, are taken as direct indicators for human development by the United Nations (UN). On the way to a more peaceful, equal, and prospering planet, the UN set a series of critically important goals, at least five of which are directly connected to the advancement of manufacturing [1].



Figure 1.1: Human development goals connected to manufacturing

- **Affordable, reliable, sustainable and modern energy**

Energy efficiency is one of the main drivers in industrial innovation. Lower energy consumption reduces manufacturing cost and requires fewer resources.

- **Sustained and inclusive economic growth & productive employment**

The industrial sector is a main pillar for employment. A prospering economy ensures value chains from which local societies benefit. Manufacturing means are therefore constantly improved by research and industry, in order to open new markets and amend current production.

- **Resilient infrastructure, promoted industrialisation & fostered innovation**

The previous goal of economic prosperity relies on a network of regional and international infrastructure, both for financial support and technological competitiveness.

In particular research projects provide the required momentum of innovative production solutions.

- **Sustainable consumption and production patterns**

Future industry is required to make efficient use not only of energy, but also of materials and human resources. Smart, automated, and integrated production lines aim to reduce material waste, while more effective manufacturing techniques boost the sustainability of industry by more direct approaches.

- **Action against climate change and its impacts**

All the above goals eventually lead to the notion of saving the ecological environment that we live in. Combat against climate change is the most fundamental responsibility to fulfil in developing more efficient and sustainable production methods and the best motivation for innovation and research.

International research institutions like the European Research Council (ERC) and national funding organisations like Science Foundation Ireland (SFI) layout their support strategies and agendas accordingly. Their mission statements promote directing funding into promising new fields with high potential and far-reaching impact (ERC [2]) and in particular to support the best engineering research to advance Ireland's society and economy (SFI [3]). In face of this, advanced manufacturing techniques are one focal point of research and innovation, and they tightly tie in with the human development goals of the UN, potentially more than other branches of engineering: the manufacturing sector represents the realisation of engineering concepts, is therefore the largest contributor to non-financial business economy value added and makes up a significant fraction (10%) of all enterprises in the European Union (EU), which is a total of 2.1 million companies with 29.9 million employees (values for 2014) [4]. As a consequence, research and innovation in this vast field has significant impact on our society and carries vast potential for contributions to the development goals. In this context, Cold Spray is an outstanding example of such innovative manufacturing processes, representing Additive Manufacturing (AM) in general [5].

In an additive process, material is added to components rather than subtracted like in traditional techniques. In combination with computerisation and automation, the EU

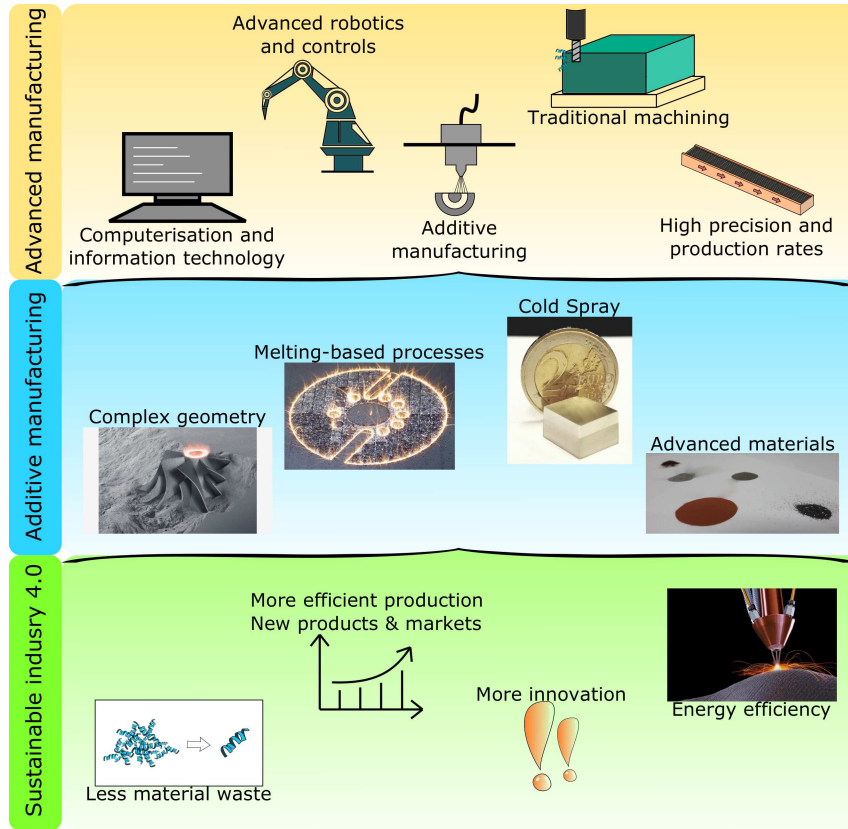


Figure 1.2: The potential of Additive Manufacturing among advanced manufacturing techniques

factually identified AM processes as the family of technologies with the “highest growth potential in industry”, providing “unprecedented opportunities for businesses” [6]. In the long term, production can be done locally through 3D-printing, rather than being required to be relocated to low-cost countries. This reduces logistical effort and the respective pollution and energy consumption. By being a more direct approach to building components, AM minimises the waste of raw materials and lowers the energy use during manufacturing. The impact on society even goes beyond these aspects, since vast customisation opportunities and new markets will give rise to a “maker-culture”, in which the character of the majority of jobs will shift from repetitive to innovative work, and the phase of these deep changes has already started.

What is offered by melting-based technologies like Selective Laser Melting (SLM) to date, *e.g.* producing complex geometries, is going to be complemented by Cold Spray (CS), a relatively recently developed method among a group of “thermal” spray processes. Due to a set of very unique advantages, it enables that previously incompatible materials

can be combined within parts, unweldable components joined directly without build-up of oxides, and material properties adjusted continuously throughout a product. Now primarily used to apply coatings to engineering components, examples of its benefits are corrosion and wear resistance, but its potentials are more numerous: customised and optimised geometries, advanced material and composite parts, optimal manufacturing times and resources, as well as making assemblies leaner and more reliable [7].

Cold Spray, more than any other additive manufacturing and thermal spray process, relies on the fluid flow of a gas-particle mixture. It achieves the deposition of material through high speed impacts of particles, which bond with a substrate. For this, high velocities are required, realised by a supersonic gas-particle jet that impinges on the component [8]. This gas-solid, hence two-phase, accelerating nozzle and jet flow is the heart of the process. Addressing this importance, one main focus of studies in Cold Spray is the dynamics of the nozzle flow. Over the last twenty years, the particle velocity and gas structures in the jet were measured, in order to gather understanding of the working principles, to design better components, and to find optimal process parameters. Computational techniques were introduced in this process, not only because they help to save time and resources, but also because some aspects of the flow are hidden in the nozzle and cannot be measured.

Models used for investigative and design purposes have limited experimental validity and are difficult to improve. Moreover, many studies focus their models on the capabilities to predict particle impact velocities for simplified conditions, but drop adequate representation of spray dispersion and particle impact statistics. If the critical hurdle to assess the validity of numerical tools in entirely new ways could be overcome, they would reach a level of accuracy currently not possible. Opening doors to a new level of understanding of the Cold Spray flow process advances this part of a high-potential engineering field towards a more innovative, efficient and sustainable future of industry.

1.1 Context of aims and objectives

The growth of the Cold Spray field in the last decades is owed to its immense usefulness. With vast innovation opportunities yet to be discovered, many searched for ways to com-

bine incompatible materials, improve strengths of deposits, and design nozzles with very specific performances. It appears natural that the potential has lead to strong and fast application-oriented progress. As the literature (chapter 2) shows in detail, new difficulties were encountered hand in hand with this process: how can difficult-to-reach locations be accessed on components, how can detrimental nozzle wear be reduced, how could the particle beam be controlled, or how can the efficiency of the deposition be estimated? These aspects emerge from the applications, but are deeply rooted in the physics of interactions of gas and particles. Computational simulations were already used to design short nozzles to ease access difficulties. However, the models were not ready to explain the performances of different nozzle shapes and could not assess the impact conditions for the whole powder rather than single particles, which impeded on the efficiency prediction. The calculation of particle dispersion in the jet, or the explanation of why nozzles are getting clogged by the powder under various conditions, were could not be investigated either. This narrative describes the situation at the beginning of the present work: how can a computational model be amended to assess these advanced aspects?

For such improvements to be achievable, it is required to gain better understanding of how the particles behave inside the nozzle, rather than after they exit from it. This is important, because most critical mechanisms, the acceleration and the dispersion, hence all gas-particle phase-interactions, occur within the nozzle. The conflict arises when researchers rely on those computational models that, based on their assumptions, do not cover the responsible mechanisms that take place here. To avoid this, two routes can be taken. An empirical/application-oriented approach offers the advantage that already established measurements, in the jet or based on deposits, can be conducted with no need to measure nozzle-internal mechanisms. Such established observations can be correlated to some optimum through large numbers of conditions, while it is not generally possible to assure the causal reasons for the observed behaviour. The latter may however be a prioritised aim, because the additional physical insight entails more general solutions that can be applied to other systems, or leads to unforeseen possibilities to exploit the mechanisms. In this case, an in-depth investigation through a novel measurement that can identify the mechanisms in question is necessary. However, this is possible only for a selected set of circumstances and one main challenge regarding model amendment results from this:

since, naturally, the region inside the nozzle is not generally accessible to measurements, obtaining direct data about the particle movement seems prohibitive at first. If this could be realised nonetheless, the benefit would be significant, as derived models would be based on the actual observed particle behaviour.

This work takes the challenging second path and aims to measure the particle behaviour inside a Cold Spray nozzle. It aims to use the gathered new insights to rethink the computational modelling and include the identified aspects. This is done in order to provide means to computationally assess the advanced challenges that are faced in CS applications on the basis of physically validated methods.

With this in mind, the aim of this research project is to design a new experiment for nozzle-internal particle velocity measurements and use the novel experimental analysis to demonstrate the validity of an advanced computational model. In this respect, the following objectives are defined for the research project:

1. The phase coupling in Cold Spray is not fully clarified. Extend current knowledge on the importance of gas-particle momentum exchange, on the particle dispersion, and on the influence of mass loading for nozzle-external observations. Identify possible drivers originating from inside the nozzle.
2. In order to overcome the discovered confinement in model quality, investigate the conditions during particle acceleration directly. To this end, develop an experimental set-up for nozzle-internal measurements.
3. Measure and analyse particle behaviour within the nozzle and suggest physical mechanisms that explain the observations that following influences induce.
4. Construct a computational model that covers the observed phenomena and demonstrate new validation opportunities based on the novel experimental data.

To summarise, since the understanding derived from computational studies and estimates of particle properties greatly depends on the validity of the used computational models, the notion of direct measurements of the particle acceleration and dispersion carries high potential. More detailed knowledge of the fully coupled gas and particle phases within the Cold Spray nozzle would be beneficial for facing modern design and operation challenges, like efficiency and nozzle clogging. It entails new validation methods that

result in higher model quality and more predictable quantities, in the long term, even the statistical distribution of powder impact conditions and the onset of nozzle clogging. The pursuit of these aims and objectives is reported during the forthcoming chapters, as outlined in the following synopsis.

1.2 Thesis synopsis

Chapter 2 - Background

This chapter presents the general concepts of the Cold Spray process and differentiates it from other thermal spray technologies. Besides the introduction of the process and its parameters, an overview of the essential aspects of flow theory is provided, with emphasis on the particle motion. Because this study aims to provide means of better model validation and discuss the limits of current models, the core part of the review focuses on the most common computational modelling approaches. The experimental methods for flow analysis and particle velocity observations are subsequently summarised. The chapter foregrounds unanswered questions within the field that give rise to the area of investigation of the present work.

Chapter 3 - Experimental methodology

The novel methodology introduced in this work is experimental in nature and a detailed discussion of the equipment and the procedure can be followed in this chapter. Besides the presentation of the Cold Spray system, the flow control devices and the operation parameters, the component design is strongly in focus. Most importantly, the transparent Cold Spray nozzle development is reported. Moreover, the measurement technique is described and the accuracy of the measured data is assessed in this context.

Chapter 4 - Experimental results: Indirect jet flow characterisation

Until now, the effect of significant coupling between the particle and gas phase, and with it fluid dynamic considerations beyond a drag force, were only reported through implicit

studies with ambiguous results. The implicit character results from the observation of particle velocities in the jet, *i.e.* downstream of the nozzle, in which the acceleration takes place. In order to clarify the necessity of deeper two-phase analysis, this chapter extends this implicit approach of measurements in the jet by adding changing feedstock materials and operating pressures to the spectrum of results. In order to advance the understanding of the particle feed rate as a process parameter for applications nozzles, this chapter reports a structured investigation of it in a Cold Spray nozzle from previous work. This aims to identify possible driving mechanisms of the phase interactions and to make a connection between the two-phase character of the flow and the crucial process quantities of velocity and dispersion.

Chapter 5 - Experimental results: Direct nozzle-internal measurements

The actual mechanisms that cause the particle behaviour from the previous chapter are still unknown, and experiments in the jet are insufficient for clarification. Subsequent to such indirect jet-measurements, velocimetry within the transparent nozzle is expected to be the new required step. Therefore, the basic concept of this chapter is to analyse the statistical behaviour of particles within the nozzle under different injection and operation configurations. This study is the very first report of a quantitative measurement of a nozzle-internal particulate flow and introduces a variety of novel findings about injection velocity, nozzle internal dispersion, particle-particle interactions and the acceleration of particles along the nozzle. In addition, those observations are connected to the velocity and dispersion of particles within the jet.

Chapter 6 - Computational results: Analysis of a new computational model

This chapter leads the argumentation back to the beginning of the study and hence to its main focus. The experimental analysis presented in the previous chapter is applied to a computational model with the widely employed commercial software package *Ansys FLUENT v16.0*. Based on the nozzle-internal measurements, this final chapter provides

validation of the velocity profiles. It discusses and explains the limits of the predictive capabilities with respect to particle dispersion and suggest possible improvements. After introducing the computational model, the chapter attempts to answer three main questions that arise about Cold Spray computations: Can the velocity level be predicted, is the dispersion captured, and what does this mean for the assessment of residence times and wall-interactions - two very important current operational challenges for researchers working on Cold Spray applications.

Chapter 7 - Conclusions and future work

Finally, this chapter concludes the main aspects and combines them in a bigger picture, such that the overall outcomes of the research points the field in a new direction.

Chapter 2

Background

2.1 Introduction

This chapter aims to inform the reader in a concise manner about the general concepts involved in the Cold Spray (CS) process and the experimental and numerical analysis methods of gas and particle flow applied in the past. Within this context, it is the focus of this chapter to foreground unanswered questions that give rise to the area of investigation of the present work.

2.2 Cold Gas Dynamic Spray

This section aims - with no claim to completeness - to rank CS within the group of conventional thermal spray processes, to provide an overview over the most important features and parameters of CS, and to outline some important coating properties.

2.2.1 Early modern Cold Spray

The process of CS was initially developed by a group of researchers lead by Dr. Anatolii Papyrin during the 1980's at the *Institute of Theoretical and Applied Mechanics of the Russian Academy of Sciences* in Novosibirsk [8]. During experiments on supersonic flows around cylindrical bodies, the scientists discovered that their tracer particles deposited onto the wind-tunnel model. The phenomenon appeared to be connected to the cold deformation of the particles, giving rise to follow-on studies, and a deposition procedure

was ready for patent registration in the 1990's [9–11]. Over the following decades, the technology was further developed by a rapidly growing number of research groups, at first located in Russia and the United States, subsequently also in several European and Asian countries, as well as Australia [12].

As CS is a relatively recently established method, it is considered the “new-comer” among other thermal spray technologies. This is a group of coating manufacturing processes, of which some examples should be given, categorising them according to the source for feedstock heating: electrical or chemical.

The first category is found in processes such as arc spraying and plasma spraying. In arc spray, the spray material forms wire electrodes that are molten by applying an electric arc and accelerated towards the substrate by a compressed air stream. It stands for high deposition rates and high energy efficiency. Limitations are the conductivity of the wires and the moderate adhesion strength of the coatings [13]. Plasma spray utilises an electric arc to generate an ionised plasma as a source of heat for a driving gas that passes through a nozzle. The feedstock is injected into this hot gas jet in form of powder, molten and accelerated. The main advantage comes from the extreme temperature level (up to 8000°): high-melting materials, in particular ceramics, can be deposited. The downside is a complex, less efficient system [14].

The second category can be represented by combustion processes, such as flame spray or High-Velocity-Oxy-Fuel (HVOF). Flame spray uses the heat from the combustion of a fuel gas to heat the wire or powder feedstock material [14]. In the HVOF process [15], a fuel is similarly combusted by oxygen to heat the feedstock material, but at a higher flow velocity. This enables suppression of extreme thermal degradation of the spray particle by lowering gas temperatures [16].

All these processes are, to different extents, operated at temperatures in excess of the melting temperatures of the coating material. This is the aspect in which Cold Spray is a very distinct process: in CS, melting temperatures are generally not crossed and the energy required to enable bonding between coating particles and substrate material is provided in a kinetic rather than thermal way - the lower temperature levels are made up for by much higher particle impact velocities. Considering that coatings can be applied in a strictly solid-state manner, a completely new field of applications can be framed for

surface engineering and additive manufacturing. Low thermal input goes hand in hand with a massive reduction of detrimental thermal effects, such as oxidation, evaporation, high residual stresses or crystallisation [17]. This provides a possibility to coat oxidation-sensitive materials and to combine materials with very different melting temperatures that are in no other way compatible [8, 18]. These core advantages aside, the process can still deliver coatings with very low porosity and high adhesive strength [19].

2.2.2 Cold Spray process

Despite possible more complex installations, the basic principle of Cold Spray can be explained with a minimum set of components that can be seen in the arrangement in Figure 2.1.

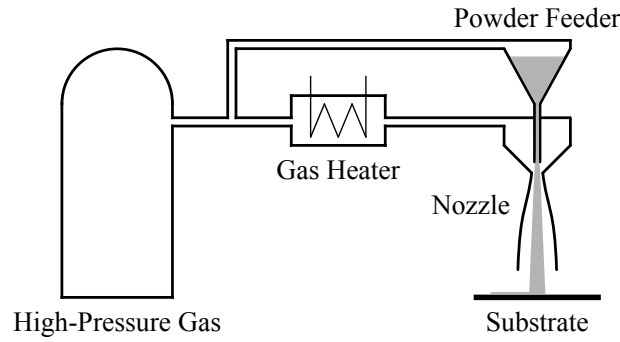


Figure 2.1: Basic cold spray process

High pressure gas (Helium or Nitrogen) is fed from a supply into a main line and a feeder line. The main line gas (process gas) is heated to a desired temperature and subsequently lead into a converging-diverging De-Laval nozzle, in which the gas expands and accelerates, forming a low-velocity/high-temperature and a high-velocity/low-temperature part. Simultaneously, the unheated feeder line gas (carrier gas) transports the feedstock powder to the nozzle and injects either into the converging or the diverging section. A drag force of the fast moving process gas acts on the individual particles and accelerates them to velocities in the range of $300 - 1200\text{ m/s}$ [20]. This two-phase flow system is above all other aspects responsible for the coating formation. Upon impact of the fast-moving particle onto the substrate surface, it undergoes high plastic deformation and reaches a mechanical bond. The particles must achieve velocities above a certain material-dependent value, the critical velocity, in order to realize bonding [17]. For this, the following most

important process parameters need to be set carefully for each application.

Process gas

Given a specific nozzle design, the supersonic exit velocity of the gas is a function of the exit Mach number. Consequently, the velocity can be raised by increasing the speed of sound. Because of its lowest molar mass and highest heat capacity ratio, Gilmore *e.g.* [21] documented that helium achieved particle velocities twice as high as nitrogen at the same temperature level. More detailed studies analysed the microscopic changes in the coating with gas type [22], or investigated gas mixtures using Computational Fluid Dynamics (CFD) [23]. Because of the high cost of helium as a process gas, it is economically desirable to save expenses utilising nitrogen when possible. In order to make up for the lower sound velocity, nitrogen is more commonly pre-heated [24]. Another option is to use a gas recycling system for helium [25].

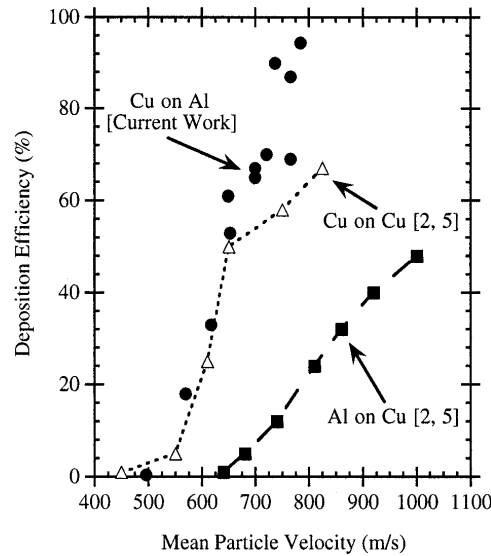


Figure 2.2: Measurements of the deposition efficiency of Copper and Aluminium indicate the critical velocities for $19\mu m$ -particles and varying temperatures and pressures [21].

Deposition efficiency

By the late 1990s, the increasing effect of the particle velocity on the deposition efficiency (DE) was quantified and used for nozzle optimisation [26]. DE quantifies the capability of the process to build a coating from a specific amount of feedstock. It is the proportion of

deposited particle mass over all injected particle mass [20]. A steep increase of measured DE with increasing particle velocity is characteristic just above the critical threshold [21], as shown in Figure 2.2. The average particle velocity, at which the DE is non-zero, corresponds to the critical velocity.

It is an important general principle of CS that for given nozzles, the goal is the increase of the particle velocity above this lower threshold to the so-called deposition window. Particles need to impact below a second, upper threshold, the erosion velocity, where the impact becomes overly strong. Too high kinetic energy may result in the destruction of the single particle upon its impact, or in the bulk deformation and erosion of whole layers of the substrate under formation (see section 2.2.3). The range of velocities in between those values forms the deposition window [27], visualised in Figure 2.3. Very brittle materials generally cannot be deposited (without further adaptations). Apart from the material selection, the window of deposition changes implicitly with other parameters, such as temperature, by changing both the critical velocity and the impact velocity.

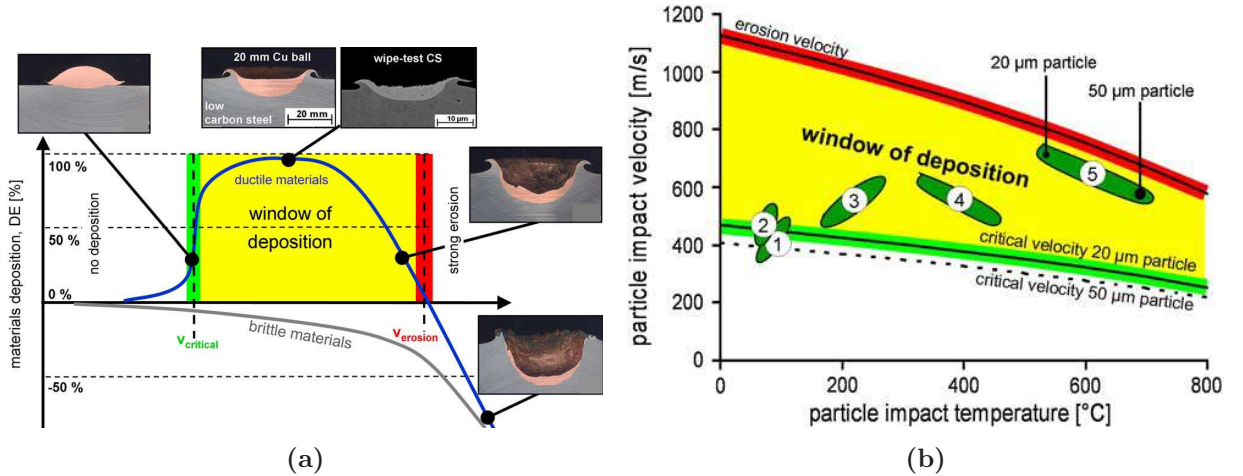


Figure 2.3: The deposition efficiency depends on the impact velocity and the specific critical and erosion velocity. (a) Change of impact structure with impact velocity, (b) implicit effect of temperature, from [27].

With a seemingly strong sensitivity to a wide variety of parameters, the critical velocity was required to be quantified more precisely. Indeed, using both experiments and calculations, the critical velocity was first determined for a small selection of materials (copper and aluminium) [28], yielding a first empirical formulation. This fitting expression based on yield strength, material density and melting point primarily indicated that

particles need to exceed this threshold in order to overcome their material strength and hence succeed in deforming. Because this was considered a material-dependent process, here, the size of particles was not considered in the expression.

$$v_{crit} = 667 - 0.014\rho + 0.08(T_m - T_R) - 10^{-7}\sigma_u - 0.4(T_m - T_R) \quad (2.1)$$

with T_R as the reference temperature of 293K, the melting point T_m , the initial particle temperature T_i , the particle density ρ_p and the yield strength σ_u . While the critical velocity primarily depends on the material strength as covered by this equation, the size of impacting particles may nonetheless have secondary influences. The different surface-to-volume-ratios of smaller sized particles may entail a detrimental higher amount of adsorbents and surface oxides. Another, more important, origin may be the change in length scales of heat conduction and size dependent differences in strain rate hardening. Consequently, Schmidt *e.g.* [29] later further improved the expression for various materials to:

$$v_{cr} = \sqrt{\frac{4F_1\sigma_u}{\rho_p} \left(1 - \frac{T_i - T_R}{T_m - T_R}\right) + F_2c_p(T_m - T_i)} \quad (2.2)$$

Hence, the critical velocity is reduced by increasing initial particle temperature and particle density, or by lowering the yield strength and melting point. The substantial difference to equation 2.1 is that it implicitly includes the particle size based on the equation calibration: the first term in the sum under the square root represents the interplay between material strength and dynamic load, combining the tensile strength with the Johnson-Cook equation for thermal softening. This represents the mechanical impact balance and is calibrated for a specific particle size through fitting coefficient F_1 . The second term under the square root represents the energy balance of impact, which is added to the equation to account for the thermal dissipation through provided kinetic energy. This is also calibrated through correlation factor F_2 . It is worth noting that the authors additionally quantified a minimal (threshold) particle size based on the thermal length scale, below which no localised heating can lead to bonding.

More recently, Assadi et al. [30] again extended this relation (Eq. 2.2), accounting for the deformation character of small and large particles in a more detailed way. Large

particles can exhibit drastically localised heating (hence softening). In contrast, small particles have rather uniform temperature and, when approaching the melting temperature, inevitably bond due to total melting (for details of bonding mechanism, see section 2.2.3). Further studies derived of deposition windows of various materials and their categorisation according to deformation patterns [31–33].

Stagnation pressure and temperature

Based on good empirical understanding of the critical velocity for a number of materials, the notion of a coating quality parameter, $\eta = \frac{v_{pi}}{v_{cr}}$, the ratio of the particle impact velocity and the critical velocity, was introduced along with parameter selection maps, as in Figure 2.4 [30].

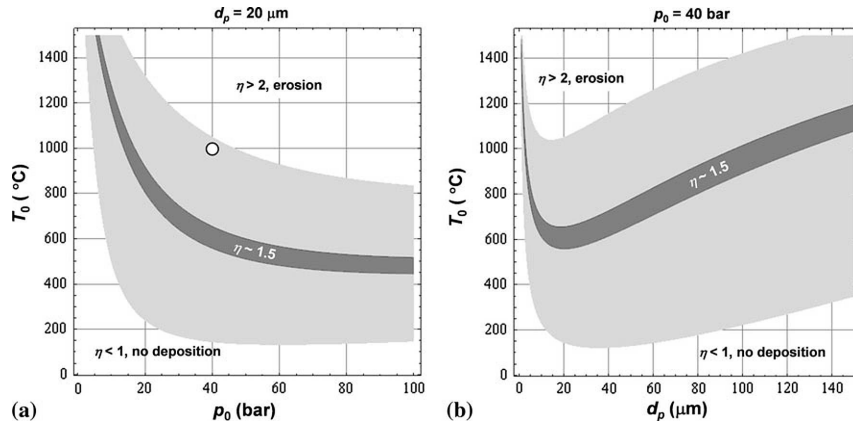


Figure 2.4: The parameter selection maps for Copper indicate the evolution of the deposition window (expressed as regions of coating quality parameter η) with pressure p_0 , temperature T_0 (a) and particle size d_p (b) [30].

Relatively low pressures are required at high temperatures due to the reduced critical velocity, and vice versa, making gas pressure and temperature most influential process quantities besides materials [19]. High operation pressures are favourable in that they enable the use of nozzles with high expansion ratios and hence high Mach numbers. In turn, for a fixed stagnation pressure, there is an optimal expansion ratio which guarantees the maximum particle velocity [34, 35].

The temperature effect is twofold. Firstly, the gas temperature influences the particle temperature, depending on the particle residence time within the flow. Secondly, and most importantly, the speed of sound increases with increasing temperature. These principles

are crucial and therefore discussed in more detail in a review of the gas-particle flow field in section 2.3. Because the focus here is on the process itself, it should be emphasised that, through this connection, a drastic dependency of the pressure and temperature on DE exists [36], and that a result of increased temperature is often a change in micro-structure and mechanical properties of the coating, e.g. porosity and tensile strength [37]. Others [38, 39] verified that DE increases with temperature with the limit of onset of partial melting. Thermal effects on the deposition behaviour of particles are also subject of numerical studies [40].

Feedstock material and substrate

The above discussion already indicates that the particle-substrate material combination and the particle size are important. Similarly, the substrate material, angle, curvature, and size influence the gas impingement and the particle deposition and were constantly kept in focus in CS related research [20]. Gas-atomized powders are commonly used in CS applications, most of which exhibit an approximately logarithmic-normal distribution of particle sizes in the range of $10 - 100\mu m$, as in Figure 2.5a [41, 42].

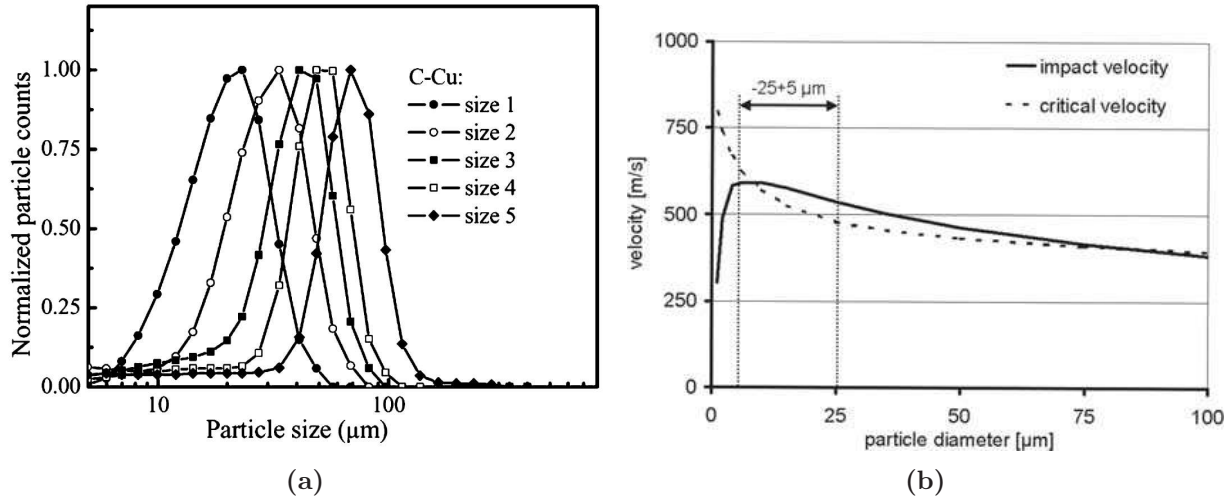


Figure 2.5: (a) Representative copper particle size distribution for various mean sizes [41] and (b) typical influence of the particle size on the deposition [42].

The particle size and weight determine how the particles follow the gas flow, interact with shock waves and impact onto the substrate [43, 44]. Light-weight particles follow the flow more closely and reach higher maximum velocities. Due to its supersonic character, the impinging jet forms a high-density/low-velocity compression layer in front of the

substrate (see section 2.3). Small sized/light particles decelerate in this layer much more noticeably than a particle with more inertia. A too heavy particle however may not cross the critical velocity, limiting the particle size range that can be deposited, as illustrated in Figure 2.5b [42]. Moreover, a change in particle morphology is directly connected to the drag coefficient of the particle and hence the acceleration - irregular particles can reach higher speeds, but follow less predictable paths and impact in a less controlled way [41, 45].

Early work in CS has shown important effects of the stand-off distance (SoD), the distance between the nozzle exit and the substrate [19, 26]. In particular, due to varying impact velocities, the coating quality eventually decreases for large values of the stand-off distance ($>50\text{mm}$) [36]. More recently, three regions of stand-off distances provided explanations:

- 1) small, where particle interactions with the bow shock reduce deposition;
- 2) medium, where the bow shock is weak or has disappeared (favourable);
- 3) large, where gas velocity is below the particle velocity, causing deceleration [46, 47].

A decreasing particle velocity component normal to the substrate has adverse effects on the deposition [21]. As the tilt grows, the shock wave first becomes weaker and the overall velocity of particles increases. Nonetheless, the normal component steeply drops concurrently [48]. The substrate size changes the flow field due to shock curvature, especially for small substrates [49]. Similarly, the normal component of the impact velocity changes for a curved substrate, such as a cylinder, having detrimental consequences for deposition with distance from the jet centreline [50].

More specific substrate treatment can be beneficial: substrate pre-heating [51] and particle pre-heating [42] showed to decrease the critical velocity and particle surface oxides or mixtures of abrasive ceramics and metallic powders can amend the deposition due to surface activation [52, 53].

2.2.3 Cold Sprayed coatings

There is no conclusive physical explanation of the bonding mechanisms between particle and substrate as well as particles and other particles. There are, however, prevailing theories that are widely accepted as the basis for ongoing experimental and numerical

studies. One key feature in the bonding is the formation of sideways-directed jets of substrate and particle material away from the particle-substrate contact zone, to be seen in figure 2.6 [28, 54]. The existence of these jets is evidence of high deformation in a localised zone in the vicinity of the contact.

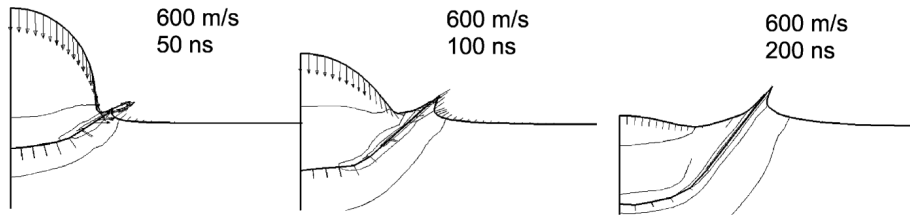


Figure 2.6: Simulation of an impacting copper particle onto a copper substrate, adapted from [28].

It was firstly surmised that bonding is entirely solid-state, also in this contact region [54, 55]. Subsequent investigations, however, predicted localised melting due to adiabatic shear instabilities in the close vicinity of the contact surface [28, 56]; a process in which shearing of the material becomes highly localised due to softening of heated regions that are unable to remove the dissipated heat during the short time scales of impact. Under ideal circumstances, the particle deformation progresses from the centre to the outer region. Sweeping away possible contaminants, it leads to a drastic increase in surface area of conformal particle-substrate contact under high local pressures that results in bonding. In addition, the particle deformation in the dented substrate surface induces mechanical interlocking.

This metallurgical and mechanical bond is described by Van Steenkiste [55] to be one factor of the more complex overall interaction of substrate and particles during coating formation. Four regions, shown in figure 2.7, were identified: 1) the particle-substrate bond in the first layer, as explained above; 2) the compression and realignment of particles in the direction of least resistance, triggered by follow-on particles, leading to void reduction; 3) the formation of metallurgical bonds between particles in upper layers; 4) potential bulk deformation of coating material if the kinetic energy is too high (erosion of layers).

So-produced coatings have some unique advantages, *e.g.* that the material properties of the coating are very close to those of the initial feedstock material: contrasting other thermally sprayed coatings, oxide contents are less than or equal to those of the feedstock [57, 58]. The low thermal input also prevents significant grain growth, such that the grain

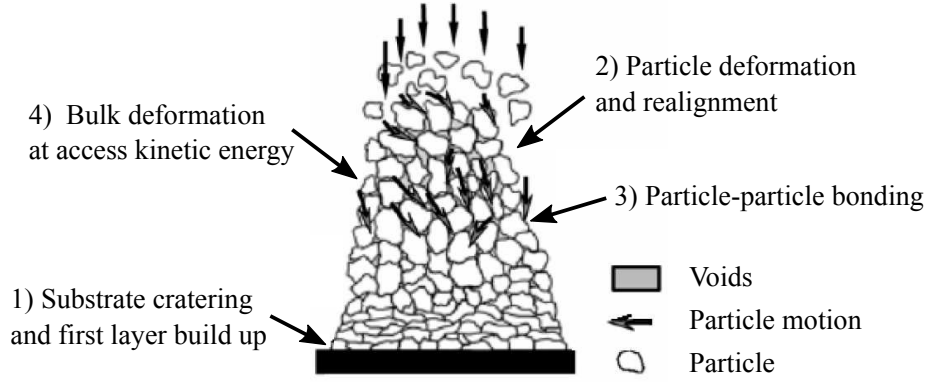


Figure 2.7: The four stages of coating formation in Cold Spray, emulated from [55].

size of the feedstock can be maintained [55, 59]. Hereby, the resulting coating microstructure is generally inhomogeneous with a primary orientation normal to the impact direction. The continuous compression of deposited layers of particles during coating formation leads to void reduction, and hence low porosity of the coating, typically between 0 and 5% [60]. The coating bond strength is crucial for parts that are to be used in mechanical applications and can be as high as 67MPa for aluminium and 50MPa for titanium coatings [61, 62]. Due to excessive work hardening during formation, the coating micro-hardness can be more than twice as high as compared to the feedstock [59]. In copper coatings, the low oxide content and porosity cause an electrical resistivity close to pure copper [57].

This overview of the *modus operandi* and the advantages of the process and coating properties has set the framework in which an advancing understanding of the flow is beneficial. The following paragraphs shift the attention away from the coating formation itself to more fundamental aspects of the two-phase nozzle flow as the central element of the scope of this work.

2.3 Fundamentals of nozzle flow and particle motion

Cold Spray employs a supersonic de-Laval nozzle for particle acceleration, which makes compressible gas dynamics the most important fundamental physics of the process. Likewise, the effects occurring due to the introduction of solid particles in the flow have to be clearly outlined. In the following section, the key features of single- and two-phase flows

are introduced on a general level, in order to introduce all required notions in a compact way. For further information, the interested reader may refer to established literature for gas dynamics [63]. In succession to this section, the ongoing investigations on the gas-particle flow in the field of Cold Spray can be discussed without disrupting fundamental explanation or definition.

2.3.1 Gas dynamic principles

Compressible gas dynamics can be governed fully by the Navier-Stokes equations (NSE) (2.3-2.5), which comprise the conservation of mass, momentum, and energy of a continuum of Newtonian fluid. Here, the variables v , T , E , p are the gas velocity, temperature, specific energy and pressure, and the gas properties ρ , μ , and k denote the density, dynamic viscosity, and thermal conductivity respectively. For further notation refer to the nomenclature.

$$\frac{\partial \rho}{\partial t} + \nabla \cdot (\rho \vec{v}) = 0 \quad (2.3)$$

$$\frac{\partial}{\partial t}(\rho \vec{v}) + \nabla \cdot (\rho \vec{v} \vec{v}) = -\nabla p + \nabla \cdot \mu \left[(\nabla \vec{v} + \nabla \vec{v}^T) - \frac{2}{3} \nabla \cdot \vec{v} I \right] + \rho \vec{g} + \vec{F} \quad (2.4)$$

$$\frac{\partial}{\partial t}(\rho E) + \nabla \cdot (\vec{v}(\rho E + p)) = \nabla \cdot \left(k_{eff} \nabla T - \sum_j h_j \vec{J}_j + \bar{\tau}_{eff} \cdot \vec{v} \right) + S_h \quad (2.5)$$

This set of partial differential equations is mathematically complex and has to be solved approximatively in the majority of cases. It can only be simplified significantly if certain aspects of the flow are less important, *e.g.* if the viscous terms can be omitted. This is particularly helpful to point out some important flow properties like in the following introduction.

Incompressible fluids have the property that local pressure changes are instantaneously transmitted through the entire flow field. In contrast, fluid elements in compressible fluids are elastic and, by changing density under pressure fluctuations, transmit the pressure change to neighbouring elements. This induces an isentropic (specific entropy s remains constant) wave-like spreading of the pressure impulse through the flow field with a certain wave-velocity, the speed of sound c . This is a function of the temperature for an ideal gas,

involving the specific gas constant R of the gas and the heat capacity ratio γ .

$$c = \sqrt{\left(\frac{\partial p}{\partial \rho}\right)_{s=\text{const.}}} = \sqrt{\gamma RT} \quad (2.6)$$

In subsonic compressible flow, the fluid velocity is smaller than the speed of sound, and, the Mach number, which is the ratio of the two, is lower than unity.

$$Ma = \frac{|v|}{c} \quad (2.7)$$

When Ma approaches unity, the transonic regime is reached, in which simplifications for subsonic calculations do not hold. Consequently, Ma larger than one represents fluid velocities surpassing the speed at which pressure information can be transmitted through the medium. This leads to a fundamental change in the fluid behaviour. While in subsonic regimes, properties both upstream and downstream of the fluid element have an influence on its state, in supersonic flows, this influence is limited to upstream properties. This gives rise to the possibility of discontinuous compression waves, so-called shock waves, and an inverse fluid acceleration behaviour. The latter can be illustrated by the area-velocity relation, or Hugoniot-equation, where A denotes the cross-sectional area of the channel.

$$\frac{dv}{v} = -\frac{dA}{A} \frac{1}{1 - Ma^2} \quad (2.8)$$

It states, that a supersonic flow with increasing cross-sectional area will accelerate, and with decreasing area, decelerate respectively. This trend is the opposite for subsonic flows. Descriptively, this means that fluid elements at supersonic speeds cannot “know” how the pressure is going to change downstream, so the only way to respond to more space is to expand. This expansion increases the velocity and decreases the local speed of sound, increasing Ma further. This is the principle of a converging-diverging nozzle, figure 2.8, which triggers a supersonic flow by a sonic or “critical” flow condition in the restriction.

A useful reference state in compressible flows is the stagnation state, indicated by subscript “0”, which describes the state of a fluid that thought to isentropically decelerate to zero velocity. All energy is hence stored in the stagnation pressure p_0 and stagnation temperature T_0 . The concept of isentropic flow is important, as it can be used to reduce

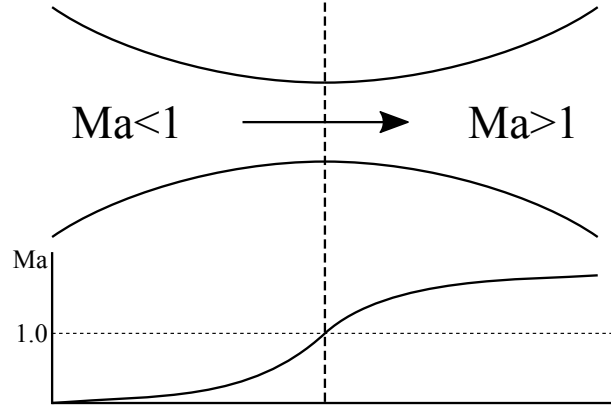


Figure 2.8: Area-velocity or Hugoniot relation, illustrating the principle of a de-Laval shaped nozzle.

a complex compressible nozzle flow to a one-dimensional flow. The Hugoniot relation, in combination with the equation of state for an ideal gas and the isentropic relations of pressure, density and temperature (with the heat capacity at constant pressure c_p)

$$p = \rho RT \quad (2.9)$$

$$p_0 = p + \rho \frac{v^2}{2}, \quad T_0 = T + \frac{v^2}{2c_p} \quad (2.10)$$

$$\frac{p_0}{p} = \left(\frac{T_0}{T} \right)^{\frac{\gamma}{\gamma-1}} = \left(\frac{\rho_0}{\rho} \right)^{\frac{1}{\gamma}} = \left[1 + \frac{\gamma-1}{2} Ma^2 \right]^{\frac{\gamma}{\gamma-1}}, \quad (2.11)$$

can be used to connect the local Mach number to the local cross-section of the nozzle. A given nozzle geometry therefore gives a fixed idealised Mach number distribution, which is exploited in nozzle design procedures and holds high value for approximate flow calculations. The idealisation mainly originates from the fact that no real flow is isentropic. Although gases tend to have relatively low viscosity, the resulting shear stresses are significant in some flow regions. Approaching a stationary wall, for instance, the velocity decreases, forming the boundary layer. The occurring viscous forces are connected to dissipation and therefore entropy production that causes the layer to grow. The main characteristics are described by the Reynolds number Re ,

$$Re = \frac{\rho v l_{ref}}{\mu} \quad (2.12)$$

the proportion of inertial and viscous forces in the fluid, which depends on a problem-

specific reference length l_{ref} . In case of boundary layers, the boundary layer thickness can be used as the reference, in case of flow around a particle the particle diameter (the latter is most important in this work).

Boundary layers change the mass flow and momentum flux of the fluid by displacing the constant velocity region away from the wall. In long ducts or nozzles, this displacement can be sufficiently big for the boundary layers from adjacent walls to meet and form a fully developed flow, which represents a limitation to the centreline velocity of a duct or nozzle.

A second, even more massive contribution to entropy production are turbulent fluctuations. Turbulence is a complex fluid phenomenon - a random motion state that occurs in flows above a case-dependent critical Reynolds number. The onset of turbulence can be thought of as an unstable point of the inertial forces, causing flow structures to build up and collapse in on themselves, producing eddies of a broad band of length scales. For (locally) isotropic turbulence, these eddies are pictured to decay into smaller and smaller eddies until they finally dissipate entirely on the smallest scales, the Kolmogorov scales [63, 64]. As the onset of turbulence is a matter of flow perturbation and flow velocity, very high speed flows tend to be entirely turbulent. Therefore, this phenomenon is fairly important in CS nozzle flows.

One limitation of the above statement, that the nozzle geometry defines the Mach number, is the expansion state of the nozzle. Under- and over-expansion can occur if the nozzle is operated at too high or too low pressure relative to the ambient conditions downstream of the nozzle exit. Under-expansion means that the gas has not expanded, *e.g.* accelerated, enough and still exhibits higher than ambient pressure at the exit. This causes the gas jet to expand further after exiting from the nozzle. The opposite is true for over-expansion, that is characterised by an overly widened nozzle exit, a lower than ambient pressure and hence a compression of the nozzle gas by the environment. This compression can be an oblique shock wave at the exit, or even a shock that moves upstream into the nozzle, if the pressure difference is drastic. Figure 2.9 illustrates this connection and can be used to follow the possible expansion states.

A shock wave is the discontinuous adaptation of low upstream to high downstream pressure in the supersonic flow. This is always connected to massive irreversible, hence,

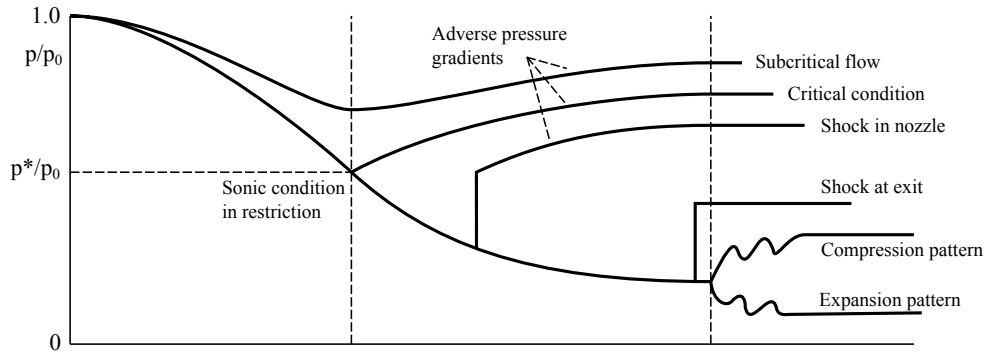


Figure 2.9: Schematic of flow through a de-Laval shaped nozzle, showing different expansion states.

entropy-producing processes, and results in a sudden deceleration of the fluid. The strength of a shock wave and the respective losses depend on the upstream Mach number and the angle of the shock wave, so the higher the pressure difference across the shock wave, the steeper the angle; a normal shock wave is most intense for a given Mach number. Strong shocks are additionally characterised by a sub-sonic velocity on the downstream side and can also be triggered by obstacles, such as a substrate.

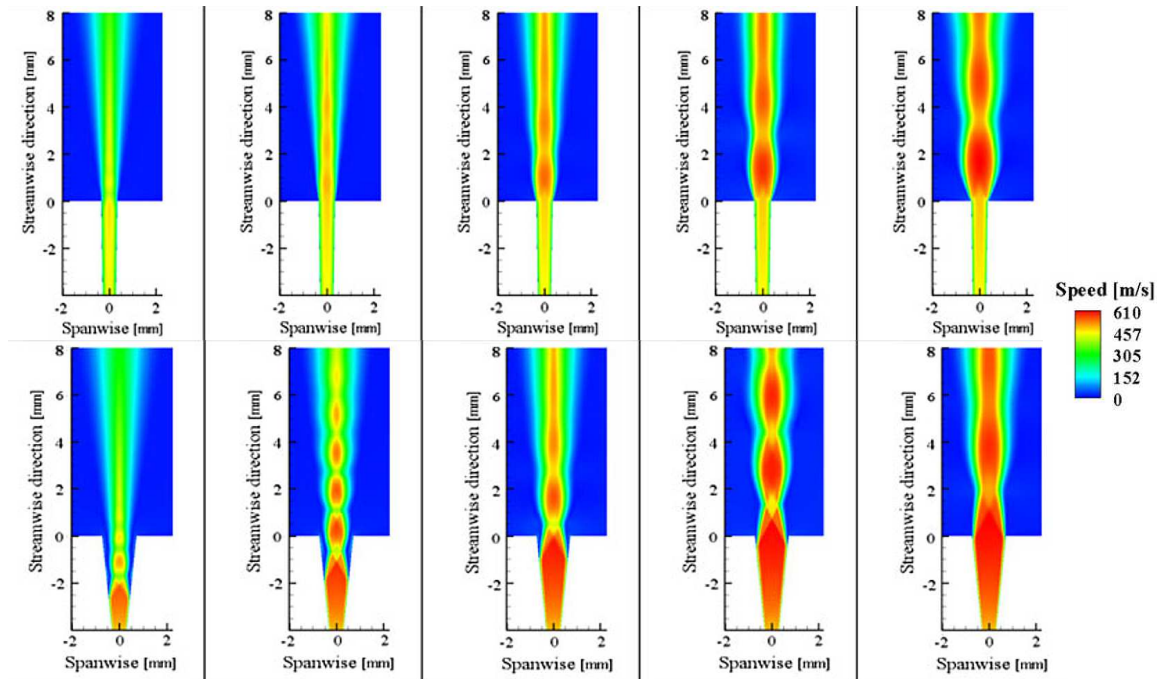


Figure 2.10: CFD results illustrating the influence of increasing pressure on jet patterns: (top) under-expansion due to small nozzle exit (bottom) over-expansion due to wide nozzle exit, from [65].

In reality, the flow features that are created by shock waves and expansion fans are much more complex, as they interact with each other and are affected by turbulence and

viscous stresses. Figure 2.10 shows these structures for the under- and over-expanded case. It can be seen, that the expansion and compression waves reflect on the jet edge and collide on the centreline of the jet. This produces a pattern, which disintegrates with distance from the nozzle due to dissipative losses. More detail of the supersonic over-expanded jet structures can be found in literature [66].

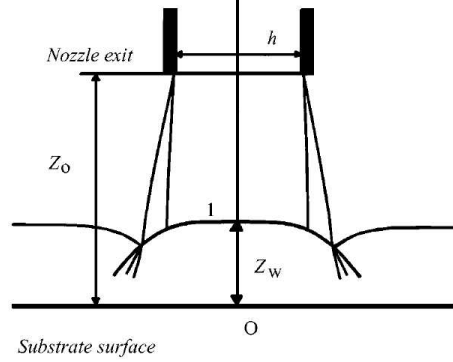


Figure 2.11: Jet Impingement and Normal Shock Wave [67]

Approaching the close vicinity of the substrate, the supersonic jet is forced to decelerate to subsonic speeds and compress by means of a normal shock wave. Fluid elements passing through it are then diverted outwards in the radial direction. In reality, the shock interacts with the shear layer and curves, causing several secondary features, which then reflect off the substrate wall and produce a complex flow pattern, as can be seen in figure 2.11. The zone between the substrate and the shock is called the compression layer and has a certain thickness, that depends on the Mach number.

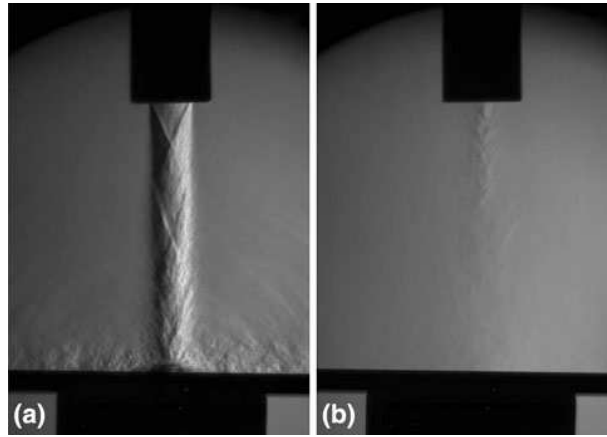


Figure 2.12: Schlieren shadowgraphy image of a typical over-expanded supersonic jet (a) and subsonic flow (b) [22]

It is useful to clarify some concepts of jet flows at this stage. Figure 2.12 shows a

Schlieren shadowgraphy image of a CS jet for supersonic (a) and subsonic (b) operation. The shock structures can nicely be identified. Due to the low co-flow velocity, the jet has a large excess momentum with respect to its environment after exiting from the nozzle. Conventionally, this is expressed as a momentum radius, which corresponds to a theoretical displacement of the jet edge to compensate for excess momentum, similar to the momentum thickness of a boundary layer. The low and high velocity regions are separated by a strong shear layer, which grows with distance from the nozzle. The region within the jet that is encapsulated by these layers is called the potential core of the jet. Correspondingly, a sonic core can be identified for supersonic jets, which represents the region, where the local Mach number is still larger than one. The growth of the jet is driven by fluid entrainment from the co-flow; a combined effect of the radial velocity component of fluid into the jet and the crossing of the axial component into the widening jet [68]. At the point where the shear layers merge, the potential core ends and a self-similar regime establishes at 10-15 times the nozzle exit diameter downstream. It is characterised by a Gaussian velocity profile when normalised by the jet width and the centreline velocity. This profile is maintained downstream despite the absolute velocity decrease, which, in itself, follows a logarithmic trend with distance from the exit [69]. In cases of more complex jets, the behaviour can deviate from this self-similarity, *e.g.* for over- and under-expanded cases [70] or with particle properties in case of a gas-particle flow [71].

This leads directly to the considerations of how the particle motion can be described and what influence this may generally have on the fluid flow.

2.3.2 Particulate phase considerations

The discrete phase is substantially different from the gaseous phase, which is introduced in this section. Again, readers may refer to established literature for more detail [72, 73]. The second phase consists of discrete, non-connected solid particles, that can physically interact with each other and the gas phase by momentum and energy transfer, but not exchange any mass. While the gas phase is treated as a continuum (index c), the dispersed phase (index p) generally cannot be treated as such. This depends on the classification of the mixture, in particular on the volume fraction of particles α_p . This is the volume occupied by the particles in a discrete volume of the flow ΔV_p in relation to the total

discrete volume element ΔV , which approaches an adequately large size with a sufficient number of particles, such that a mixture (index m) can be defined.

$$\alpha_p = \lim_{\Delta V \rightarrow \Delta V_m} \frac{\Delta V_p}{\Delta V} \quad (2.13)$$

The bulk density $\bar{\rho}_p$ has an analogue definition using the particle mass instead of the volume, and can be formulated likewise for the continuous phase respectively. The particle loading can also be expressed in terms of the mass concentration C and the mass loading Z , as the proportions of the mass or mass flow rate.

$$C = \frac{\bar{\rho}_p}{\bar{\rho}_c} \quad (2.14)$$

$$Z = \frac{\dot{m}_p}{\dot{m}_c} = \frac{\bar{\rho}_p v_p}{\bar{\rho}_c v_c} \quad (2.15)$$

A second important notion for the classification of particle laden flows is the particle response time, more precisely, the *momentum or velocity response time* τ_V . Derived from a simplified equation for the motion of a single particle, a characteristic time can be obtained, that represents the time scale of response of the particle velocity to a change in relative velocity of the continuous phase.

$$\tau_V = \frac{\rho_p d_p^2}{18\mu} \quad (2.16)$$

In order to estimate whether a flow is rather dense or dilute, the response time can be compared to the mean time between particle collisions. Therefore, both the volume fraction and the response time determine the classification of the flow regime. If the volume fraction is very low *and* the response time short, the flow is called dilute; dense in case of the contrary. There is a broad range in which the flow is neither clearly dense nor dilute. However, for micron sized particles, the transition from dilute to dense can be estimated for volume fractions in the order of $\alpha_p = 0.01$ -1%, or even lower for dense materials [73]. A very dilute flow is dominated by particle-fluid interactions, whereas the particle-particle interactions primarily influence the flow in a highly dense regime.

The phase coupling must be distinguished from the diluteness of the flow, although an extremely dense flow most likely exhibits strong phase coupling and in reverse. The

degree of coupling of the phases is based on the momentum and energy exchange between the phases. Therefore, it is useful to classify the flow additionally according to a strong or weak coupling.

Based on the response time, the momentum Stokes number Sk can be formulated as the proportion of that response time and a characteristic flow time scale τ_F , which depends on the phenomena of interest. For example, the turbulence interaction could be characterised by the respective turbulent time scale, such as the integral length scale divided by the mean convection velocity.

$$Sk = \frac{\tau_V}{\tau_F} \quad (2.17)$$

The momentum coupling parameter can be used for the estimation of the strength of the momentum exchange. It is defined as the relation of the drag force acting on the particles in a volume and the continuous phase momentum flux through the volume. This can be transformed using the mass concentration C (equation 2.14) to the following expression.

$$\Pi_{mom} = \frac{C}{Sk} \left(1 - \frac{v_p}{v_c} \right) = \frac{C}{1 + Sk} \quad (2.18)$$

Hence, in principle, for large Stokes numbers ($Sk \gg 1$), the particles have little time to respond to changes in fluid flow. In case of a sufficiently low concentration, the motion of the gas phase can be then considered decoupled from the disperse phase, called a *one-way coupling*. If the Stokes number is low ($Sk \ll 1$), the particles can be considered in velocity equilibrium with the continuous fluid, but for high concentrations, this implies strong momentum exchange. Additionally the momentum exchange for moderate Stokes numbers and concentrations can be increased by a large difference in velocity. In either case, the phases are expected to be coupled; a *two-way coupling*. For denser flows, the particle-particle interactions become increasingly significant and extend the coupling to a *four-way coupling* [74]. The above formulations can also be obtained for the energy coupling, based on thermal response times.

For a dense regime, in which particle motion is driven by strong interactions with their neighbours like in the continuous phase, it is possible to reformulate the conservation equations for the gas-particle mixture as a two-fluid model, making use of the volume

fraction and bulk density. Since these equations are derived in a locally fixed, Eulerian reference frame, this approach is called an Eulerian-Eulerian framework, in which both phases are solved as inter-penetrating continua. It requires the volume fraction to be a continuous function in time and space and only few CS applications were faced in this manner.

A different, more common approach is the Eulerian-Lagrangian framework, in which the gas phase is calculated in an Eulerian reference frame and the elements of the dispersed phase are computed in a Lagrangian reference frame that moves with the particles. It is a particularly useful description if the mixture is sufficiently dilute for individual particles to be only influenced by the surrounding fluid. In this case, the respective trajectories are directly calculated by a simple integration of the drag-driven particle momentum conservation equation, so for a one-dimensional case it can be expressed as the following scalar equation.

$$m_p \frac{dv_p}{dt} = \frac{1}{2} \rho C_D A_p (v_c - v_p) |v_c - v_p| \quad (2.19)$$

In this 1D-expression, the connection to the particle Reynolds number can already be noted through the relative velocity magnitude $|v_c - v_p|$. It is now required to define the particle Reynolds and Mach numbers, which characterise the relative flow of gas around particles.

$$Re_p = \frac{\rho d_p |v_c - v_p|}{\mu} \quad (2.20)$$

$$Ma_p = \frac{|v_c - v_p|}{\sqrt{\gamma R T}} \quad (2.21)$$

Using these and extending the momentum conservation for a single particle to three spatial dimensions, the expression can be transformed to a differential vector equation per unit mass, that shows the role of the particle relaxation time τ_r and the involvement of the Reynolds number in it; here finally given in vector notation for 3D.

$$\frac{d\vec{v}_p}{dt} = \frac{1}{\tau_r} (\vec{v}_c - \vec{v}_p) + \frac{\vec{g}(\rho_p - \rho_c)}{\rho_p} + \vec{F} \quad (2.22)$$

$$\frac{1}{\tau_r} = \frac{18\mu}{\rho_p d_p^2} \cdot \frac{C_D Re_p}{24} \quad (2.23)$$

The gravitational term can usually be dropped except for buoyancy driven flows, whereas

additional body forces, such as added mass or lift forces, may arise in the term $\vec{F}$ on the right hand side. The calculation of the drag coefficient C_D is a subtle topic in itself, because it depends strongly on the flow conditions and the particle shape. It represents the micro-flow around the particle, in particular depending on Re_p and Ma_p , and therefore varies with location and flow conditions. Some models to calculate these quantities for CS are discussed in the review of numerical approaches in section 2.4.

Having now set the basic terminology of gas-particle nozzle flows, all concepts are introduced that are required to follow the respective details in published CS research as well as the present work.

2.4 Gas-particle flow in Cold Spray: simulations and experiments

This section reviews the analysis methods with respect to the flow in CS and summarises interesting findings. Therein, it is a useful approach for clarity to separate computational and experimental approaches, and only merge the two when necessary. It will become clear in this section, that despite numerous work in the CS flow analysis, both experiments and models do not yet provide the required means to fully understand the particle acceleration. This especially prevails with respect to the injection process and operation at increased particle feed.

2.4.1 Analytical modelling

Historically, analytical models were the common method to predict the flow inside the cold spray nozzle and are still sometimes employed due to their ease of use [43, 75–78]. The 1D-isentropic model is a drastically simplified approach, but results in surprisingly reliable predictions in some cases. However, a significant shortcoming of the model is the negligence of ambient conditions and the resulting free jet physics, as well as viscous losses, turbulence, boundary layer, and the bow shock [26, 43, 79].

Some of these downsides were addressed nonetheless: boundary layers and the bow shock were included into the 1D model by virtue of experimental data [67]. Likewise, Kosarev *et al.* developed an empirical model to describe the gas flow properties, extending

the selection by 2D velocity distributions in the jet cross section and compressed layer [80]. Despite good agreement between the analytical results and measurements, these formulas are highly specific and mathematically rather complex, which prevents them from being a general solution. Also, empirical models limit the depth of physical insight, they do not answer “how” phenomena arise.

In an analogous way, the particle motion can be approximated using simplified formulae. If in the equation of motion for a particle only one dimension is considered and the drag coefficient as well as the gas state are assumed constant, a simplified expression for the particle velocity can be obtained [75]. The assumptions generally do not hold, leaving significant inaccuracies in the prediction. In addition, the particles’ passing through the series of shock waves is neglected, that could primarily be addressed (semi-)empirically to obtain a particle impact velocity. Further similar approaches can be found in this respect [30, 79].

Moreover, these simplified concepts were used to optimise the nozzle shape by virtue of maintaining a maximal particle acceleration [26, 67, 75]. This identifies another very important purpose of analysis methods: the nozzle design is a crucial aspect of CS and requires robust tools. An inviscid calculation advanced to 2D is the method of characteristics (MoC) - giving the respective type of nozzles their name. Characteristics are particular directions in the flow field, along which the solution is continuous and does not depend on the derivatives across them. In 2D-irrotational steady inviscid supersonic flows, the concept can be exploited to calculate how Mach lines are produced and annihilated to form a smooth supersonic flow, *e.g.* obtaining CS nozzle wall contours of the divergent section [25, 27, 42, 81]. A convenient feature of the method is that it is discretely solvable and hence can be computed on digital machines. It is thus a rudimental element of computational fluid mechanics (CFD).

2.4.2 Numerical analysis

Numerical models and methods

In order to assess a greater variety of nozzle geometries, more complex CFD methods (*i.e.* Finite Volume Method) were progressively employed in the design procedure of nozzles [82]. Similarly, such CFD is considered a valuable analysis tool for particle and gas behaviour,

much more precise and adjustable than analytical and empirical models. Moreover, it can massively reduce experimentation cost and effort, and even provide direct insight into non-measurable flow quantities. With increasing computational power, CFD has grown to an essential means of work in CS, that is reviewed here before giving an overview of findings based on these methods.

In cold spray modelling, the flow can be regarded as a steady state, described by the NS equations as introduced in section 2.3.1 (eqn. 2.3-2.5), in an time- or ensemble-averaged form, the Reynolds-Averaged Navier-Stokes equations, with the averaging procedure

$$\Phi = \bar{\Phi} + \Phi' \quad (2.24)$$

$$\bar{\Phi'} = \lim_{T \rightarrow \infty} \frac{1}{T} \int_{t_0}^{t_0+T} \Phi' dt = 0. \quad (2.25)$$

where Φ is a placeholder for any flow quantity and t_0 is an initial time. The set is closed by the equation of state for compressible flow, generally the ideal gas law. The source terms appearing in the momentum and energy equations are often neglected, but under certain conditions, *e.g.* high powder feed rate, the momentum and energy transfer between the gas and the particulate phase can be accounted for by these terms [77, 83–85]. The gas viscosity is usually set constant or calculated by the temperature-dependent Sutherland law [46, 50, 86, 87]. Specific heat and thermal conductivity are mostly assumed constants, with few exceptions [88, 89].

Solutions are calculated in a discretised computational domain, normally on a structured mesh as the more efficiently converging choice over the unstructured meshes. In some 3D models, unstructured meshes were necessary to deal with complicated geometry [77, 81, 90]. The discretisation of the NS equation set is done in a variety of schemes, which can affect the results greatly. Low-order schemes result in low accuracy but help the convergence and save the computational time, while the character of the scheme may have additional stability effects and influence the accuracy of the solution, particularly of the shock-wave structure [91]. So far, no publications paid significant attention to the selection of discretisation schemes, although mostly second-order upwind schemes were reported [23, 48, 88, 92, 93]. A systematic comparison of the schemes for CS applications is recommended for future computational work.

Turbulence model	Model theory	No. of equations	References
Standard k - ε	RANS	2	[44, 47–49, 76, 77, 85, 93–105]
RNG k - ε	RANS	2	[23, 34, 35, 45, 53, 65, 90, 106–112]
Realizable k - ε	RANS	2	[81, 86, 90, 113–115]
RSM	RANS	7	[36, 50, 83, 84]
SST k - ω	RANS	2	[116]
Spalart-Allmaras	RANS	1	[87, 117]
Compressibility modified k - ε	RANS	2	[86, 92]
Thermally modified k - ε	RANS	2	[88, 89]
Multiphase modified k - ε	RANS	2	[118]
LES	LES	-	[119]
Laminar	-	-	[120]

Table 2.1: Summary of the turbulence models employed in cold spray modelling, originally published by the author in [121]

The fluctuating components that were filtered out in the RANS formulation, leaving only the term that effects the average distribution depending on the average fluctuation, the Reynolds stress tensor $-\rho \overline{u'_i u'_j}$. Its components must be modelled for the closure of a turbulent flow. Table 2.1 summarises the turbulence models employed in cold spraying. Most models follow the Boussinesq assumption that the Reynolds-stress components can be correlated to the mean velocity gradients. In this case, only one or two additional transport equations need to be solved in order to model the eddy-viscosity. The Spalart-Allmaras, as a 1-equation model, was initially designed for aerospace applications involving wall-bounded aerodynamic flows, but it produces relatively large errors for free shear flows, especially plane and round jets. The standard k - ε model is the most frequently used model. It solves two transport equations of the turbulent kinetic energy k and the turbulent dissipation rate ε . As such, it is widely applicable, relatively simple to implement and normally converges stably. The RNG k - ε model introduces an additional term in its ε equation that significantly improves the accuracy for rapidly strained flows. The realizable k - ε model introduces a different transport equation to describe the turbulent dissipation rate, and imposes a restriction on the eddy-viscosity to ensure positivity of normal stresses and a similar “realistic” condition on shear stresses. This model is able to accurately predict the spreading rate of both planar and round jets, providing superior performance for flows involving rotation and boundary layers under strong adverse pressure gradients. The SST k - ω turbulence model can also be used for the compressible flow in divergent-convergent nozzles [122, 123]. It solves the near-wall region of the boundary layer by the robust k - ω model and switches to the k - ε in the free shear flow, combining the advantages of both models. In addition to the aforementioned models, some modified k - ε models for

special usage were also employed in different studies, which will not be discussed in detail. The advanced Reynolds stress model (RSM) was also used occasionally, in which the eddy viscosity approach is omitted and the independent Reynolds stress components are solved separately. It accounts for non-isotropic turbulent effects in flows with streamline curvature, swirl, rotation, and rapid changes of strain rate and thus leads to more accurate prediction of complex flows compared to other models. However, one disadvantage of this model is that it requires a high computational effort. It should be noted that particle-laden turbulent jets in reality exhibit a high degree of non-uniform turbulence, which has an effect on the particle dynamics and dispersion, depending on Stokes number [71, 124]. Moreover, a large eddy simulation (LES), which directly solves the large eddies in the computation and implicitly accounts for the small scale eddies using a sub-grid scale model, also can be found in literature. As a more advanced method, LES normally requires massive computational time, thus it is also not recommended for a general approach.

Under the assumption of a small volume fraction, one-way Lagrangian discrete phase modelling (DPM) method was predominantly used in CS to compute the particle velocity and temperature. After the solution of the gas flow, the particle trajectories are obtained through a single integration of the particle equation of motion, as introduced in section 2.3.2 (2.23). Some publications employed a two-way coupled Lagrangian approach for the calculation of the particle trajectories, which accounts for the effect of the particulate phase on the gas phase [77, 83–85, 94], and requires an iterative solution of the equation. This method is more suitable for the high particle loadings than one-way Lagrangian methods, however, still neglects the volume of particles and particle-particle interactions. Generally, the body force term was not included in the drag force balance equation, but some works added a pressure gradient force [50, 113]. Also, gravity force [112], thermophoretic force [81, 107], lift force [108], and electrostatic force [112, 119] were included in some other studies. Rarely, the dispersed phase was also considered as an Eulerian continuum [113, 118], and the particle velocity is calculated by solving the mixture equations, which may result in more realistic results when modelling a dense particulate flow with strong exchange between particle and gas phase. A conclusive study on the adequacy of each model is lack in literature.

Determining the biggest contribution to the particle force, the drag coefficient is the

No.	Authors	Equations	Range	Application	References
1	Schiller and Naumann [125]	$C_D = \max\left(\frac{24}{Re_p} (1 + 0.15Re_p^{0.687}), 0.44\right)$	laminar	Incompressible	[89, 116]
2	Morsi and Alexander [126]	$C_D = a_1 + \frac{a_2}{Re_p} + \frac{a_3}{Re_p^2}$	$0.1 < Re_p < 50,000$	Incompressible	[49, 84, 87, 93, 96, 97, 99, 100, 105, 107, 127, 128]
3	Clift et al. [129]	$C_D = \frac{24}{Re_p} (1 + 0.15Re_p^{0.687}) + \frac{0.42}{1 + 4.25 \times 10^4 Re_p^{-1.16}}$	$Re_p < 300,000$ $Ma_p > 0.4$	Incompressible/ Compressible	[23, 34, 46, 86, 92, 106, 109, 111, 115]
4	Haider and Levenspiel [130]	$C_D = \frac{24}{Re_p} \left(1 + b_1 Re_p^{b_2}\right) + \frac{b_3 Re_p}{b_4 + Re_p}$	$Re_p < 260,000$	Incompressible	[45, 105, 117]
5	Crowe [131]	$C_D = (C_{D, incomp.} - 2) \times \exp\left(-3.07\gamma^{1/2} \left(\frac{Ma_p}{Re_p}\right) g(Re_p)\right)$ $\dots + \frac{h(Ma_p)}{\gamma^{1/2} Ma_p} \times \exp\left(\frac{-Re_p}{2Ma_p}\right) + 2$	$0.2 < Re_p < 10,000$ $0.1 < Ma_p < 2.0$	Compressible	[50, 83, 118]
6	Henderson [132]	$C_{D1} = 24 \left[Re_p + S \left(4.33 + \left(\frac{3.65 - 1.53 \frac{T_p}{T}}{1 - 0.353 \frac{T_p}{T}} \right) \times \exp\left(-0.247 \frac{Re_p}{S}\right) \right) \right]^{-1}$ $\dots + \exp\left(-\frac{0.5Ma_p}{Re_p^{1/2}}\right) \left(\frac{4.5 + 0.38(0.03Re_p + 0.48Re_p^{1/2})}{1 + 0.03Re_p + 0.48Re_p^{1/2}} + 0.1Ma_p^2 + 0.2Ma_p^8 \right)$ $\dots + 0.6S \left(1 - \exp\left(-\frac{Ma_p}{Re_p}\right) \right)$ $C_{D2} = C_{D1}(Ma_p = 1.0, Re_p)$ $\dots + \frac{4}{3} (Ma_p - 1) (C_{D3}(Ma_p = 1.75, Re_p) - C_{D1}(Ma_p = 1.0, Re_p))$ $C_{D3} = 0.9 + \frac{0.34}{Ma_p^2}$ $\dots + 1.86 \left(\frac{Ma_p}{Re_p} \right)^{1/2} \left(2 + \frac{2}{S^2} + \frac{1.058}{S} \left(\frac{T_p}{T} \right)^{1/2} - \frac{1}{S^4} \right) \left(1 + 1.86 \left(\frac{Ma_p}{Re_p} \right)^{1/2} \right)^{-1}$	$C_{D1} : Ma_p \leq 1.0$ $C_{D2} : 1.0 < Ma_p \leq 1.75$ $C_{D3} : Ma_p \geq 1.75$	Compressible	[45, 53, 76, 77, 81, 87, 95, 113]
a_1 - a_3 are constants from several ranges in Re_p , b_1 - b_4 are constants depending on particle sphericity, $C_{D, incomp.}$ from law 3, $\log_{10}g(Re_p) = 1.25(1 + \tanh(0.77\log_{10}g(Re_p) - 1.92))$, $h(Ma_p) = (2.3 + 1.7(T_p/T)^{1/2}) - 2.3\tanh(1.77\log_{10}(Ma_p))$, $S = Ma_p(\gamma/2)^{1/2}$					

Table 2.2: Summary of models for drag coefficient for particle velocity computations, first published by the author including formulae in [121]

main driver for velocity and trajectory prediction. Table 2.2 is a summary of the commonly used equations for modelling the drag coefficient in cold spraying [126, 129–133]. Drag law no.1 by Schiller and Naumann was originally developed for laminar flows. Drag coefficients 2, by Morsi and Alexander, and 3, by Clift *et al.*, have simple expressions, are integrated in commercial software and hence widely used. Drag law 4 is developed for non-spherical particles. Despite their numerous applications, these expressions originally were derived for incompressible flow and do not consider compressibility effects, which is in conflict with fundamentals of the process. The law by Clift *et al.* incorporates a correction for particle Mach numbers exceeding 0.4 and can hence be applied to such compressible regimes, but remains invariant under Mach number changes. As reported in a previous study [72], in particle-laden flows where the particle Mach number exceeds 0.6, shock patterns may form on the particles that significantly affect the particle motion. Drag coefficients which include Mach number terms are therefore favourable: the drag coefficients after law no. 5, by Crowe, and 6, by Henderson, are such kind of coefficients, which lead to significantly increased velocity results [45]. Some rarely used drag coefficients also can be found in previous publications, which are not discussed in detail [44, 84, 120]. Although many kinds of drag coefficients have been used to predict the particle velocity in cold spraying, a systematic comparison between different coefficients and the development of a CS-specific law is still missing.

With respect to heat transfer, particles can be assumed to have uniform temperature; their Biot number, the ratio of the conductive resistance inside the particle and the convective resistance at its surface, is much smaller than 0.1 [36, 77, 87, 134, 135]. The particle temperature can be calculated by a forced convection formula [136], in which the particle recovery temperature is a function of the Mach number and the heat transfer coefficient is normally calculated from the Nusselt number using a Ranz-Marshall relation [137]. Some modified forms of such relations for the Nusselt number were also used occasionally in previous works for high particle Reynolds number [138], high Mach number [139], or boundary layers on the particle surface [140]. Similarly, heat transfer to nozzle and substrate walls can be considered by enabling the respective boundary to solve a conduction equation of heat into the solid body.

Heating and the issue of nozzle clogging

Although the analysis of heat transfer is of subordinate significance in this study, it should be noted that extensive work was done on this topic, both experimentally and computationally. As stated in section 2.2, substrate, particle and gas heating affect the deposition and coating properties. Higher substrate temperature helped the coating formation [141–143]. CFD studies investigated temperature distribution in the substrate [101] and effects of operation temperature, materials [102], stand-off distance, substrate size, and spray angle [49, 128, 144] that all have influence on the heat transfer. Additionally, the turbulence model was determined to mainly contribute to thermal prediction accuracy [88].

Nozzle clogging is an often-encountered issue during nozzle operation, which is mostly connected to the thermal aspects of CS. It is the effect of unwanted feedstock deposition on the internal surfaces of the CS nozzle. It has been frequently observed and is especially problematic with low-melting feedstocks, such as Al or Zn alloys. It is suggested that such powders are prone to clog the nozzle, as excessive thermal softening reduces the critical velocity and causes nozzle internal deposition [102, 106]. Nozzle clogging is a gradual process and usually occurs after long spray times: spraying an Al-Si feedstock at about 500°C caused clogging after 20 minutes [145]. The same effect can also appear for the injector needle, as its tip is exposed to high temperatures, where it submerges carrier gas into heated process gas. Resulting clogging can be prevented by sufficient (self-cooling) carrier gas flow or cooled injector needles [77, 94].

Clogging of the whole nozzle is much more challenging to prevent. In some studies, the feedstock was mixed with ceramic particles to lower the risk of clogging, *e.g.* Al_2O_3 particles were added to spray a Ni-Cr alloy at elevated temperatures [146]. The alumina would continuously wash off deposited small feedstock particles and hence push the limiting conditions. Positive side effects were the surface activation and shot peening effect of the ceramic particles. Other means against clogging were developed by choice of nozzle material. A study used a shortened ceramic nozzle to prolong operation until clogging of commercially pure Al [147]. This is helpful because low-conductivity nozzle materials, *e.g.* stainless steel, cause higher wall temperatures and therefore raise the risk [102]. Spraying of Nickel based super-alloys like Inconel-718 is generally difficult and also very prone to nozzle clogging. This material was tested to spray best avoiding clogging with a anti-

clogging-material nozzle in combination with a water cooling system [148], as the thermal effect could not fully explain this behaviour. Aiming to reduce the wall temperature in the throat region, where clogging was expected to be most likely, a cooling device was shown to amend the nozzle performance [106]. Another possibility to improve such a performance is to use polymer nozzles (Polybenzimidazole). A problem remains that these nozzles wear out rapidly, are difficult to manufacture and are therefore expensive [149].

It was not until recently, that studies picked up a computational analysis of the particle behaviour within the nozzle, advertising that the wall-impacts due to dispersion are just as crucial as the temperature [89, 106]. Such computational analysis of the actual mechanisms of nozzle clogging are still ongoing: it was calculated that fine particles cause initial clogging because they heat up (and respectively stick) easily [150]. Consequently, thermal softening is an important factor that was addressed in several ways. However, it cannot be the key feature in every case of nozzle clogging, which shows that the phenomenon is not yet conclusively investigated. Nickel was found to clog nozzles at temperatures of 450°C , hence significantly below the melting point of 1450°C [151]. Moreover, the material deposited in the diverging section; local temperatures are relatively low due to the expanded gas, such that nozzle cooling is of no benefit. This study suggests that, for some cases, the temperature becomes secondary and the role of the wall-interaction velocity becomes the primary factor, such that injecting into supersonic section with narrow needle is proposed for avoiding clogging.

CFD work on gas and particles

Apart from basic gas dynamics covered in section 2.3, more sophisticated CS-specific findings were reported with the aid of CFD. On the gas phase, for example, the powder injection conditions have a noticeable influence; the gas velocity and temperature in front of the nozzle restriction decrease with increasing injection pressure due to the heat exchanges between the low-temperature carrier gas and high-temperature main gas [107, 109], while higher injection temperatures provide extra energy input and result in elevated velocities [111]. Moreover, the gas velocity and temperature distribution in the nozzle was modelled to be more uniform if the injection was located in the pre-chamber [95, 107].

Modelling of varying stand-off distances showed a two-fold influence on deposition

efficiency through particle impact velocity. As paraphrased in section 2.2, it was proposed that the sonic core length determines the bow shock strength and hence the particle velocity drop [46]. In addition, the pressure oscillations due to the shock pattern in the supersonic regime could provide a favourable substrate location, namely where pressure is lowest [50, 118]. It seems that future work will clarify which of the effects is predominant for a given application. This also applies to substrate shape and size (section 2.2). Several publications state that the particle velocity at the nozzle exit decreases as particle size or density increase; the reverse is true at the bow shock [21, 27, 30, 36, 42, 108, 109, 111, 120, 152].

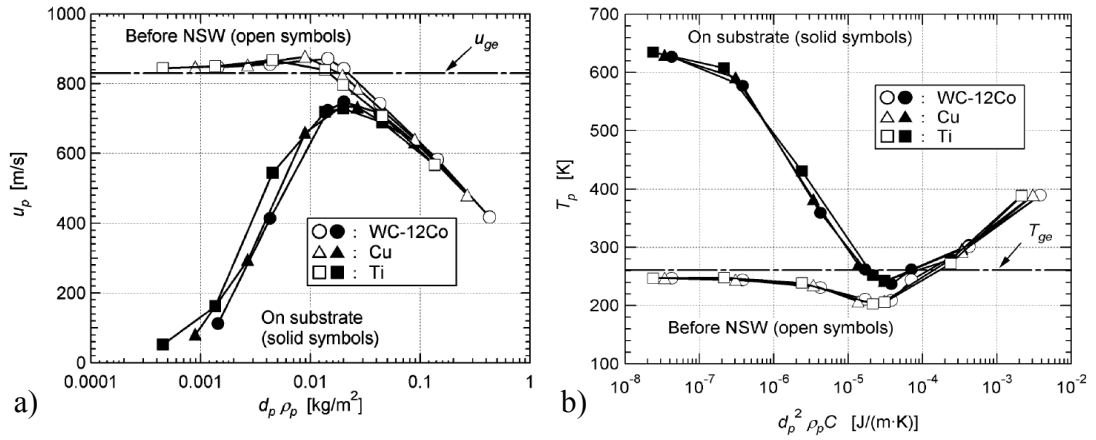


Figure 2.13: Particle velocity vs. PVP (a) and particle temperature vs. PTP for 3 materials across shock wave [44].

Katanoda *et al.* summarised this (and an equivalent thermal) connection in the Particle-Velocity-Parameter (PVP) [44]. Particles with high PVP are difficult to accelerate but maintain their velocity in the compression layer, see figure 2.13. Analogously, the Particle-Temperature-Parameter (PTP) is important for particle pre-heating and cross-shock heating. Because the temperature is high where velocity is low, an injection sufficiently far upstream from the nozzle throat corresponds to long residence times of particles in a high-temperature environment, resulting in stronger heating. Low-PTP particles lose this thermal input entirely in the cold jet and regain some temperature in the compression layer. Thermally more inert particles preserve more of their temperature in the cold jet and do not react to the hot compression layer. Interestingly, PVP was earlier implicitly also identified to be the upper limit of the Mach number for nozzle design due to progressively stronger bow shocks [43].

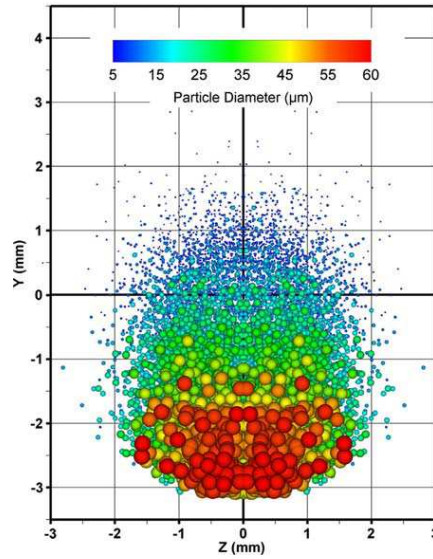


Figure 2.14: Asymmetric particle footprint of different particle sizes at side injection [50].

Particle dispersion is an important topic for the understanding of impact conditions, because the jet-radial gas velocity profile forces particles far away from the centreline to decelerate [81, 109, 113, 115]. Karimi *et al.* [86] developed a CFD model to explore the particle trajectories and distribution in the gas stream, with a sideways injection of particles at an angle of 45° , and found a 3D skewed footprint on the substrate. A similar study shows that, with increasing diameter, particles disperse less severely but impact at larger distances from the centreline, which results in an asymmetric footprint as shown in figure 2.14 for the particles between 5 and 60 μm on a flat substrate [50]. For axial injections, other studies report that particles disperse downstream of the injection point in the pre-chamber or nozzle convergent part with some particle concentration in the central region [89, 95, 109, 115]. Dispersion is increased by collisions between particles and the nozzle wall and increases the risk of nozzle clogging. Besides, higher injection pressure augments the particle dispersion in the convergent part [109]. It should be noted that particle-particle interactions and effects of particles on the gas phase were not subject of these studies and may well have an interesting influence on dispersion.

Understanding of loading

Literature so far does not provide details of fundamental phase interaction processes; a statement, which applies mainly to the sensitivity of the drag force to C_D -formulations

on the one hand, as well as to the physical mechanisms of phase coupling on the other. While the drag coefficient is merely transferred from other fields, in many of previous numerical studies in CS, gas phase changes with particle loading were neglected because of a low estimate of volume fraction. Nevertheless, results have ambiguous tendencies, and whether uninvestigated connections are negligible, generally remains at the discretion of the researcher.

For instance, Gilmore showed a velocity drop with loading (figure 2.15), while Schmidt *et al.* [27] state that a 5% velocity loss at 10% mass loading is perceived as small, and that particle-particle collisions are rare. They and many others (see section 2.2), state at the same time, deposition efficiency is very sensitive to changes in velocity, at least close to the boundaries of the deposition window. Such negligence has a weak foundation, if low-sensitivity and high-sensitivity parameters combine, but only few studies investigate the aspect in some detail. Results demonstrated by Samareh *et al.* [83, 118] indicate that

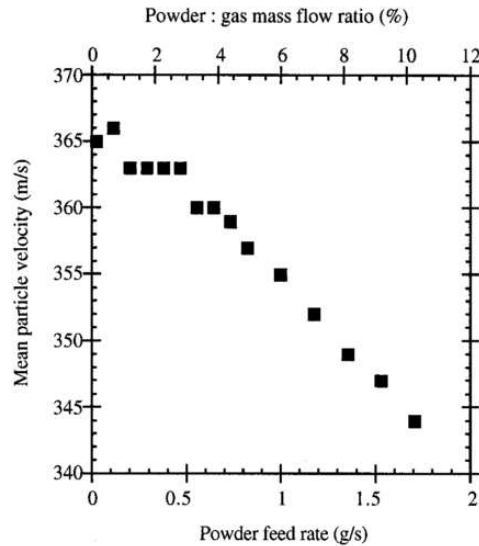


Figure 2.15: Initial evidence of particle mass loading effect on particle velocity [21].

the gas flow structures weaken as evidence of lowered gas phase momentum under high loadings. Consequently, the particle speed reduces as their mass fraction increases (see figure 2.16).

The effect is similar for a range of particle diameters and can be approximated to 7-10% by a coupled Lagrangian CFD approach, although only one database may not be sufficient evidence and more studies employing different techniques seem required. The simulated

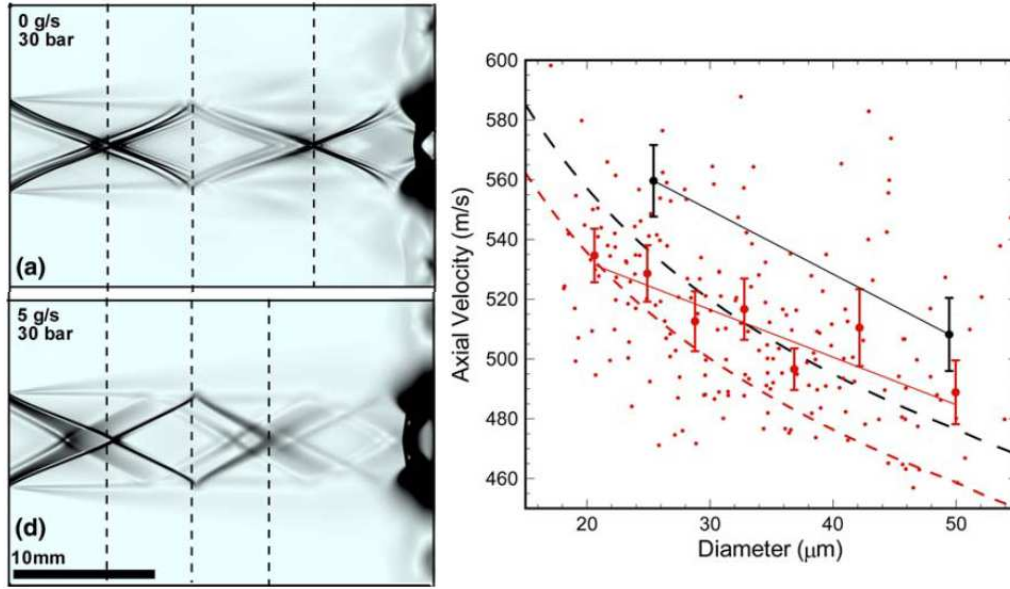


Figure 2.16: Gas structure change simulation (left) and particle velocity loss measurement and simulation (right) with particle mass loading, from [83].

effects are more drastic, if the CFD approach is fully Eulerian, particularly close to the centreline, where local subsonic regimes may occur merely due to particle loading. This remains without experimental validation. Accordingly, a study by Lupoi [153] showed that a number of experimental deposition efficiencies of various nozzle geometries could not be explained by one-way coupling CFD techniques, which could be improved by a phase coupling [154]. Although, by some researchers, the particle velocity was calculated to exhibit a very slight decrease with loading at generally low levels of feed rates [81, 85], CS applications are increasingly set up in a feed rate range, for which this assumption may not hold. A reason for this is that faster processing times in CS manufacturing applications can be achieved by higher feed rates. A cost analysis of the CS process by Stier [155] provides evidence that understanding the mass loading effects enables the optimisation of gas and powder consumption without loss of deposition efficiency, and is hence very interesting on an economical level.

In fact, in most CS publications (*e.g.* [81, 107]), the volume fraction is not explicitly mentioned, but stated to be less than 10-12%, yielding to a dilute flow assumption. However, it is consistent with the mixed observations in the field that these concentrations already imply a dilute-to-dense transitional regime. In this respect, it is useful to investigate some values briefly. Samareh *et al.* [118] reported a feed rate of 0.66-117g/min,

the respective volume fraction range was 0.024-4.2% and the mass loading 0.1-15%. Volume fractions of the same order, $\mathcal{O}(0.01\%) \cdots \mathcal{O}(1\%)$, can be regarded as a dense-dilute transitional regime according to theoretical estimations from section 2.3.2. Since such powder feed rates are rather typical for CS, it is possible to infer for other studies, which mention only the feed rate, *e.g.* 0-102g/min [21] or 70g/min [51], that the volume fraction is usually in a similarly transitional regime. The vast majority of reports do not explicitly indicate the Stokes number either, but the particle size range is usually given. For a comparison of different CS jet flows, typical values of Stokes numbers of $Sk=0.88-131$ were published by Li *et al.* [120] for particles between 5-50 μm in size, hence in a moderate to high range, meaning approximate momentum coupling parameters up to the order of $\mathcal{O}(10^{-1})$. Consistent with some measurable effects, this represents a regime in which coupling plays a role. It is important to note that the fluid time scale, that these Stokes numbers are based on, was determined from the nozzle exit diameter and the speed of sound. This is a reasonable choice of scale when investigating the particle response to the shock waves, as done in respective study. For the overall interaction of particle and gas along the nozzle, this fluid time scale is not representative. CS nozzles are relatively long nozzles, explicitly for the purpose of compensating for the large particle response time. Hence, the nozzle length and the gas velocity could be a more adequate choice for the fluid time scale representing the acceleration process. Assuming conservatively that only the flow through the diverging section provides the advective fluid time, the resulting Stokes numbers could be well an order of magnitude below the given, strengthening the coupling parameter. Moreover, this argumentation does not yet include any consideration of particle-particle interactions, originating from a sufficiently high volume fraction. The expanding nature of the CS process implies that the relevant phase interactions even vary locally. For instance, the low speed section exhibits a denser mixture and may exhibit particle collisions, whereas the dilute jet may still be free of any such effect. It becomes clear that these considerations are “rules of thumbs” and therefore a matter of definition; any argumentation based upon them is weak for the case of CS nozzle flows. In order to make meaningful statements, the actual physical mechanisms of interaction have to be observed and understood. As stated here, very limited work on this aspect can be found in literature to date.

CFD applied to nozzle design

Despite the lack of strong experimental validation as highlighted in the sections above, the described CFD models used to date have been applied to optimisation processes of the nozzle design. Beyond aforementioned analytical optimisation processes and the smooth MoC-nozzle design, CFD techniques were employed to investigate the nozzle length and expansion ratio, as well as the injector design, which can be summarised to be core elements of the design. Resulting from supersonic gas dynamics (section 2.3), there is an optimal expansion ratio for a given pressure level that guarantees the maximum particle velocity [34, 35], which is particularly important for nozzles with limited length specifications [93].

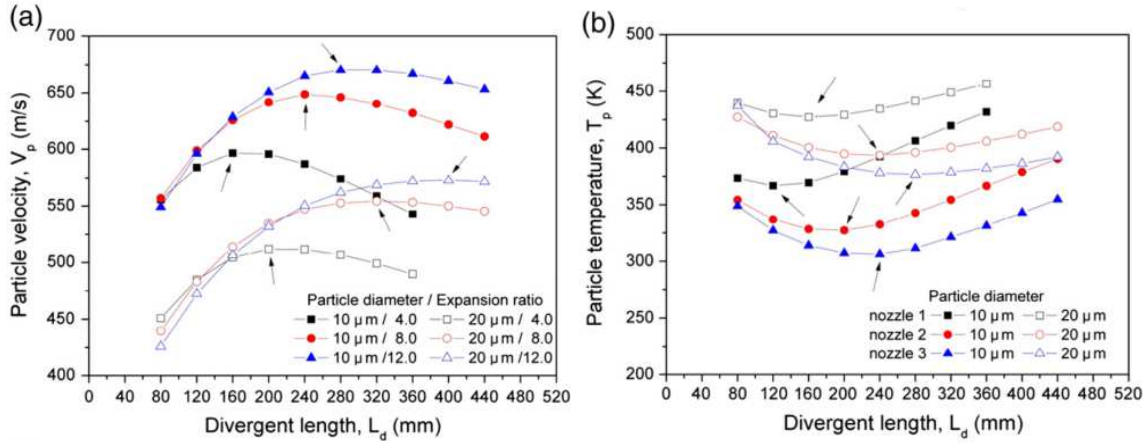


Figure 2.17: CFD applied to nozzle design optimisation: influence of the nozzle length on particle velocity and temperature [34].

This is illustrated in figure 2.17. The nozzle divergent length poses significant impact on the particle impact velocity [34, 97]. With increasing divergent length, the particle velocity grows at first due to the increased acceleration time. At a certain length however, the particle velocity begins to decrease with further increase of the divergent length because the gas velocity level experiences aggravating viscous and turbulent losses - particularly after a full development of the nozzle internal flow. In order to keep the majority of particle trajectories close to the centreline and therefore lower the risk of dispersion-driven deceleration, studies suggest to use nozzles rectangular and circular in cross-section [81, 99, 100].

The powder injection location is important for particle acceleration behaviour. Releasing particles in the high-pressure section, the injection position is computed to rarely affect the particle velocity because the particle acceleration mainly takes place in the divergent

part [99], consistent with measurements of DE [25]. However, if the injection point is in the low pressure part and shifted downstream, the impact velocity decreases due to the shorter acceleration time [99]. Injecting into the pre-chamber augments the particle temperature because of the longer heating time [95, 107]. Moving the injection point far from the nozzle inlet may lead to strong particle dispersion, of which the detailed consequences, in particular experiment-wise, are unknown. CFD was also used to design more exotic devices, such as micro-nozzles in the context of internal surface deposition [116, 117], barrel-shaped nozzles [96], and CS assisted with liquid feedstock [84] or shock tubes [156]. These designs were validated in part by comparing coating properties (micro-structure, hardness) with a reference coating, or by particle in-flight measurements.

2.4.3 Experimental analysis

Most experimental work in the field of CS is associated with the coating itself, material combinations, and applications of the technology. In this domain, the flow is seldom directly measured and flow properties are often inferred from the coating result. This part of the field has a product oriented attitude and evaluates characteristics such as micro hardness, porosity, conductivity, and uses the deposition efficiency as an indicator of the adequacy of the flow; a natural approach if the researcher is mostly interested in the usability of the applied coatings. Making a connection to the previous paragraph, the topic of injection is a good example. Pattison [25] analysed a variety of injectors and measured, for instance, carrier gas flow rate and DE. Although a trend identifies an optimum flow rate for each injector location, it is impossible to know what mechanisms entail these results. The main problem is that the particle dynamics of the injection itself cannot be observed directly. One valuable consequence of a better understanding of the nozzle-internal particle motion, particularly with respect to the injection, might be the possibility to manipulate it, resulting in adjustable particle streams.

Apart from such indirect measurements, a vast set of studies followed a more causal attitude, yearning to find reasons and mechanisms for specific optima. Again, as most of the process is inaccessible to optical measurements, such experiments are restricted to the jet region, although the interesting processes are taking place within the nozzle. As a consequence, many concepts are only backed up by simulations, or can only be

investigated by them to begin with. As can be seen from the previous section, a great amount of detail of flow processes in the field originates from simulations with indirect or no validation. Optical measurements for such validation of employed models are possible downstream of the nozzle exit, as outlined in the following paragraphs. It is however some limitation that only the resulting outcome of physical mechanisms is observable, instead of direct observation of such. This may be adequate for cases in which acceptable agreement is obtained, but learning from mismatches is difficult and transferring findings to other geometries and parameters undermines model validity. Nonetheless, greatly important works have been carried out in this area to support findings of CFD. In this context, flows in CS were experimentally analysed by two main means, qualitative flow visualisation and quantitative measurement.

Flow visualisation

Visualisation of the gas phase is a useful technique to analyse flow features, which give implicit information about the gas state and are helpful to detect motion in colourless gases. Most popularly, flow visualization is realised by Schlieren photography, in which the variation of the refractive index of the gas with density gradients is exploited by virtue of a transfer into different light intensities that can be observed directly by cameras. Some works have realized the visualization of the CS supersonic jet; an example of the Schlieren image was already shown in figure 2.12, showing the shock and expansion waves and the compressed bow shock. Likewise, more Schlieren photographs can be found in literature [22, 46, 80, 103, 157], indicating the strength of shock waves, and qualitative changes with parameters of the set-up. A particularly interesting observation is the Schlieren photography of an internal nozzle flow in a barrel section with constant width, published by Katanoda *et al.* [158]. They visualised the flow pattern inside a low-pressure cold spray nozzle, exhibiting discontinuity reflections on the nozzle walls as shown in figure 2.18.

This work provided a reference for visualising the flow inside the conventional high-pressure cold spray nozzle, but no such study has been reported yet. Schlieren photography was successfully used to study effects of important parameters, *e.g.*, velocity and temperature, but an inherent limitation is the qualitative nature of the technique. Flows cannot be assessed quantitatively, therefore, in most cases, Schlieren photography was only used

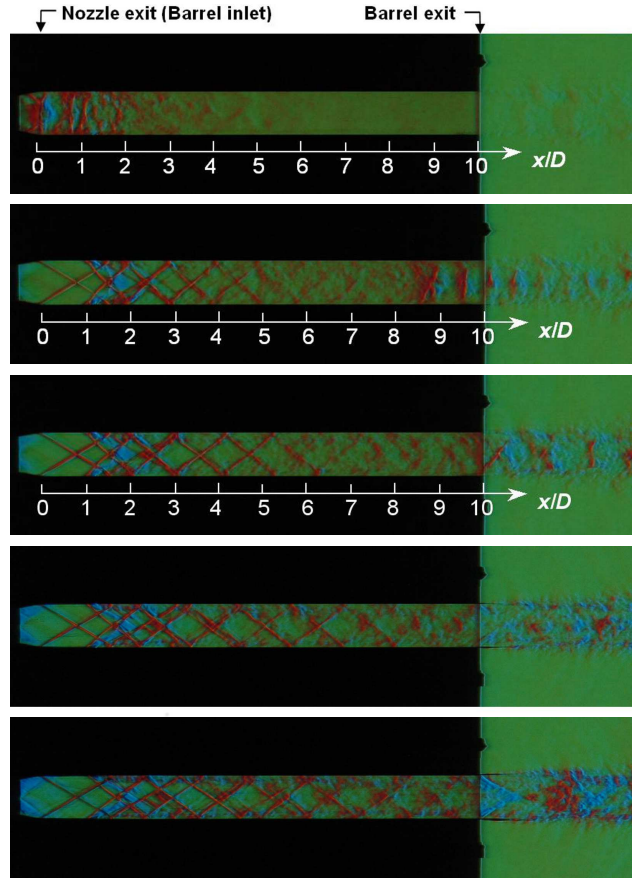


Figure 2.18: Schlieren imaging in barrel section of cold spray nozzle, visualizing structures at increasing stagnation pressures $p_0/p=2.0-7.8$ [158].

to validate CFD models pertaining to limited phenomena of the gas phase - the most important of which for this work is the shift and weakening of flow structures with particle loading (as discussed in section 2.4.2) by Samareh *et al.* [83]. It is interesting at this stage, that, based on these images and the constant-lag-interpretation, the notion of an effective, slightly decreased specific heat ratio due to particle loading was introduced: the Schlieren images reveal that a two-phase jet behaves like a single-phase jet of a hypothetical gas with an increased number of internal degrees of freedom, so that γ may drop by a few percent. Although the two-phase flow generally cannot be modelled as a continuous mixture, some gas phase effects can still be interpreted as such.

Velocity measurements

In order to quantify the particle motion behaviour and critical velocity in cold spraying, optical velocity measurement techniques have been employed. Over the past decade, the

non-intrusive methods can be categorised in three main approaches, namely, Laser-2-focus (L2F), doppler picture velocimetry (DPV), and particle image velocimetry (PIV). Detailed descriptions of the most widely used set-ups can be found in literature [76, 113, 159]. Figure 2.19 gives examples of particle velocity results in the jet using DPV and PIV. In an early stage, L2F was used to measure the particle velocity [21, 159], allowing for the measurement of single particle speeds, but due to the use of a limited volume, with very low spatial resolution. It was hence no longer applied in later works and replaced by DPV and PIV-based techniques. DPV is more advanced than L2F due to its capability to measure the particle velocity at higher spatial resolution. It works by constant illumination of the particle stream and detecting the signal delay produced by a particle passing in front of a two-slit photo mask. Like the L2F, DPV is therefore only suitable for single particle velocity measurements and can represent a spatial distribution by scanning the flow field by displacing the relative nozzle-sensor position and averaging over the separately recorded data. This may degrade the measuring accuracy and require a large amount of time.

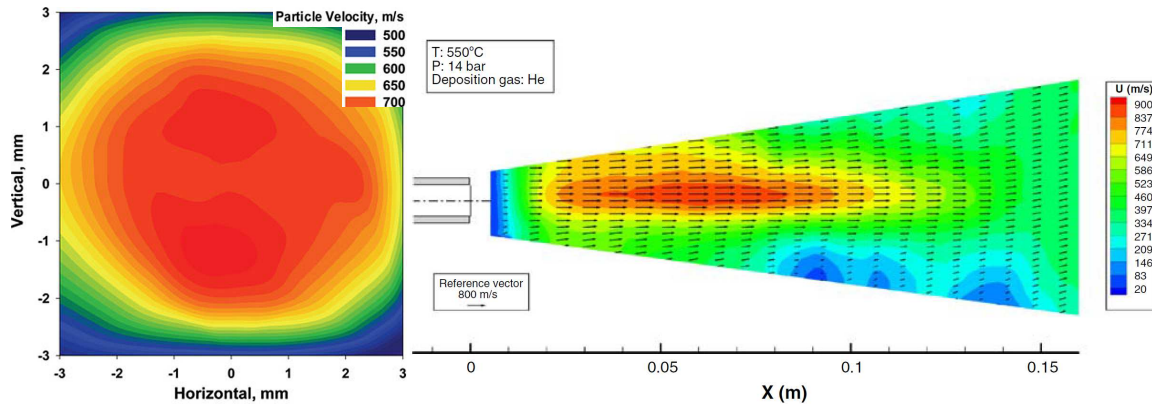


Figure 2.19: Particle velocity distributions - left: cross-section of jet by DPV [113], right: longitudinal section by PIV [160]

This is advantageous in PIV techniques, as they allow for the recording of the instantaneous velocity distributions with high spatial resolution, significantly increasing the measuring accuracy and reducing the experiment time. PIV is a method for the acquisition of velocity field data and is briefly described here based on established literature [161]. The particles are illuminated with two (or a sequence of) subsequent laser pulses, formed to a light sheet in the plane of measurement. A camera system captures images of the scattered light respectively, which are post-processed by a cross-correlation algorithm, obtaining the displacement of the particles or particle groups. By knowledge of the pulse

separation time, the displacement data can be interpreted as velocity information. This principle is illustrated in figure 2.20.

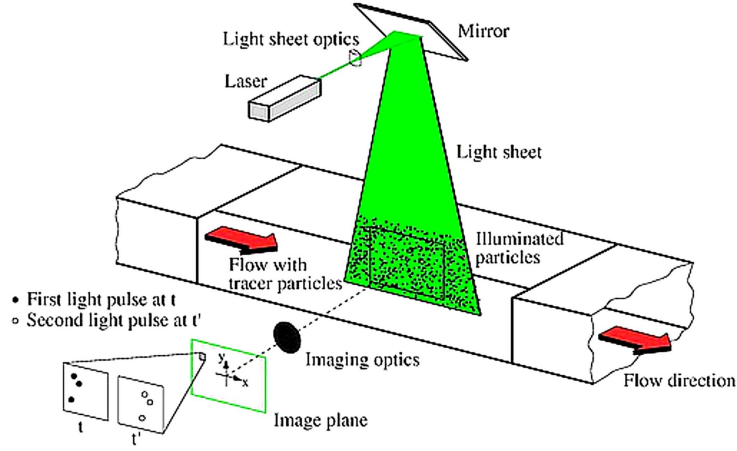


Figure 2.20: Principle of Particle Image Velocimetry [161]

In common PIV measurements, the images are divided into small interrogation windows (*e.g.* 32x32px), in which the particles are used to obtain a minimum quality of the cross-correlation peak. All particles within the window therefore participate to the same velocity vector, which makes a minimum number particles per interrogation window necessary to avoid any accuracy deterioration due to the in-plane and out-of-plane losses of particles. For each particle, the optimal image size for sub-pixel displacement accuracy is about 2 pixels, because random errors, originating from noise and algorithm, are minimised in this way and effects like “peak locking” (statistical tendency of the particle position to be biased to a full pixel position) do not yet occur. The non-uniform light scattering behaviour of the particles (Mie-scattering) is very important, as it depends on the particle reflective index, the surrounding medium, the wavelength and polarisation of light, and finally the particle shape. It can be detrimental if particles of strongly varying size and shape exhibit high differences in particle image intensity, causing a strong increase in the random error. This is the main weakness of the method, as it is sensitive to the particle morphology. Apart from aforementioned advantages, PIV can handle large fields of view and relatively high image densities, and can therefore be used for high loading cases in CS. Although it is difficult to assess the overall uncertainty of PIV due to a vast amount of relevant influences, it is usually in the sub-pixel range and therefore better than other techniques.

Having several sub-categories with slightly different principles, this basic PIV-approach to obtain a smoothed field of velocity data of the particle phase was applied in some cold spray projects [25, 46, 142, 160, 162–164]. In these studies, different powder materials, shapes, and sizes were tested and the respective conclusions pertaining to radial and axial particle velocity trends, changes with gas type and conditions, critical velocity prediction, and more, were generally in qualitative agreement with findings from CFD as outlined in section 2.4.2. An overview over the most prominent examples of particle velocity measurements and the respective findings is given in table 2.3, a collection that was published by the author in a similar form [121]. A second approach from the PIV-family is Particle Tracking Velocimetry (PTV), in which single particle displacements are identified instead of raster groups. This approach can be used more easily for low particle image densities [161], is thus particularly suitable for spray plumes, and was implemented in commercial packages. It was consequently applied often in CS [45, 76, 78, 145]. A similar method is used in this work, which is described in section 3.5. Another technique is to illuminate the particles continuously and to obtain “stripes” of particle tracks by a defined camera exposure time [117]; a method that has not asserted itself significantly in the field. PIV and PTV are very powerful as employed in these studies, but with respect to what detail may be observable in other flows, it is still limited due to the extreme speeds. The short time scales make equipment that can resolve the flow temporally unaffordable.

A study on a cold spray related investigation by Buchmann *et al.* [165] can demonstrate to the interested reader how an astigmatism technique with high speed cameras can be used to track individual particles three-dimensionally through the jet to the substrate, including a repelling reflection, see figure 2.21 for an illustration of the results. The particle acceleration was made observable by using a fully under-expanded (critical) nozzle instead of a typical CS nozzle and it was revealed that the flow structures (shocks and recirculation region) affect particle distribution and the impact angle of particles. Despite the vast amount of insights into cold spraying gainable from such concepts, they are not applicable for a wide range of studies due to the high cost equipment, the experimental effort, and the requirement for very narrow particle size distributions. Nonetheless, such individual tests may deliver high quality data for validation purposes. Some researchers also used another PIV category, which works with diffusely back-lit measurement areas, thus shadow-graphy

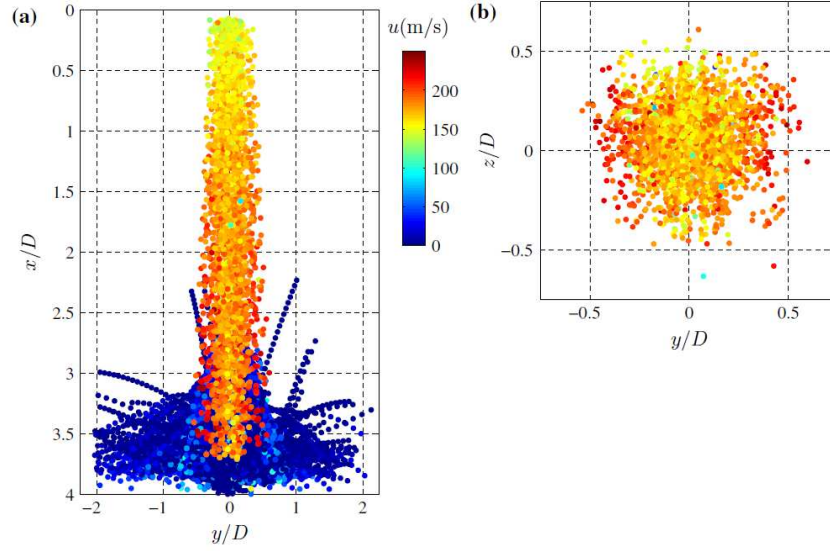


Figure 2.21: High speed 3D particle tracking results by astigmatism PTV [165]

techniques [81, 166, 167]. This version of approach offers the advantage of being able to infer the particle size from the shadows and to combine it with velocity information obtained from a single-frame tracking technique, which allows the investigation of size dependent particle velocities. Its main downsides are a restriction to moderate particle image densities and a relatively small field of view due to magnification constraints (which can result in larger uncertainty in the velocity data as compared to PTV).

Some results are to a degree contradictory, indicating that they are case-dependent, *i.e.* connected to the specific experimental set-up and conditions. Further investigations employing several methods are required for clarification and, when possible, more precise and detailed resolutions should be pursued. An example is the loading effect, discussed critically in connection to the respective modelling approaches in section 2.4.2 - for which literature provides experiments showing no effect [167], small but subjectively unimportant effects [27], or even significant effects [83]. Another example is the influence of the stand-off distance and respective explanations: Pattison *et al.* reported that titanium particle velocity increases as stand-off distance rose before dropping again [46], while Zahiri *et al.* differed by observing a velocity decrease for a very similar spray [164]. Both the changing trend [46] and the continuous trend [47] can be backed up by deposition efficiency trends, corresponding to particle impact velocity. It is possible that a combination of yet indeterminable effects give rise to this, *e.g.* differing nozzle designs that cause different

	Authors	Material	Shapes	Size [μm]	Conclusions
L2F	Gilmore <i>et al.</i> [21]	Cu	Spherical	19, 22	Particle velocity increased with increasing gas pressure and temperature Particle velocity decreased along radial direction Helium resulted in higher particle velocity than air Particle velocity decreased with increasing powder feed rate CFD model validation
DPV	Champagne <i>et al.</i> [113]	Al	Spherical	15	Particles footprints mainly concentrated at the central region
	Champagne <i>et al.</i> [168]	Cu	Spherical	...	Helium resulted in higher particle velocity than air
	Fukanuma <i>et al.</i> [169]	316L	Spherical	...	Gas temperature was more influential than pressure
			Irregular	...	Irregular particles resulted in higher particle velocity
	Legoux <i>et al.</i> [170]	Al, Zn, Sn	Spherical	36, 13, 10	Nozzle gun test
	Suo <i>et al.</i> [23]	ZK61	Spherical	58	CFD model validation
	Huang <i>et al.</i> [114]	Cu	Irregular	30	CFD model validation
	Ajdelsztajn <i>et al.</i> [171]	Fe alloy	Spherical	...	Particle velocity increased with increasing gas temperature
	Nickel <i>et al.</i> [156]	Al-12Si	Spherical	...	Critical velocity prediction
					Particle velocity increased with increasing gas temperature
					Low-velocity particles account for larger share than high-velocity particles
	Jodoin <i>et al.</i> [172]	Al alloy	Spherical	25, 28	Irregular particle resulted in higher particle velocity
		Al alloy	Irregular	25, 28	Particle velocity decreased with increasing particle size in the free jet without substrate
	Dewar <i>et al.</i> [135]	Al	Spherical	17-33	Particle size measurement
PIV					Initial conditions for other calculations
	Ning <i>et al.</i> [45]	Cu	Irregular	21-62	Particle velocity decreased with increasing particle size in the free jet without substrate
		Cu	Spherical	12	
	Pardhasaradhi <i>et al.</i> [167]	Cu	Spherical	33	Rectangular nozzle was better than circular one
		Cu	Spherical	104	Gas temperature was more influential than pressure
		Al	Irregular	35	Powder feed rate had negligible effect on particle velocity
		WC-12Co	Spherical	22, 56	Particle velocity decreased with increasing particle size and density in the free jet without substrate
	Pattison <i>et al.</i> [46]	Cu	Spherical	22	Particle velocity of Cu increased with increasing stand-off distance
		Al	Spherical	17	Particle velocity of Al maintained steady with increasing stand-off distance
		Ti	Irregular	21	Particle velocity of Ti increased with increasing stand-off distance
		Ti	Irregular	27	Helium resulted in higher particle velocity than air
	Wu <i>et al.</i> [145]				Particle velocity decreased along the radial direction
		Al-12Si	Spherical	25	Particle velocity increased with increasing gas pressure and temperature
					Particle velocity decreased along radial direction
	Jodoin <i>et al.</i> [76]	Ni	...	19	Particle velocity increased with increasing gas pressure and temperature
	Raletz <i>et al.</i> [78]	Cu, Ni	...	16	For critical velocity prediction
	Tabbara <i>et al.</i> [81]	Cu	Spherical	5-25	Particle velocity decreased with increasing particle size in the free jet without substrate
					Helium resulted in higher particle velocity than air
					CFD model validation
	Sova <i>et al.</i> [117]	Cu, Zn, Al	...	...	Micro-nozzle configuration validation
	Fukumoto <i>et al.</i> [142]	Cu	Spherical	5, 10, 15	Particle velocity increased with increasing gas pressure and temperature
					Particle velocity decreased with increasing particle size (measuring position unknown)
	Zahiri <i>et al.</i> [164]	Ti	Irregular	22	Particle velocity decreased with increasing stand-off distance
	Bray <i>et al.</i> [163]	316L	Spherical	22-36	High-velocity particles was mainly at the central region
					For validation of the CFD model
	Buchmann <i>et al.</i> [165]	...	Spherical	54, 110	Astigmatic tracking method for particle trajectory resolution in under-expanded jet
	Meyer <i>et al.</i> [173]	Al ₂ O ₃	Irregular	60, 30	Under low stagnation pressure, feed rate increase to 120g/min leads to velocity losses up to 14%, strongly material dependent, 2-way coupled model can calculate trend but not quantitatively accurate
		Stellite (St)	Spherical		Under high stagnation pressure, heavy materials lose momentum due to mass loading, light-weight materials additionally due to volume fraction.
	Meyer <i>et al.</i> [174]	Al,Ti,St	Spherical	30	(in addition to [173, 174]) Particle velocity measurements inside a (rectangular) quartz glass CS nozzle: two-way coupling and collisions have noticeable effect on particle dispersion and speed, especially for light-weight materials, increasingly for higher feed rates. Advanced computational methods can include such effects to calculate clogging risk, spot size, impact statistics.
	Present work	Al,Ti,St	Spherical	30	

Table 2.3: Summary of work regarding particle velocity measurements, originally published by the author in [121]

particle velocity gradients within the nozzle and different geometries of gas jet features that contribute to the particle behaviour downstream of the nozzle exit. Besides this, an interesting phenomenon is that the results obtained by DPV normally tend to be underestimated by CFD [23, 113, 114, 175], *i.e.* the measurements are higher than simulations. Conversely, PIV/PTV analysis, when accompanied by model results, are overestimated, *i.e.* their values are lower [81, 163]. Besides the possibility of a coincidence, it may have reasons within the procedure, for instance a bias of detected/undetected particles, experiment design, or insufficient error assessment. Future comparisons may reveal this, but the impression is that higher accuracy measurements need to be attempted in order to validate models and design components more reliably. Within a different scope, more experimental work about particle bow-shock interactions and particle in-flight temperature seem required.

2.5 Summary and research objectives

This review introduced the CS process, drawing attention to the importance of the two-phase nozzle flow, along with an essential minimum of supersonic nozzle flow fundamentals. Subsequently, a review of numerical and experimental flow analysis methods pointed out that both approaches have helped understanding the most important process parameters, such as stagnation pressure and temperature, and advanced nozzle designs with respect to gas flow and single-particle acceleration. The review concluded that many numerical investigations are weakly supported by validation: trends can be captured, but estimations lie inconsistently over or under measurements to varying degrees. This was attributed to a large number of differently adequate models, most importantly with respect to phase coupling aspects. The few studies that consider particle loading relevant report differing results: volume fractions are often in the order of 0.01-1%, which nearly always falls in a dense-dilute transitional regime for the inert particles in CS, and the corresponding mass loading values affect the momentum exchange between the phases.

Since experiments only indirectly validate the outcome of the actual phase interactions, there are limitations in the assessable model detail. It is not yet possible to computationally guide researchers through challenges in CS applications that are connected to the

nozzle-internal gas-particle flow: the deposition efficiency cannot be estimated based on CFD, the particle beam in the jet cannot yet be controlled, and in particular, the effect of nozzle clogging cannot be conclusively analysed numerically. A future advanced class of models should enable the assessment of such often-faced challenges, by offering currently impossible features:

- Higher accuracy and reliability estimations of particle velocity and temperature
- Statistically valid impact properties enabling derivation of deposition efficiency
- Possibility to calculate particle dispersion, impact angle and spray spot
- Development of nozzle designs compensating for loading and dispersion
- Prediction and prevention of nozzle clogging with respect to powder injection

This represents a long-term goal, for which this work aims to provide a necessary impulse. For this to be achievable, it is first required to gain better understanding of how the particles behave inside the nozzle, rather than observing the outcome of acceleration and the dispersion indirectly by measuring the jet properties. An indirect empirical approach could avoid measuring the particles inside the nozzle, but would not lead to physical insight and explanations of the observed behaviour. Moreover, the vast amount of influences onto the process would make a reliable correlation of findings to parameter ranges difficult, *e.g.* it would be almost impossible to cover a significant number relevant materials. For more general solutions with high potential of transfer to other systems and circumstances, physical explanations need to be provided, for which an in-depth investigation of the particle behaviour inside the nozzle is required. This leads to the necessity to set-up an experiment that enables nozzle-internal measurements, which is naturally difficult for such non-transparent devices. Although meaning a significant change to the layout of standard CS nozzles, a specific transparent nozzle is a possible solution. For selected circumstances, the particles could be characterised within this nozzle, which could in turn be exploited to identify mechanisms of particle behaviour and use these to develop computer models of strong validity. The data could be used to gather very new insights into nozzle design and to rethink the computational modelling.

This is the approach chosen in this work: the aim of this research project is to design a new experiment for nozzle-internal particle velocity measurements and use the novel experimental analysis to demonstrate the validity of an advanced computational model. In this respect, the following objectives are defined for the research project:

1. Gas-particle phase interaction mechanisms for conventional CS nozzles are still uncertain. Clarify loading effect by experiments with an application-tested CS nozzle. Extend current knowledge on the importance of gas-particle momentum exchange, on the particle dispersion, and on the influence of mass loading.
 - Analyse the result of increasing particle phase mass loading (ca. 0-30%) on the velocity range (ca. 250-450m/s), and on particle footprints.
 - Vary pressure conditions (up to 30bar) and materials (density between 2700-8440kg/m³), and identify characteristic differences.
 - Challenge established indirect approach to observe particles merely in the jet and define a new required approach.
2. Momentum and energy exchange occur within the nozzle, such that measurements at the impact zone do not provide understanding of how the particles reach their state. In order to overcome this limitation, investigate the conditions during particle acceleration directly. To this end, develop an experimental set-up for nozzle-internal measurements.
 - Construct a test rig for velocity measurements with optical methods on a Cold Spray system.
 - Design a visually transparent Cold Spray nozzle that can be used for imaging of the internal particles.
 - Integrate a Particle Tracking Velocimetry (PTV) system.
3. Measure and analyse particle behaviour within the nozzle and suggest physical mechanisms that explain the observations that following influences induce.
 - Identify the influence of the pressure level (0-10bar) on the internal particle acceleration and distribution.

- Compare the characteristic particle behaviour for low and high particle loading cases (0-30%).
 - Investigate how a change in material density affects the particle motion by changing feedstock material.
 - Vary the internal particle motion actively by changing the location of the particle injector needle in the converging section.
4. Develop a holistic computational model and demonstrate the new validation opportunities based on the internal experimental observations. Analyse the present nozzle flow computationally, determine the limits of the model and derive means for further model improvement.
- Develop a holistic computational model using *Ansys FLUENT v16.0*, including all features that are deemed required based on the experimental analysis, e.g. drag force, momentum coupling of phases, turbulence modulation, particle-particle collisions.
 - Apply gathered experimental data to case validation and compare increasing levels of model complexity; drag, coupling, turbulence modulation, particle collisions.
 - Analyse the present flow with respect to advanced challenges, such as nozzle clogging and particle dispersion and statistical distributions. Thereby establish and explain limits of the model's predictive capabilities by virtue of challenging the adequacy of the model details (drag-, collision-, turbulence model, coupling).
 - Suggest details of improvement opportunities for computational models by identifying physical mechanisms that are not covered by models, or by pointing out beneficial corrections for the employed models.

Chapter 3

Experimental methodology

3.1 Introduction

Models with capabilities that exceed the current state-of-the-art would be key to the computational estimation the particle statistics at impact, the calculation of a spray spot, and the analysis of particle dispersion inside the nozzle. Such models can eventually lead to new designs of nozzles, that can exploit the loading and dispersion influences and solve advanced challenges in CS applications, such as calculating deposition efficiency and avoiding or controlling nozzle clogging.

In order to achieve this, it is foremost necessary to understand more details of the gas-particle interactions in the different flow regimes, and then to use this knowledge to derive engineering measures with respect to modelling and consequently design. Recapitulating the final goal introduced in section 1.1 and the detailed objectives defined in section 2.5, the achievement of the following intermediate goals will set foot on this path:

- Gas-particle phase interaction mechanisms for conventional CS nozzles are still uncertain. Clarify loading effect by experiments with an application-tested CS nozzle and refute the sufficiency of current measurements for this application case.
- Momentum and energy exchange occur within the nozzle, such that measurements at the impact zone do not provide understanding of how the particles reach their state. Realise a new measurement method to directly observe particles nozzle-internally.
- Measure and analyse particle behaviour inside the nozzle under varying conditions

by identifying important effects that are so far neglected in models.

- Develop a holistic computational model and demonstrate the new validation opportunities by determining limits of the model and deriving means of improvement.

Systematically pursuing these goals, a new CS rig is required that can integrate a PTV system and CS nozzles, the actual transparent nozzle needs to be designed and manufactured, and specific powder injectors are due for the new type of nozzle. This chapter will introduce this equipment, explain details of its design, and assess the quality of the experimental data.

3.2 System for Cold Spray velocity analysis

In the following sections, the general arrangement of the experimental equipment is described. The respective components and devices were available and did not have to be designed for this work, with only small adaptations required.

3.2.1 General set-up

The Cold Spray System is of in-house design and was developed in a row of projects prior to this work. Figure 3.1 illustrates a schematic of the experimental arrangement. The gas is fed from a high pressure gas supply line to a set of two regulator valves, one for the main gas line (process gas) and one for the feeder line (carrier gas). Both lines comprise individual pressure gauges and regulators for variable setting of the overall flow rate and their flow ratio r . In addition, they possess individual flow meters to quantify this ratio.

$$r = \frac{\dot{m}_{c,feederline}}{\dot{m}_{c,mainline}} \quad (3.1)$$

The feeder line merges into a wheel-type powder feeder. Under well-set conditions, the carrier gas in the feeder line and the particles form a homogeneous mixture, which is then merged with the main flow downstream of the powder feeder in the nozzle head. This injection is of axial type with a central injection point of variable position along the longitudinal direction of the nozzle, henceforth denoted as x-axis. The main line gas is introduced into the nozzle head with a slight offset. An additional pressure gauge

is positioned within the head to assure measurement of the nozzle inlet pressure. Both streams, from main and feeder line, enter the nozzle that is attached to the head, and subsequently eject from it into the container. This study does not involve deposition analysis and hence no substrate is placed in front of the nozzle, excluding the interaction of the gas-particle jet with an obstacle.

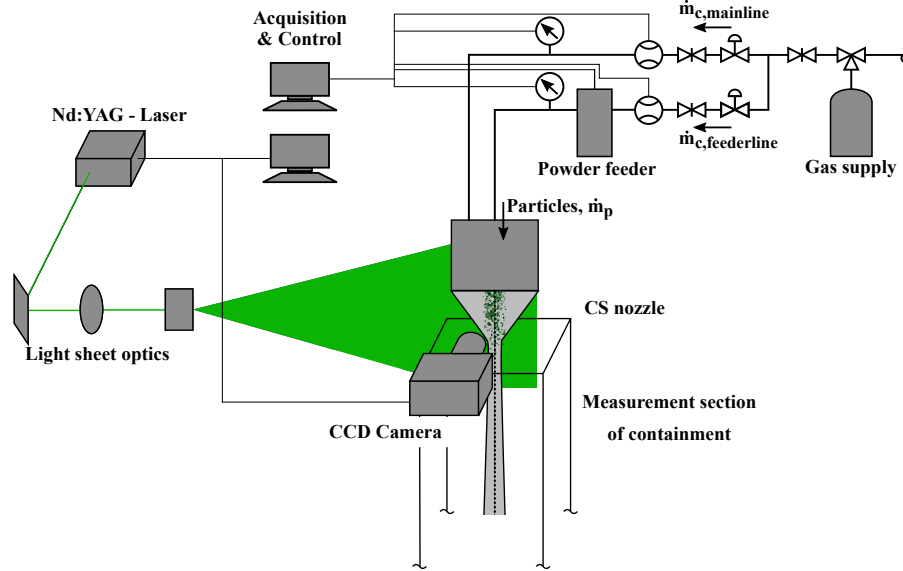


Figure 3.1: Schematic of CS process set-up and measurement arrangement

3.2.2 Gas supply, flow meters and gauges

The gas supply is a manifold of ten standard bottles of nitrogen (N_2) gas, each with an approximate fill volume of 10.5m^3 and an initial pressure of 230bar. Independently of the changing pressure level of the manifold under operation, a constant 50bar-g is regulated in the supply line before entering the laboratory, assuring continuous flow conditions.

The flow meters integrated in the CS system are *Brooks* SLA5863 devices. They are thermal mass flow measurement systems, which consist of a restrictor and a flow sensor. A pressure differential across the restrictor causes a portion of the gas to flow in the sensor and, because they have the same linear pressure drop to mass flow relationship, the ratio of the two flows remains constant. The flow sensor itself comprises two temperature sensing elements upstream and downstream of a heating element. Constant power is applied to the heater, such that with no flow present, the amount of heat reaching each temperature sensors is equal. Gas flowing through the tube causes a convective asymmetry and imposes

a temperature difference between the two sensors. The difference can be expressed as directly proportional to the gas mass flow according to $\Delta T = A \cdot P_{el} \cdot c_p \cdot \dot{m}$, a convention specified by the manufacturer, where A is introduced as proportionality constant in the units of $[\text{s}^2\text{K}^2/\text{kJ}^2]$, and P_{el} is the power input. This temperature difference is amplified and interpreted by a bridge circuit, producing an electrical signal directly proportional to the gas mass flow rate. The mass flow meters are configured by the manufacturer for Nitrogen and the signal has an output unit of standard litres per minute (slpm) with a range of 100-2500slpm. In this volumetric unit, the reference standard conditions are a temperature of 0°C and a pressure of 1atm. The devices are secured by the manufacturer to be accurate to $\pm 1\%$ of the measured rate for operation above 20% of the full scale, and to $\pm 0.2\%$ below it. Due to limited linearity of the accuracy, the operation in excess of 1200slpm is covered by an accuracy of $\pm 1\%$ of the full scale, however such flow rates are not reached in this study. The repeatability is within the range of $\pm 0.2\%$ of the measured rate.

The used digital pressure gauge sensors are *Gems Sensors* 3000Series Compact High Pressure Transmitter with a measurement range of 1 to 40bar. They rely on the piezo-resistive effect, which changes the electrical resistivity of a semiconductor or metal under mechanical strain. Increasing pressure causes a diaphragm and the gauge to deform which affects its resistivity. This change is converted into an electrical signal proportional to the pressure by a bridge circuit. The sensors are calibrated by the manufacturer, specifying tolerances of $\pm 0.5\%$ of the value span for the calibration limits. The measurement accuracy is within $\pm 0.25\%$ of the full measurement scale, *i.e.* $\pm 0.1\text{bar}$. There is an additional $\pm 1\%$ thermal error over 100°C, which, however, does not apply in this study, as the gauges are operated at room temperature. One analogue pressure gauge is integrated at the nozzle head, in order to control the pressure loss from the powder feeder to the nozzle head. The used type, a *WIKA* 7203556, relies on bellow deformation with a measurement range of 0 to 10bar. It is accurate to $\pm 2.5\%$ of the full scale, *i.e.* $\pm 0.25\text{bar}$.

3.2.3 Powder feeder and load cell

The powder feeder in this CS set-up is a *Uniquecoat* HP-PF100WL. This feeder is of wheel-type, and as such, delivers powder particles to the flow of the carrier gas by continuously

transporting parcels of material over a spinning wheel and dropping them into the stream. The powder reservoir and the delivery side of the feeder are connected to equalise the pressure level and hence avoid disturbing gas flow. The particle is approximately volumetric, but the delivery depends on the pressure conditions, the flow-ability and material density of the powder, the dimensions of feeder tube and wheel. Adjusting the particle feed rate in this system is done by varying the rotational wheel speed, set in units of the percentage of the maximum wheel spindle speed. It should be noted that the set variable is therefore the wheel speed, not the feed rate directly. Since there is no fixed relation between the wheel speed and the delivered mass of particles, the mass feed rate of the solid phase can then be tracked by an integrated load cell. The load cell weighs the whole set of powder reservoir and feeding unit, such that a settling time during start-up of the system is required while pressure builds up.

When the flow rates and the pressure in the feeder are constant, the wheel can be started and deliver powder into the stream. In lack of manufacturer data on the load cell accuracy, the device was validated by the following procedure. Under constant pressure and flow rates, alumina powder was sprayed for 120 seconds. This powder was captured in the containment downstream of the nozzle. The mass of this powder was measured with a precision scale (*Kern&Sohn GmbH* PFB3000-2, readout 0.01g). From this, an average feed rate over the time span is obtained, which is compared to the recorded data of the integrated load cell. This procedure was repeated three times for three wheel speed settings of 10, 20 and 30%. The observed differences lie within a range of $\pm 2\%$ of the measured feed rate values.

Because the mass loading, volume ratio, and velocity are deduced quantities, the propagation of the error needs to be considered. While the velocity uncertainty is analysed in detail in the later section 3.5, the mass loading and volume fraction are directly connected to the amount of fed particles and hence discussed here. Remembering, that the mass loading Z and volume ratio F are

$$Z = \frac{\dot{m}_p}{\dot{m}_c}, \quad \text{and} \quad F = \frac{\dot{V}_p}{\dot{V}_c} = Z \cdot \frac{\rho_c}{\rho_p}, \quad (3.2)$$

both the accuracy of the load cell and of the flow meters play a role for Z as well as for F the additional density. Since the relations are products and quotients, their relative errors

add. According to this, the mass fraction relates to

$$\frac{\Delta Z}{|Z|} = \frac{\Delta \dot{m}_p}{|\dot{m}_c|} + \frac{\Delta \dot{m}_c}{|\dot{m}_c|}, \quad (3.3)$$

if Δ indicates the absolute uncertainty range of each quantity. Using above values, we obtain a maximal uncertainty of 3% for the mass loading. While the particle density ρ_p is constant, the gas density ρ_c is a function of pressure. Being proportional through the ideal gas law, the pressure uncertainty can be substituted and the uncertainty of F is associated with

$$\frac{\Delta F}{|F|} = \frac{\Delta Z}{|Z|} + \frac{\Delta p}{|p|}, \quad (3.4)$$

Since the pressure gauge uncertainty is absolute, the relative error depends on the different operation points and is calculated during processing of the data sets. Naturally, the highest error occurs at the lowest pressure, which in this study leads to the maximum uncertainty in F of 6.13%.

3.2.4 PTV integration and data acquisition

As can be seen in above figure 3.1, the CS system was merged with a PTV system. It is discussed in detail in the following section 3.5, where the velocity measurement principle is introduced. It comprises essentially a *NewWave SoloII-15* PIV Nd:YAG laser, a *PCO SensiCam SuperVGA* CCD camera and light sheet forming optics. These devices are installed on a measurement rig, which allows for proper adjustment of all parameters and secure positioning. Figure 3.2 displays the experimental arrangement and illustrates the location of the respective components on the rig. This figure also shows the transparent quartz nozzle, which is discussed in section 3.3 to follow. A computer with a programmable timing unit (PTU) to control the camera and laser sequencing and respective measurement data acquisition software (*DaVis v7.2*, *LaVision GmbH*) is part of this system. More detail of the measurement system can be found in section 3.5. An additional data computer is connected to the sensors of pressure gauges, flow meters and load cell. This PC handles data signals of diverse types (*LabView*, *National Instruments Ltd.*), and in particular allows to capture data of the pressure and flow sensors with a rate of 50Hz and of the load cell with a rate of 2Hz in comma separated value format. In post-processing, the data

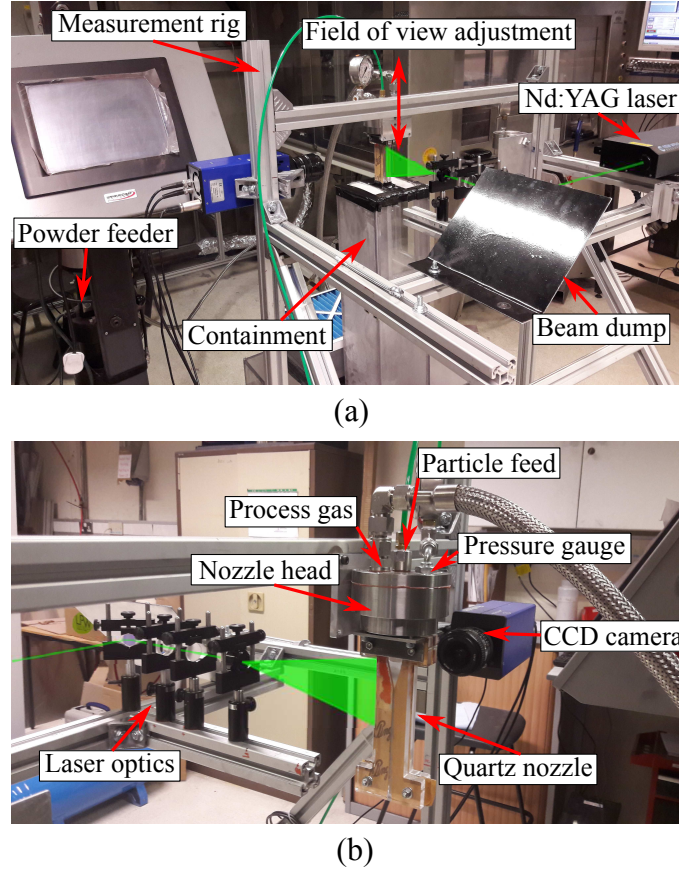


Figure 3.2: Experimental arrangement from two sides (a) and (b) illustrates the merged quartz nozzle CS and PTV systems

from the different acquisition systems (Flow & PTV) are re-formatted and joined before evaluation.

3.2.5 Post-processing

This evaluation is done by means of custom *Matlab* routines, executed in sequence. The particle velocity data exported from *DaVis* and the flow data exported from *LabView* are independently imported into this analysis environment. According to manufacturer calibration reported above, the following quantities are present as time series in *LabView*: Pressure in [bar], flow rates in [slpm] and mass in [kg]. When imported into the *Matlab* environment, the sensor data is arranged in object-like structures for the different test cases. The data time series are cropped according to those sequences, in which velocity measurements are conducted, *i.e.* in which PTV images are actually captured. The units are converted to SI. Due to the standard conditions in the flow rate unit [slpm], conversion

to [kg/s] uses $p_{St}=101325\text{Pa}$ and $T_{St}=273.15\text{K}$ in

$$\dot{m}_c[\text{kg/s}] = \dot{m}_c[\text{slpm}] \cdot \frac{1}{60000} \cdot \frac{p_{St}}{RT_{St}}. \quad (3.5)$$

The data is tested for time-invariance, representing steady-state operation. The steady state is here checked by an arbitrary small value for the gradient of the linear regression line through the feeder pressure data normalised by its average, as this represents the quantity that adjusts the slowest after start-up.

$$\frac{d}{dt} \left(\frac{p_{regression}}{\bar{p}} \right) < 10^{-4} \quad (3.6)$$

The feed rate as well as the mass loading are deduced from the signals and added to the selection of data. Again, the slope of a regression line is used to infer the feed rate from the powder mass.

$$\dot{m}_p = \frac{d}{dt} (m_{p,regression}) \quad (3.7)$$

Subsequently, a second re-organisation of the object structure assures that repetitions of test cases with the same parameter settings are treated independently, preparing the merging of velocity data into this structure. After joining the data streams, a thorough check of the signals is carried out manually, in order to eliminate potential data flaws. A correction of the coordinates due to shift in field of view is also performed. At this stage, an error analysis according to the above described logic is conducted and the respective values added to the data structure. A multitude of routines were developed in order to analyse the average trends, the velocity distribution in several locations and directions, the particle dispersion angle and statistical evaluations of probability density functions. An overview over the routines and the respective workflow can be found in the appendix (p.226).

3.3 Component development

While previous sections introduced available equipment, the following sections discuss the development work on the experimental set-up and therefore the critical components that were generated specifically for the present study.

3.3.1 Measurement rig

An experimental rig is designed to combine the CS and PIV system and position all devices appropriately. Aluminium struts form a basic construction, most efficiently using laboratory space. This type of system is stable and vibrations due to the operation of the system are minimal. Most importantly, the nozzle head, powder containment and laser system are combined in a secure manner. The optical components must be positioned in an adjustable manner, such that they can be used to optimise the laser beam and adapt the field of view. This was realised by the transverse adjustment of the optical components on the respective horizontal profiles and vertical displacement of the nozzle-head profile. At the same time, the CS nozzle is mounted to the nozzle head such that the camera field of view, the laser sheet, and the measurement plane of interest coincide. For a minimum of interaction with the surrounding environment and occupied space, the gas jet is oriented downwards.

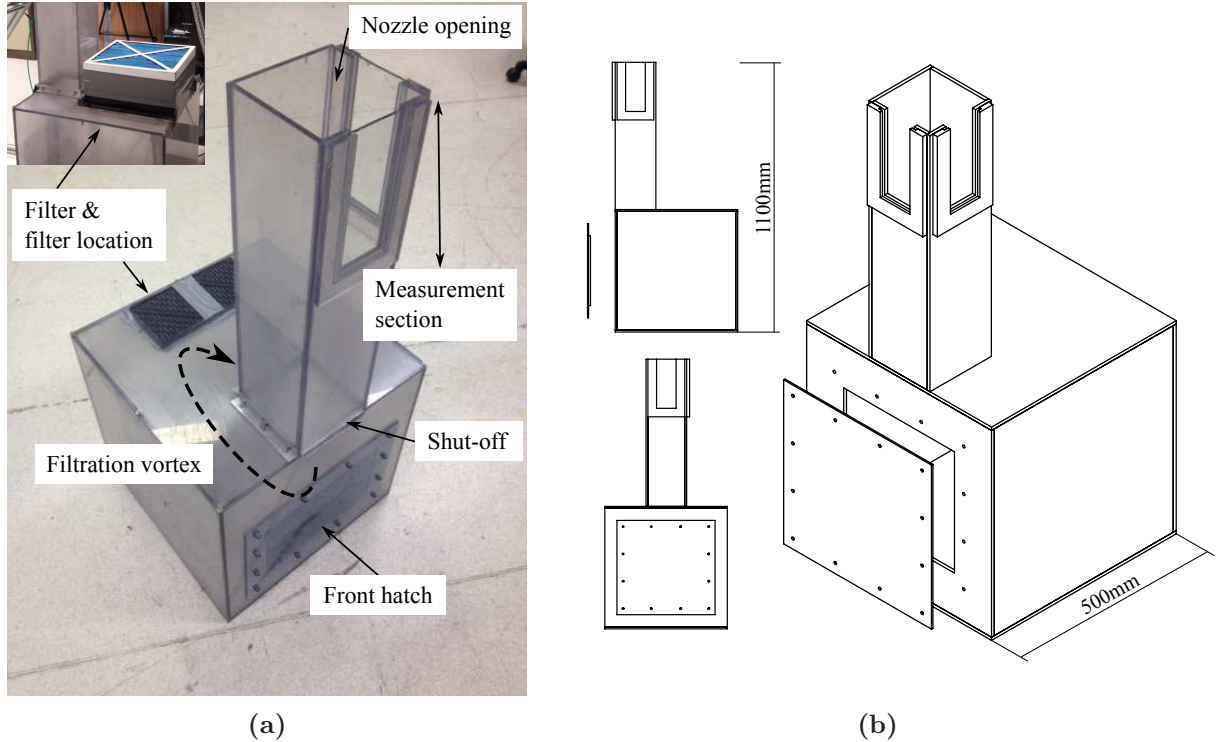


Figure 3.3: Measurement section and containment box assembly

Downstream of the nozzle, the container shown in figure 3.3 meets several purposes. Firstly, the containment device secures and shields the measurement instruments from the powder and prevents contamination. Secondly, due to its size the entire rig can be

repositioned within the laboratory, simplifying handling (exchanging nozzle parts, repositioning laser and camera, changing fields of view, etc.). Thirdly and most importantly, the containment has a measurement section, such that optical measurements can also be conducted in the jet downstream of the nozzle exit through provided glass windows. Being removable, they simplify cleaning and accessibility of the nozzle in the assembled state. Filters seal off the powder phase from the container to the environment both on the back side and in the nozzle co-flow. The back-flow of gas and particles into the measurement section from the lower containment chamber is minimised by redirecting the flow towards the filters and producing a vortex inside the containment box. A front hatch secures access for collection of the sprayed material, and a shut-off hatch can be closed at the connection of measurement section and containment box during nozzle assembly and cleaning.

The nozzle head is required to hold the nozzle in a seal-tight manner, merge main and feeder line by providing space for the injector needle, and make space for sensors available. In this study, the nozzles are rectangular in cross-section, which imposes the same exit-opening shape requirement on the nozzle head. Consequently, a head of circular basic shape is designed with a lid, which has a throat for the injector in the centre and for the remaining components with some off-set. The rectangular outlet has a shoulder for the contacting nozzle face. Drawings are appended (p.227).

3.3.2 Transparent nozzle

Overview

Because it is still unclear how particles and gas interact in detail within conventional CS nozzles, the first step in the experiments is to investigate the particle loading effect for an application nozzle. The chosen nozzle is denoted NZ1 and is a CS nozzle that was developed in-house by Prof. Lupoi prior to this work. Details on the nozzle shape are given in the following paragraph. It was designed for application of titanium and aluminium coatings and successfully used for depositions of a multitude of material combinations. This application-tested nozzle is also the template for the transparent nozzle construction and for this reason introduced at first. Subsequently, the derivation of the transparent nozzle is reported in detail, pursuing the following conceptual strategy.

Like all CS nozzles, NZ1 is an opaque nozzle, which prevents optical flow measurements

within it. In order to enable such observations, a clear material needs to be chosen to manufacture a transparent nozzle, denoted as NZTR. Quartz glass was selected for this purpose, which imposes some limitation on the construction of such a prototype. Firstly, the nozzle must have a shape that is manufacturable in this material and make the capturing of images of particles possible. This leads to a rectangular cross-section of the nozzle channel, that can be obtained by an assembly. The parts of this assembly ought to be sealed against each other. Secondly, a quartz nozzle of such an assembly is structurally not strong enough and the sealing not sufficiently tight to realise operation at the higher pressures used in CS. In order to keep these novel observations controllable and safe, the pressure was therefore limited to 10bar, which is a low pressure for CS, but still relevant for several applications. This pressure level dictates the geometrical dimensions of the nozzle. The cross-section of the restriction was maintained from NZ1 for adequate flow consumption, while the expansion ratio was adapted to the new pressure level. The nozzle length was shortened from NZ1 to NZTR, as the lower expansion ratio will cause a fully developed flow after shorter distances. Because the magnitude of the maximum achievable velocity is not of central interest, rounding the respective value off for better manufacturability was deemed to be an acceptable compromise. The shape was altered

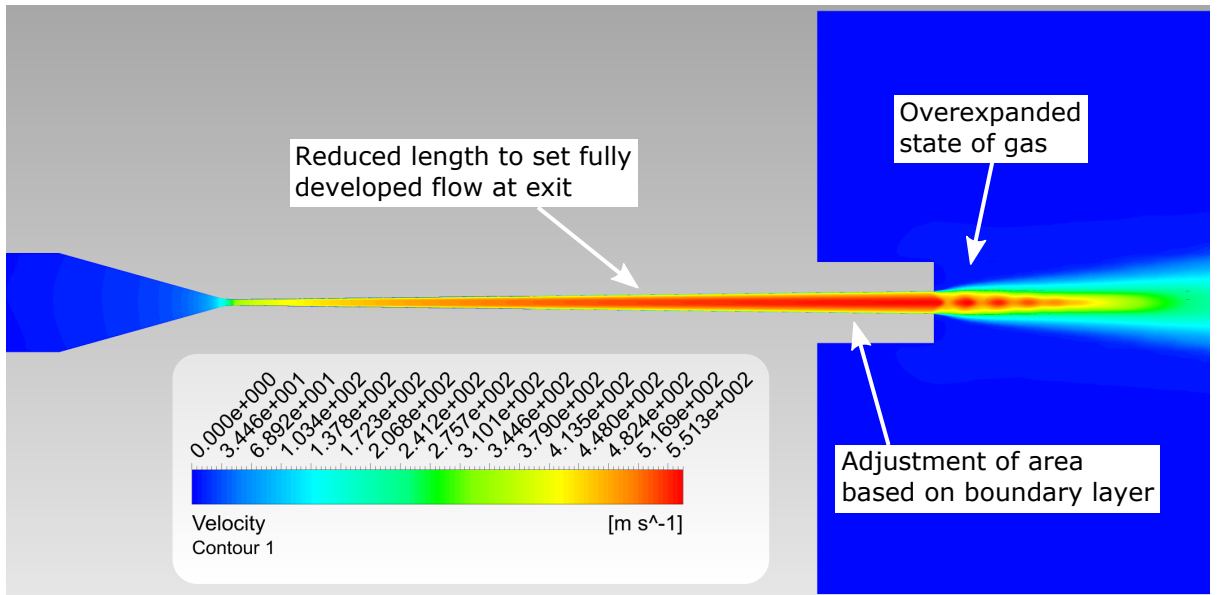


Figure 3.4: CFD optimisation of transparent nozzle NZTR after deduction from application nozzle NZ1, length and cross-section are varied for the gas phase to exhibit the present shock structure for 10bar pressure

with the use of 1D-relations as well as CFD in 2D and 3D, until the length and expansion state could be confirmed as an adequate solution, see figure 3.4. Details of the resulting shape and function of NZTR is reported in the section to follow an introduction of NZ1.

Application nozzle NZ1

This nozzle NZ1 is of in-house design (not part of this work) and manufactured from tungsten-carbide. Both converging section and diverging section of NZ1 have linear wall contours of length l_c and l_d , which are connected by a smooth throat radius r_{tr} . NZ1 is axisymmetric and has an expansion ratio

$$ER = \frac{A_e}{A^*} \quad (3.8)$$

suitable for pressure levels in the order of 20-30bar. Figure 3.5 illustrates the shape of the application nozzle NZ1 with its main geometrical parameters. A technical drawing of NZ1 is appended (pp.227). As a first step in each iteration of the shape derivation

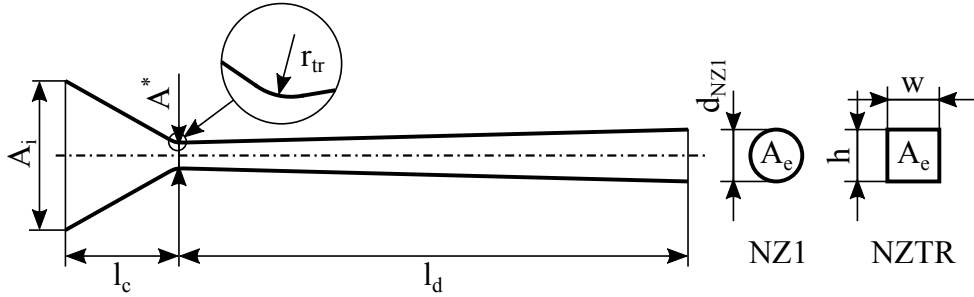


Figure 3.5: Schematic of application nozzle NZ1 and the deduced transparent nozzle NZTR

process for transparent nozzle NZTR, these basic dimensions of NZ1 were altered. Using the cross-sectional area, the circular shape of NZ1 and the rectangular shape of NZTR can be preliminarily compared via the idea of a hydraulic diameter.

$$d_{NZ1} = \frac{2wh}{w + h} \quad (3.9)$$

Here, d_{NZ1} is any cross-sectional diameter of NZ1, and w and h the width and height of the cross-section of NZTR respectively. This way, NZ1 functions as the basis for the transparent shape. Table 3.1 gives an overview over the change in the layout values.

Different than NZ1, NZTR has the shorter diverging section as explained above, and exhibits a lower ER , both due to the higher pressure level. Consequently, the converging length l_c and the cross-sectional areas at inlet A_i and A^* are the same. In contrast, the throat radius was reduced to accommodate to the change in length and exit cross-section A_e was reduced to adapt the expansion ratio.

Nozzle	A_i	A^*	A_e	ER	l_c	l_d	r_{tr}	Shape
NZ1	314	3.1	28.3	9	30	180	20	circular
NZTR	314	3.1	11.68	3.72	30	120	10	rectangular

Table 3.1: Dimensions of transparent nozzle design NZTR, compared to the application nozzle NZ1. Areas in $[mm^2]$, length in $[mm]$

Material of the NZTR assembly

The derivation of NZTR iterated through a process of finding adequate solutions for the following aspects:

- Choice of nozzle material
- Manufacturable shape
- Reconciling with imaging technique
- Structural strength and leakage

In this list, the emphasis is on the search for a suitable transparent material for the nozzle and the shape definition, since the remaining aspects are strongly linked to defining the shape. A simplified CS nozzle manufactured from polycarbonate and a grit-blasting nozzle from acrylic show that the inner surface instantly starts to erode when powder is fed, even at pressures below 10bar. This change in surface quality prevents imaging of particles inside the nozzle and rules out such soft materials. Apart from hardness, a second influence of the choice of material is the clarity of the material, as it contributes to the diffraction and aberration of light rays falling through it and therefore the accuracy of the imaging method [176]. Quartz glass, being of much higher purity than other glasses, has excellent properties in this respect, as well as high hardness, and is therefore the most favourable choice.

Geometry

Before defining the exact dimensions of the nozzle, a conceptual idea for the measurement operation and hence the nozzle assembly must be defined, as it imposes limitations of use and sizing onto the part. In order to be able to manufacture a nozzle from quartz glass, in which particle images can be recorded, a fabrication strategy is required. A round nozzle shape as in NZ1 is not realisable, due to manufacturing of the brittle material in such a long and narrow channel. Moreover, the curvature of the glass wall would additionally impede on the imaging, as light would get distorted falling through the curved walls. However, an assembly from sheet-like components can not only be manufactured, but also offers the possibility to exchange individual pieces and capture images of undistorted particles. This idea gives the flow channel a rectangular cross-section, enabling the use of PTV (illustrated in more detail below). It allows for a decomposition of the geometry into the four sheet-like pieces, that are displayed in figure 3.6. These components are two main blocks for mechanical support and imaging, as well as two contour- and illumination blocks. Being relatively straight-forward in shape, they are manufacturable by milling and can be provided with sufficient quality of (mechanically and fire-)polished surfaces. These quartz glass products were produced by *SemiQuarz GmbH, Germany*.

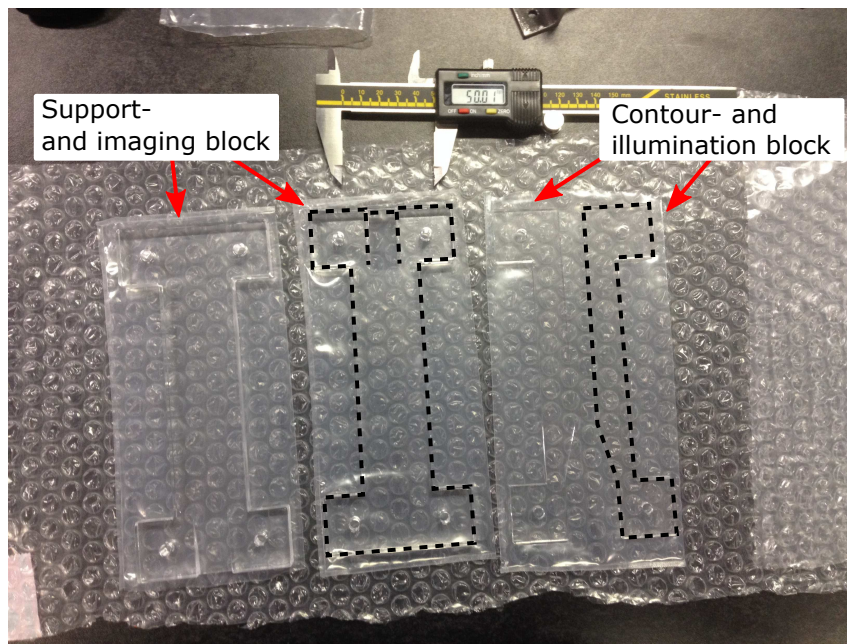


Figure 3.6: Disassembled four main nozzle components, two main blocks for mechanical support and imaging, as well as two contour- and illumination blocks

This assembly makes the intended measurements possible. It can however not bear a mechanical load equal to CS application nozzles. Its structural strength can be improved by increasing the thickness of such sheets, but this is limited by the resulting image quality and the expenses for manufacturing the components. Without experience with such a nozzle assembly, the safe operation at high pressures cannot be guaranteed. In addition, sealing the components against each other becomes increasingly difficult, as seals would require geometric features on the glass component, such as notches. This again is in conflict with the manufacturability and reduces stability even further. Based on experience values and without in-depth analysis, nozzles with a similar shape were produced from polycarbonate. Their operation was in fact possible at 9-10bar-g pressure. With these example nozzles of a material that deforms more easily, a similar pressure range (9-10bar-g) was deemed appropriate. Although a more detailed FEA analysis may have helped quantifying this process, the final experiments within this study confirmed that the assembly starts leaking and the nozzle components can crack beyond the defined pressure range. This pressure level, in combination with the rectangular shape, now defines the flow channel geometry, as described in the following paragraph.

At first, the expansion ratio is derived from NZ1 for the new pressure level by one-dimensional nozzle relations. It is subsequently modelled in a 2D-axisymmetric manner, using simplified CFD methods, and adapted for viscous effects - securing a slightly over-expanded exit state of the gas at 9bar-g. The nozzle length is shortened from NZ1 to NZTR, such that the centreline of this theoretical axisymmetric nozzle does not start to exhibit a velocity limit due to fully developed flow, as this represents an inherent limit to the capabilities of the driving gas. Rounding off leads to the final diverging section length of 120mm. This shape is transferred from the circular to the required rectangular cross-section with a constant width via the hydraulic diameter (eq. 3.9). The width w is thereby varied in order to reconcile the following points: firstly, the cross-sectional areas of the circular lay-out remain unchanged; secondly, the injector needles must be able to be well-positioned within the channel; thirdly, the nozzle exit has low aspect ratio. The latter is important, because it produces an appropriate jet: compressible rectangular gas jets of aspect ratios close to unity spread similarly to round jets [177] and have similar turbulent characteristics [178].

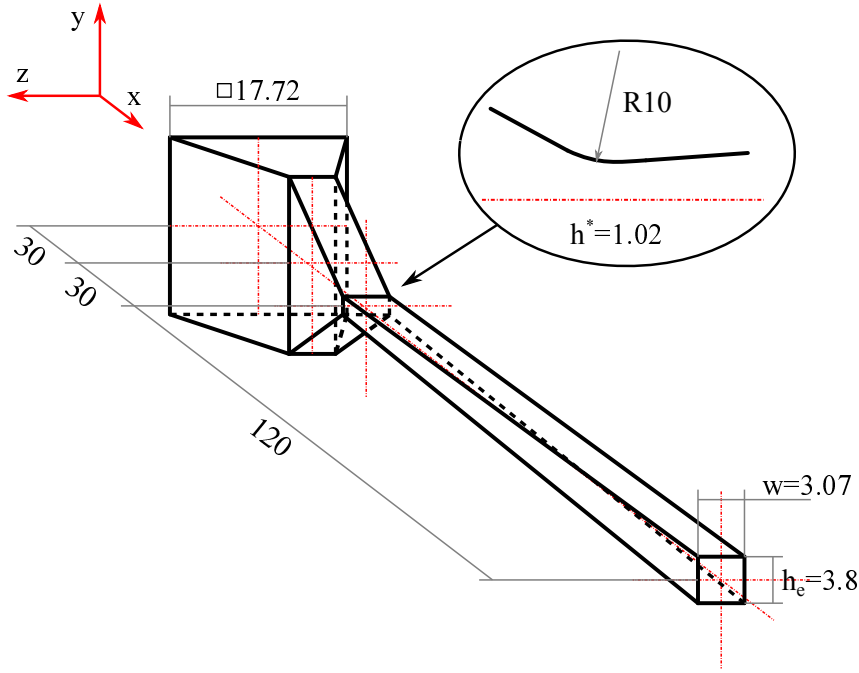


Figure 3.7: Geometry of the quartz nozzle flow channel, all dimensions in [mm].

For flexibility in handling, the outlet of the nozzle head and hence also the inlet of the nozzle channel are defined as squares. Therefore, the first nozzle part narrows this square down to a rectangle of the desired width w : the de Laval shape has a full length (x-direction) of 150mm, of which the converging section incurs 30mm. The constant nozzle width (z-direction) is $w = 3.07\text{mm}$, the throat height (y-direction) $h^* = 1.02\text{mm}$ and the exit height was $h_e = 3.80\text{mm}$. Manufacturing tolerances for the individual nozzle parts are $\pm 0.1\text{mm}$, however, the uncertainty of the nozzle dimensions depends on the assembly and is discussed in the following section. These dimensions of the flow channel are shown in figure 3.7. As introduced here, the x-coordinate specifies the longitudinal distance from the nozzle inlet, the y-coordinate is directed in the measurement plane, and the z-coordinate results perpendicularly. In the following, cross-sections hence always refer to a y-z-section, longitudinal sections always to the x-y-plane.

Assembly

When the components are assembled, it becomes clear how the concept of the measurements ties in with the shape definition of the nozzle. Drawings of the final component versions are appended (pp.227). The assembly is shown in figure 3.8.

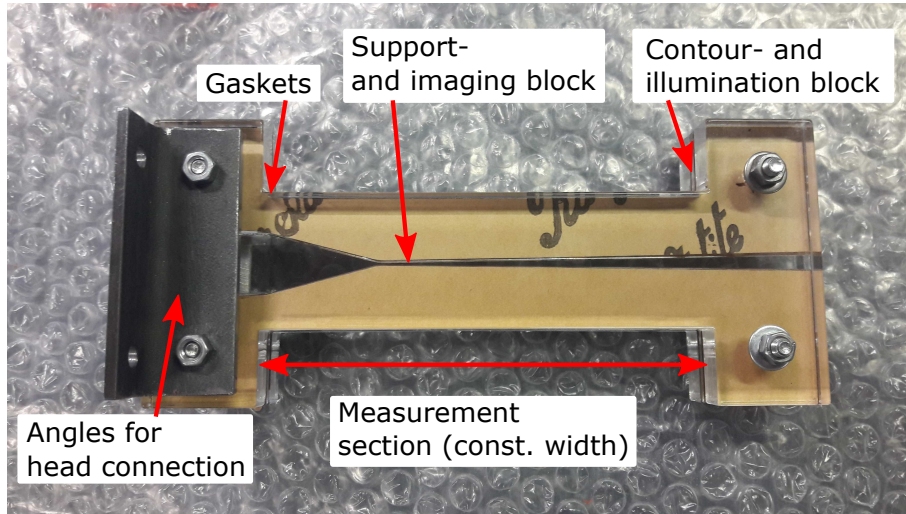


Figure 3.8: Assembly of the quartz nozzle components with sealing, measurement section and fixture angles

At the defined pressure level, it is possible to seal these four components against each other using paper-based gaskets (*Klinger* Statite 0.5mm). The parts are fixed by four bolts and, under operation, two additional clamps at the mid-points in x-direction. With this fully functioning set, no leakage was detected in the operated pressure range. The figure also shows fixture angles that are used to assemble the nozzle to the nozzle head. The connection of the nozzle inlet side and the respective shoulder surface of the head is sealed with a silicone sealing paste.

As introduced above, the converging-diverging wall contour is defined by the contour blocks in one dimension. The flat nozzle wall of the support blocks in the other dimension functions as the window, through which undistorted images can be recorded. For this recording, particles streaming through the nozzle are illuminated by a light sheet that enters the nozzle from the side, centred on the plane of symmetry. The nozzle can be shifted on the measurement rig to adjust the field of view (FoV), as shown in figure 3.9. This way, the different injection locations are observable (marked in figure) and particle velocities are measurable throughout the nozzle, with the exception of the very end of the diverging section, where the lower set of bolts prevent illumination.

The manufacturability of quartz limits the complexity and location of geometrical features. Therefore, the positioning of the components could not be realised by additional lips and such concepts had to be discarded. Similarly, positioning bolts could only be placed in locations, where they do not interfere with the illumination, which again impeded

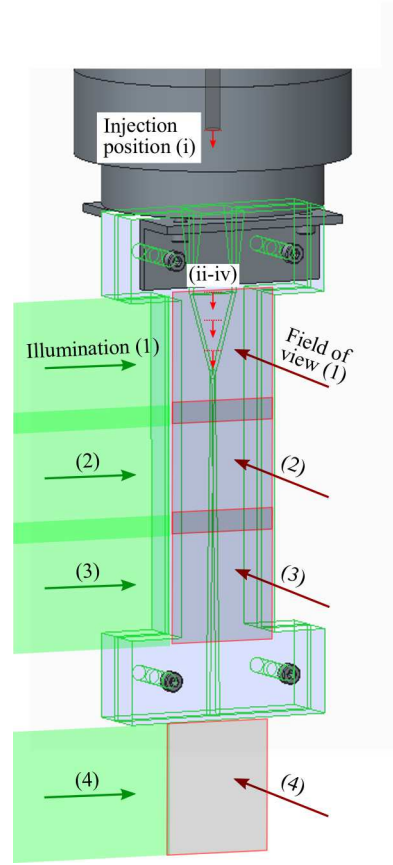


Figure 3.9: Schematic of the quartz nozzle assembly, illustrating the measurement concept

on manufacturing and stability, in particular of the thin-walled contour blocks. In the x-direction, the nozzle parts are in contact with the nozzle head surface, therefore, in this direction, the positioning uncertainty is in the order of the manufacturing tolerances $\pm 0.1\text{mm}$. In the y-direction, the bore diameter of 5.2mm exceeds the bolt by 0.2mm, hence causing a possible misalignment of 0.4mm on each side, or 0.8mm for the whole assembly. Since the contour blocks define a throat height of this order, the positioning had to be carefully controlled optically after calibration. Here, the position of the wall is checked by the appearance on the camera image, which then depends on the calibration uncertainty and the additional uncertainty originating from the image resolution.

$$\frac{\Delta h}{h} = \text{magnification error} + \frac{\Delta px}{px} \quad (3.10)$$

The magnification error is discussed in the context of the PTV system in section 3.5. Here, px denotes the distance of the walls in the image and Δpx the pixels in which the wall is

located. The wall is located within one pixel, and accounting for the minimum distance of the walls at the nozzle throat, the overall absolute uncertainty results to $\Delta h^* = 0.134\text{mm}$, which is similar to the manufacturing tolerance.

3.3.3 Injector needles

Four injectors were used for comparison. The first one, the short injector “0”, is used as a reference case, which injects the particle phase 80mm upstream of the nozzle inlet in order to provide a homogeneously mixed two-phase flow. This sort of arrangement is also typical when particles are pre-heated in this low-velocity section. The remaining three injectors “1-3” are designed for nozzle-internal injection in different positions approaching the nozzle throat. They have constant needle diameters d_{inj} and different needle lengths l_{inj} . Their properties are summarised in table 3.2.

Injector	Position (wrt. throat) $x_{inj} - x^*$	Needle size d_{inj}	Needle length l_{inj}/d_{inj}	Usage
0	-110mm	2mm	10	Far injection
1	-30mm	2mm	25	Inlet injection
2	-19mm	2mm	30	Mid injection
3	-9mm	2mm	35	Throat injection

Table 3.2: Injector configurations for various injection locations

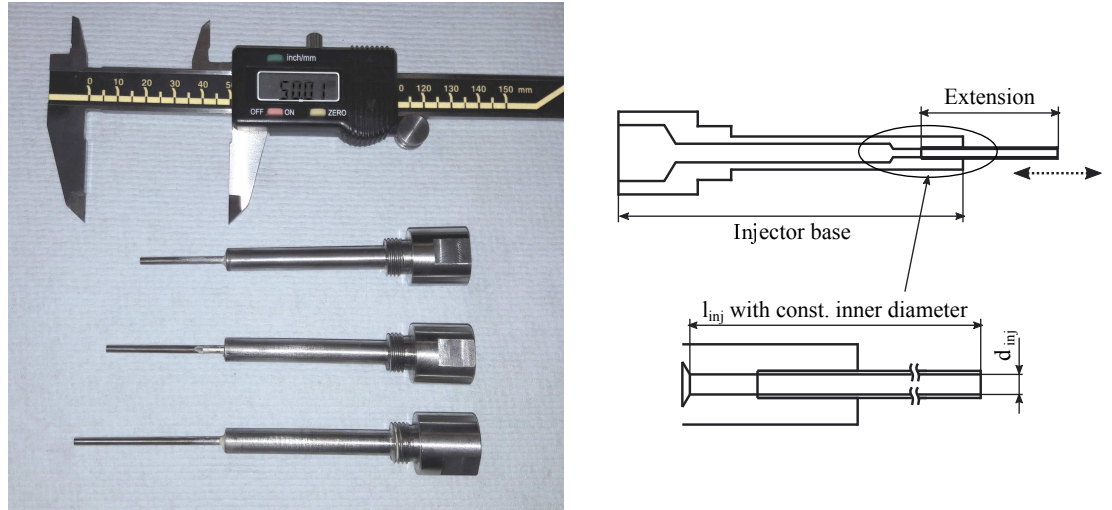


Figure 3.10: Picture and schematic of the nozzle-internal injector needles, variable extension defines length

The injectors were designed to have a prolonged part of constant cross-section, so that the needle-internal flow is fully developed. Stability is provided by a thick-walled

root section, which includes a standard fitting for direct assembly to the nozzle head. The rear side of the needle provides an internal thread for pipe connection once installed. The nozzle-internal injectors are shown in figure 3.10. The needle width is chosen based on the internal dimensions of the rectangular nozzle, as it must not block the gas flow around the needle. As a compromise however, the gap was minimised in the direction of the constant nozzle width. In case of the longest injector needle (3), the gap between the needle tip and the nozzle wall must not produce an effective restriction with critical conditions. Therefore, the effective gap area must be larger than the nozzle throat area, which is secured with a factor of 3.

3.4 Materials and parameters

3.4.1 Gas and powder

Nitrogen gas is used as process and carrier gas. It is the only gas used for measurements in this work, as a cheap and commonly used in Cold Spray. Because the maximisation of particle velocities is not subject to this work, using helium as a gas with higher expansion potential is not considered. For adjustment and testing purposes of the illumination parameters, compressed air is used at low pressure levels.

	Stellite-6/-21 (St)	Titanium (Ti)	Aluminium (Al)
Density [$kg\ m^{-3}$]	8440	4500	2700
Sieve analysis	-45+20	-45+15	-45+15
Shape	spherical	spherical	mostly spherical (Sphericity $\Phi_s > 0.99$, eq. 3.11)

Table 3.3: Feedstock powders

As feedstock powders, three main material types are used: aluminium (CP) by *Valimet, USA*, titanium (Gr2) by *Active Metals, UK* as well as Stellite by *Kennametal, Germany*. The latter is a group of cobalt-chrome alloys and, as such, highly relevant for CS corrosion protection applications. Stellite-6 and Stellite-21 are both used in different test series - which differ slightly in composition and mostly their hardness properties, while their material density and particle size distribution are identical. Likewise, aluminium and titanium are important materials for spray applications, in particular in the realm of

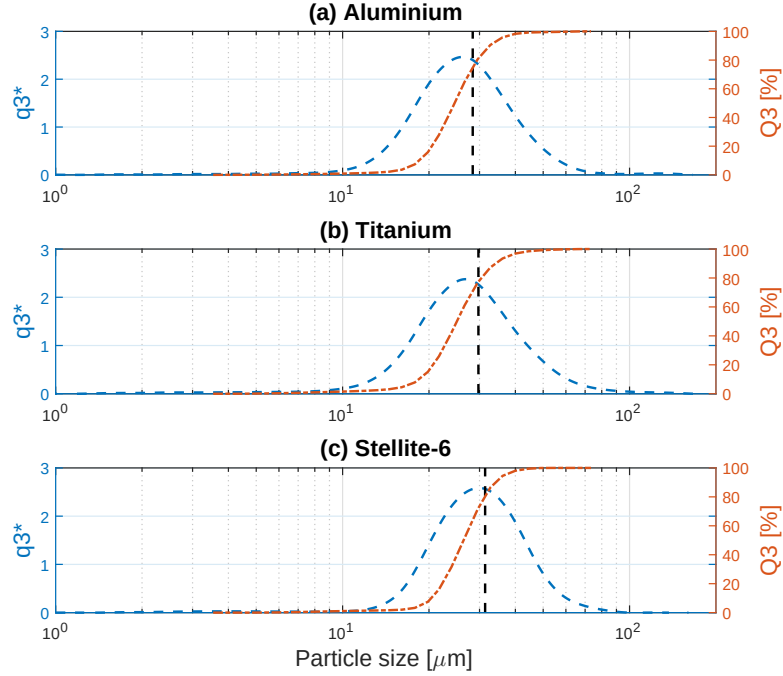


Figure 3.11: Size distribution of powder materials (a) Aluminium, (b) Titanium and (c) Stellite-6

additive manufacturing. Table 3.3 summarises the powder properties. All powders are gas-atomised and have very similar log-normal size distribution, with a size distribution between 10 to $60\mu m$ at a Volumetric Mean Diameter (VMD) of approximately $30\mu m$. This is shown in figure 3.11, where the fractional volume density $q3^*$ and the cumulative volume density $Q3$ are displayed over the particle size range. The data is obtained by a *Sympatec HELOS/BR-Cuvette* laser diffraction particle size analyser. The device uses dispersion in water using a magnetic stirrer rotating at 1200 RPM in tandem with an ultrasonic probe, enabling detection of particles of $0.25\text{--}875\mu m$ through Mie-scattering analysis.

Stellite represents the main material type, which is used in all cases to analyse the various aspects. When possible, a comparison to the other two materials is pursued, but Stellite is consistently considered first in all test series, because it is most favourable and reliable among the selected materials with respect to the important properties. These properties are, firstly, the three distinct material densities, secondly, their advantageous, smooth feeding behaviour, and finally, the ease of illumination. The latter depends on the particle size and shape, as well as the reflectivity of the surface. Coherent laser light impinging on small particles is reflected in the process of Mie-scattering, which strongly

depends on the light intensity, as well as the ratio of particle size and light wavelength, forming intensity patterns that strongly vary with scattering angle. Therefore, the intensity of light reflected towards the camera sensor is not linearly related to the particle size. Although the particle size cannot be reconstructed from this light intensity, the advantage is that small particles can be visualised in large field of views due to this over-proportional amount of light. The present materials can be nicely illuminated, due to their reliably spherical particle structure. In figure 3.12, Scanning Electron Microscope (SEM) pictures display the respective spherical powder morphology. Some degree of irregularity can be seen for the aluminium powder. The sphericity

$$\Phi_s = \frac{\pi(6V_p)^{2/3}}{A_p}, \quad (3.11)$$

where V_p is the volume and A_p the surface area of the particle, can be estimated from the SEM images by measuring the relation of the distances across the particles and assuming an ellipsoid shape. The average value is the lowest for aluminium $\Phi_s=0.995$, where 1 is a perfect sphere. Therefore influences can be assumed to be negligible for all powders.

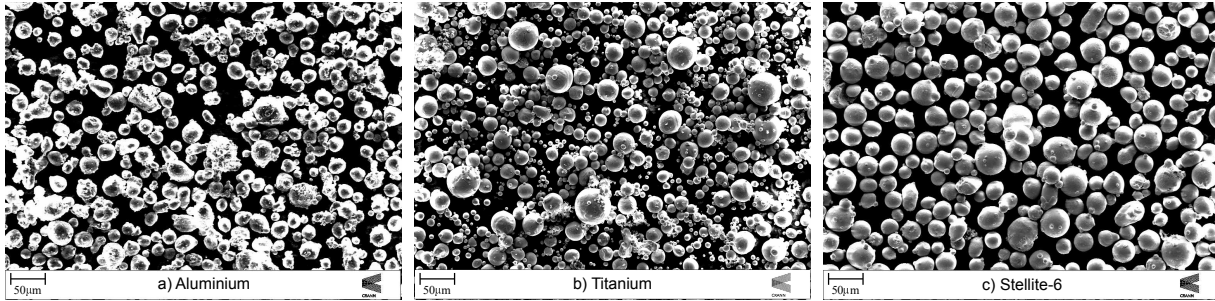


Figure 3.12: SEM images of feedstock powders

3.4.2 Operating conditions

Table 3.4 outlines the most important experimental conditions on the CS side (the measurement settings are covered in section 3.5). This table brings together the parameters used to pursue these subsequent ideas:

- Test series “Loading effect”: Particle loading study of CS nozzle in the jet.

Until now, the effect of significant phase coupling was only reported with ambiguous results. The studies are implicit in character because the observation focuses on

Test series	Loading effect				
Nozzle	NZ1				
Goal	Clarify particle loading effect for application nozzle				Comments
Location	FoV1	FoV2	FoV3	FoV4	Opaque nozzle
Feedstock				AL, TI, ST	
p_0 [bar-g]				15, 30	Ti emitting light (glowing) at 30bar, no evaluation
Feeder speed [%]				5, 10, 15, 20	Wide range
Injectors				Inj.0	
Flow ratio r [-]				0.1	
T_0 [K]				293	
Test series	Internal measurements 1				
Nozzle	NZTR				
Goal	Connect loading effect to internal motion: low-speed and high-speed behaviour				Comments
Location	FoV1	FoV2	FoV3	FoV4	Diverging section: nozzle worn
Feedstock	ST			ST	Focus on St; Al sticks to wall; Ti nozzle worn
p_0 [bar-g]	3 - 8			3 - 8	
Feeder speed [%]	2, 5, 10			5, 10, 20	Higher loading possible in jet, as particle spacing is higher (image density)
Injectors	Inj.0			Inj.0	
Flow ratio [-]	0.1			0.1	
T_0 [K]	293			293	
Test series	Internal measurements 2 & 3				
Nozzle	NZTR				
Goal	Investigate particle motion: injection, acceleration, and dispersion				Comments
Location	FoV1	FoV2	FoV3	FoV4	all FoV for St: analyse full length acceleration
Feedstock	ST, TI	ST	ST	ST, TI	Al: sticks to wall, Ti: nozzle “worn” in diverging section
p_0 [bar-g]	5 - 9	5 - 9	5 - 9	5 - 9	
Feeder speed [%]	2, 5, 10	5	5	5, 10, 20	div. section wears too fast for high loading
Injectors	Inj.1,2,3	Inj.1	Inj.1	Inj.1,2,3	
Flow ratio [-]	0.1	0.1	0.1	0.1	
T_0 [K]	293	293	293	293	

Table 3.4: Overview of operating conditions using different nozzles and measurement set-ups

particles in the jet, i.e. downstream of the nozzle, in which the acceleration takes place. In order to clarify the importance of deeper two-phase analysis, extending this implicit approach of measurements in the jet is the first objective to this study. Using nozzle NZ1, experiments are conducted at 15 and 30bar-g in the jet region (FoV4) with the three different material types, injected by the shortest needle (Inj.0), in order to analyse the effect of particle loading on the particle characteristics in the free jet. The focus is therefore on the wide range of feed rates. Light emitted from Titanium particles disturbed the images at 30bar operation, such that the respective data sets were not evaluated. This continuous “glowing” of a fraction particles left long stripes of light over the image, and particularly occurred for particles at the jet edge. A test with air instead of nitrogen confirmed this glowing effect also occurs at low pressures of 5bar, suggesting that (most likely small) particles begin reacting with oxygen producing this disturbance.

- Test series “Internal measurements 1”: Connect nozzle internal particle motion to loading effect.

The variation of particle feed rates causes unknown changes of the particle behaviour inside the nozzle. These are analysed in the high-density section of the nozzle and their trends connected to those observed in the jet. Since the series aims to learn about the principles of the particle movement in the converging nozzle part with increasing loading, the experiments are conducted with the main material Stellite. In this series, changing the material is not yet considered, which allows for extended use and set-up optimisation until the nozzle is “worn”. Here, the term wear describes the nozzle losing its function to enable measurements. Different from general understanding of wear, the particles not only function as abrasives, but cause a combination of erosion, deposition and change in surface structure on the nozzle wall over time, which is not analysed in detail. Here, it is central that the particles degrade the optical clarity of the components, to be noticed by an increase in the velocity measurement quality (image correlation). Measurements have to be excluded for this reason once the light transmission is affected. In this test series, Stellite is used for this reason only. Changing material type to Titanium is considered in a following test series about the influence of injection. Also, Aluminium particles are causing a

dust film on the inside of the wall and impede the imaging method. This material is hence not considered in the internal analysis.

- Test series “Internal measurements 2”: Measurement of acceleration and dispersion.

The transparent nozzle NZTR is investigated at all locations (FoV1-4) at various pressure settings in order to fully observe the internal particle behaviour. Having the best illumination and imaging properties, the main material Stellite is in focus of this study and measured throughout the nozzle at constant feed rate. In order to guarantee high quality measurements throughout the nozzle, the simultaneous increase of the feed rate is not considered because of heavier wear of the nozzle diverging part. This wear also prevents the use of Titanium as a comparison material in the high-speed section.

- Test series “Internal measurements 3”: Influence of Injector design.

With NZTR, the change in injector location is investigated directly at the injection port, at the nozzle throat and in the jet, using the lighter Titanium besides the heavier Stellite, while Aluminium again failed to deliver results. The three different nozzle-internal needle locations are tested with respect to particle speed and dispersion, including the influence of the mass loading.

The ratio of the main and feeder line flow rates is denoted as flow ratio and kept constant at 10% for all measurements. Likewise, the stagnation temperature is set invariant, to approximately room temperature at the inlet. Similarly to the use of nitrogen instead of helium, no gas heating for increased speed of sound is used, as rather the relative changes and statistical trends of velocities than their absolute magnitude are of interest. With a substrate absent and hence neglected deposition, thermal softening of particles is likewise irrelevant.

Imaging of Aluminium nozzle-internally is challenging due to slightly more pulsating injection and a developing dust-film of small particles on the nozzle walls in the low-speed section, which impedes imaging. Future work may address more materials and therein also resolve this issue.

3.5 Particle Tracking Velocimetry

Two-dimensional Particle Tracking Velocimetry (PTV) was used to optically measure the particle velocities. While Particle Image Velocimetry (PIV) leads to velocity field data, PTV can identify the velocities of individual particles. In section 2.4.3 of the background review, a short introduction in these measurement principles was given and is summarised here, while the following sections explain the processing methods detail: High spatial resolution of velocity data with high accuracy can be obtained by PIV, in which particles are illuminated with two subsequent laser pulses and recorded on images, from which their displacement during a known pulse separation time and hence their velocity can be inferred. This is done by a cross-correlation on interrogation windows (e.g. 32x32px), obtaining a field vector that has lowest uncertainty, if the correlation peak is distinct - achievable with mono-disperse particles with an optimal image size of 2px and low image noise. For PTV data, single particles are subsequently identified in a second step and their individual velocities calculated. As the feedstock particles are measured directly, the gas phase is not subject to measurements in this study. Because of high Stokes numbers $Sk \gg 1$ and hence significant lag between particle and fluid motion, no information about the gas phase can be inferred.

This section is dedicated to the introduction of the measurement equipment in terms of hard- and software, developed by *LaVision*.

3.5.1 Hardware

The laser in use is a *NewWave SoloII-15 PIV* Nd:YAG Laser with harmonics for a wavelength of 532nm. It consists of two cavities for the two pulses that are aligned through internal optics. It is a Q-switched laser with 3-5ns pulse duration, always operated at 4Hz repetition rate, and a maximum pulse energy of 30mJ. The pulse energy during measurement is a fractional setting of this maximum energy and differs for all materials and fields of view, as each setting needs to be adjusted in terms of illumination. Therefore, the pulse energy cannot be quantified exactly, but is in the range of a few percent of the maximum value. This low energy is possible due to the relatively large particle size (w.r.t. the wavelength). The laser has a 3mm exit beam diameter and approximately 3mrad beam

divergence. It is produced for relatively low distance operation and has an approximately Gaussian intensity profile at the measurement distance. Figure 3.13a shows the laser head and its internal structure.

After directing the laser beam towards the nozzle by a mirror, a light sheet is formed by a telescope of spherical lenses (a diverging lens and a converging lens) for the sheet thickness and a cylindrical (plano-concave diverging) lens for the sheet width respectively, illuminating the plane of symmetry in and downstream of the nozzle. Figure 3.13b illustrates the arrangement of the optical components. Positioning of the lenses was determined based on the focal point of the telescope, which lies on the nozzle centreline. Moreover, the beam convergence angle was chosen as a compromise between the sheet width and the requirement of a minimum of 20mm depth of constant illumination. The resulting light sheet within the nozzle is approximately 0.5mm thick and 60mm wide.

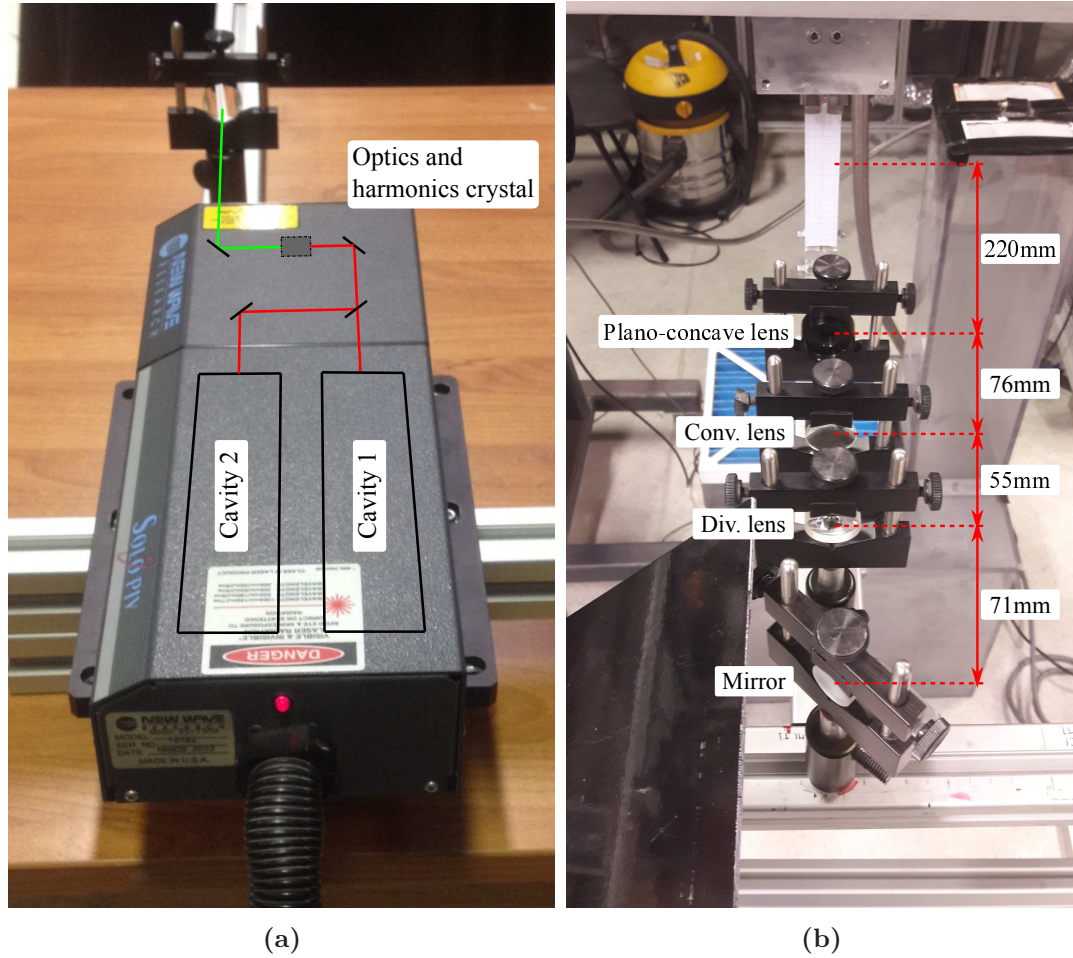


Figure 3.13: Laser head and optical components for light sheet formation

As the camera, a *PCO* SensiCam SuperVGA imaging system is used; it is a monochromic 12-bit dynamic range camera with a resolution of 1280x1024 pixels of $6.7\mu\text{m}$ pixel size, that delivers the consecutive image pairs. The camera lens is a *Nikon* 28mm-prime lens, with the aperture set to f/8.0 in order to minimise lens aberrations. The imaging magnification is defined based on the approximate desired field of view width of 50mm, and re-set for any new camera arrangement due to re-calibration. Due to the fixed focal length of the lens, it resulted always close to a magnification of 0.15560:1. The field of view is maximised under acceptable particle image sizes - this magnification leads to theoretical minimum particle image sizes due to diffraction between 1.8px and 2.3px, and the actual observed values are found between 2 and 5px. The depth of field is approximately 7mm, hence larger than the light sheet width. The 4Hz repetition rate for the system was limited by the CCD readout time of the camera. Consequently, the entire evolution of the particles through the flow field could not be tracked considering the high velocities. Therefore, in this study, the statistical analysis of the observed instantaneous data during steady operation represents an ensemble averaging procedure.

Double-frame CCD camera			Nd:YAG laser		
Resolution	1280x1024	[px]	Wavelength	532	[nm]
Pixel size	6.7x6.7	$[\mu\text{m}]$	Pulse energy	30	[mJ]
Repetition rate	4	[Hz]	Repetition rate	4	[Hz]
Dynamic range	12	[bit]	Pulse separation	10, 5, 3, 2, 1	$[\mu\text{s}]$
Magnification	0.15561	[-]	Pulse duration	3-5	[ns]
Aperture	f/8.0		Light sheet	0.5x60	[mm]

Table 3.5: PIV-hardware properties

FoV1 was observed at 10, 5, and 3 μs pulse separation time. The decreasing values are necessary to adapt for to the strong acceleration of particles within the nozzle restriction and hence prolonging the effective dynamic range of the measurement. Following the velocity trends, FoV2 was captured at 2 μs and FoV3 and 4 both at 1 μs respectively, each aiming for an approximate displacement of 5-10px for good cross-correlation. The timing unit (PTU) is set to deliver signals to the camera and the laser, synchronising their activities, with a time resolution of 10ns. Two top-hat pulses are sent to the individual laser cavities: one for the flash lamp that loads the crystal with energy, and one for the Q-switch that enables emission. Their temporal delay sets the intensity level. The first pulse

is set to occur within the time frame in which the camera captures the first frame. The second pulse is fired after the respective pulse separation time. At this point the camera is required to be in process of capturing the second frame. Due to the chip read-out specifics, this second camera exposure time is an order of magnitude longer than the first, such that it is prone to light pollution. Taking experiments in the dark is hence essential. Table 3.5 summarises the most important aspects.

3.5.2 Software

The PTU setting, data acquisition and image processing was conducted by *LaVision DaVis v7.2*. The preprocessing of images includes a standard background subtraction of an averaged image from 100 samples in absence of particle feeding, and a highpass filtering of the raw data to reduce stationary image features and lowfrequency background variations with a kernel width of 5px. The image processing involves a two-step approach, in which a PIV step is used as a predictor for the PTV algorithm that is used as a second step to identify single particle vectors. A multipass cross-correlation with reducing interrogation window size from 32x32px to 16x16px with 50% window overlap is employed. Computation of the spatial correlation is performed by a cyclic Fast Fourier Transform (FFT) and the peak-fitting algorithm is set to the standard three-point Gaussian fit. This procedure is followed by a median filter for removal of outliers as a post-processing step for the predictor. The subsequent detection of single particles is performed with a grey-value threshold of particle images. By applying the correlation algorithm on 16x16px interrogation windows that are centred on each first exposure particle image and pre-shifted by the field predictor, the individual displacements are found by evaluating the existence of a valid particle image in the second exposure.

Calibration of the camera image was conducted with a target plane, placed at the location of the measurement plane within the nozzle, see figure 3.14. In this way, any effect of the quartz glass wall on the camera image was included in the calibration procedure. As only one camera was used and placed normal to the target plane, no distortion algorithm or mapping function was used.

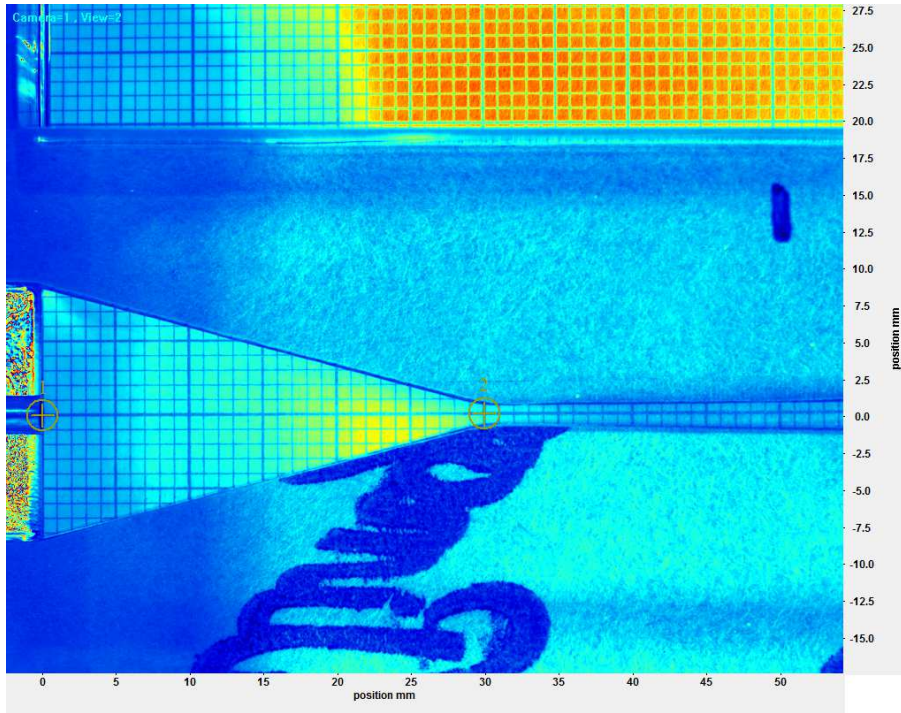


Figure 3.14: Sample image for calibration of FoV1

3.5.3 Uncertainty analysis

Repeatability

The quality of measurements depends on repeatability and uncertainty. The first is the assessment of unknown sources of error by means of repetition of the same set of experimental parameters. It is a straight forward approach to determine a possible range for measurement values for a given test. The repeatability of tests in this study is incorporated in the data analysis and marked respectively. Selected parameter sets are repeated up to 5 times. Generally, the respective results fall within a range of $\pm 5\%$. It is important to distinguish between repeatability and uncertainty, as the repeated results are all still equally affected by the precision of the devices in use.

Uncertainty

Uncertainty analysis, also denoted error analysis, is therefore the assessment of known sources of error by means of a systematic trace of their consequences. In this study, the most important error analysis is connected to the PTV methodology. A multitude of sources contribute to the measurement error in PIV and PTV. Most studies in CS

investigations do not address the optical measurement uncertainty in detail, because of the complexity of this issue and the use of commercial spray analysis solutions that make such assessments difficult. However, careful investigations need to make an attempt to address this matter: summarising from the introduction of the methodology, here is a list of some important aspects that directly influence the inherent (device- and algorithm related) measurement uncertainty [161, 179–185].

- Illumination (Light sheet quality, equality of first and second pulse)
- Light sheet width and pulse separation time (loss of particles)
- Camera lens, light pollution, camera temperature (noise)
- Particle size (sub-pixel displacement estimator)
- Particle shape, size distribution and particle image density (correlation peak quality)
- Bias to specific particles or to specific displacements
- Calibration accuracy
- Algorithm properties and calculation accuracy (e.g. interrogation window size, overlap)

Components of the overall error can be categorised into optical uncertainties, such as lens aberrations as well as calibration errors, and algorithm-related errors, such as root-mean-square (rms) or random displacement error as well as a bias or peak-locking error. For an estimate of the first source of error, the target plane images can be used. From those, a maximal calibration uncertainty that (includes lens aberrations) is estimated to 0.06px for a 10px displacement in FoV1-3 and 0.04px in FoV4 due to absence of the additional quartz wall.

Due to the amount of influences, the second source of error is much more complex to estimate, and a multitude of approaches has been and is still being developed. For well-known algorithms, like the present one, previous thorough analysis is available. Such an investigation of the present image processing methods can be found in [186, 187]. According to this analysis, the rms-error is estimated to 0.18px (1.8%) on the average

displacement for the present minimal interrogation window size. An advantage of this approach is that it involves real images from a physical flow process and hence all real effects like light pollution, different from synthetic images that are artificial but have exactly known properties. Nevertheless, because the rms-error massively depends on the image quality, which is different in any study, and the above study particularly employed a different size distribution of particles and distinct illumination, the error estimates are corrected. Based on the worst case of the primary peak ratios in the correlation plane of the measurement images, the error values are conservatively changed to 0.2px for FoV4 and 0.3px for FoV1-3. This increased uncertainty is a reflection of the increased blurriness of particle images within the nozzle due to small-scale surface imperfections of the quartz glass wall and represents a very secure estimate of the error. The used algorithm for particle tracking was shown to have exceptionally low bias error. Histograms of the sub-pixel displacement data show that it mostly was free of pixel bias. However, at higher feed rates, some tendency could be observed for FoV1, such that an additional error of the peak widths of 0.2px had to be added in these cases, see figure 3.15.

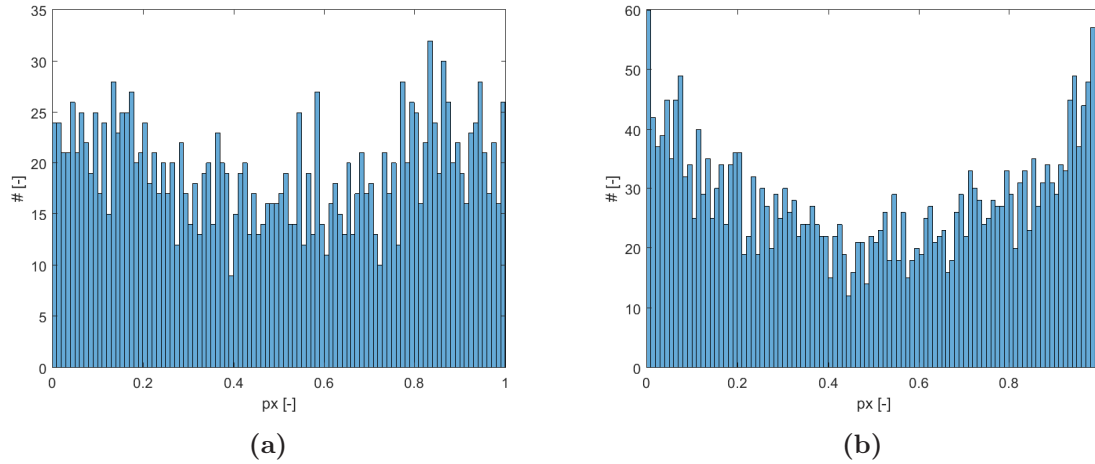


Figure 3.15: Observation of peak-locking in FoV1 images by means of sub-pixel displacement: absent at low particle feed rates (a) and present at high feed rates (b)

In addition, laser jitter is in the order of pico-seconds, and hence negligible. The particle movement during a single pulse is due to the short duration in the order of 0.06px, such that blurred particle images due to this effect are precluded.

Bias towards large particle size

The variance in the particle size not only leads to a higher noise level, but also causes a potential bias of detections towards big particles. While those always scatter sufficient light to contribute to the signal, the small-end of the size range scatters insufficient light and may be lost in the noise. This end of the size range predominantly corresponds to the high end of the velocity range, if non-drag related particle motions are small.

This bias does not represent a measurement error per se, but it is important to be aware of, as the findings do not show the behaviour of the smallest particles. It could be argued that this plays a subordinate role, as the observed behaviour is nevertheless true for the detected particles and the effects caused by presence of the (unobserved) small particles are still represented. One way to assess the issue and make an attempt to quantify the bias is a comparison of the dynamic ranges of the measured velocity data and the sizes of particles. This is only useful, if the data comes from a homogeneous section of the particle flow under low feeding conditions with low local gradients (at nozzle exit, close to centreline). In a simplified, merely drag-driven theory, the velocity and the particle size are connected through the force law. This connection is not directly proportional, but can be approximated by the proportionality $v_p \propto \sqrt{1/d_p}$ between particle velocity and diameter [26]. This can be used for a comparison of the measured velocity range and the actual particle diameter. Due to their units, the two quantities are not yet comparable and should therefore be non-dimensionalised by their average, such that rather the normalised dynamic ranges are contrasted.

Figure 3.16 depicts the respective histograms of velocity and $\sqrt{1/d_p}$ for titanium as an example. It shows that the higher end of the velocity diminishes faster than the size range, which implies that the small end of particle sizes is ignored to some extent. Approximating the capped portion of particles by integration of the curve, it must be estimated that about 8% of the total Titanium particle mass may be excluded from the measurement through non-detection of the smallest particles. For Stellite and Aluminium, these values were lower at 4% and 6%. Within this study, the bias was deemed acceptable as it relates to the very small particles that also carry the highest risk of failing at deposition. However, for future studies, it is recommended to improve this aspect in the technique.

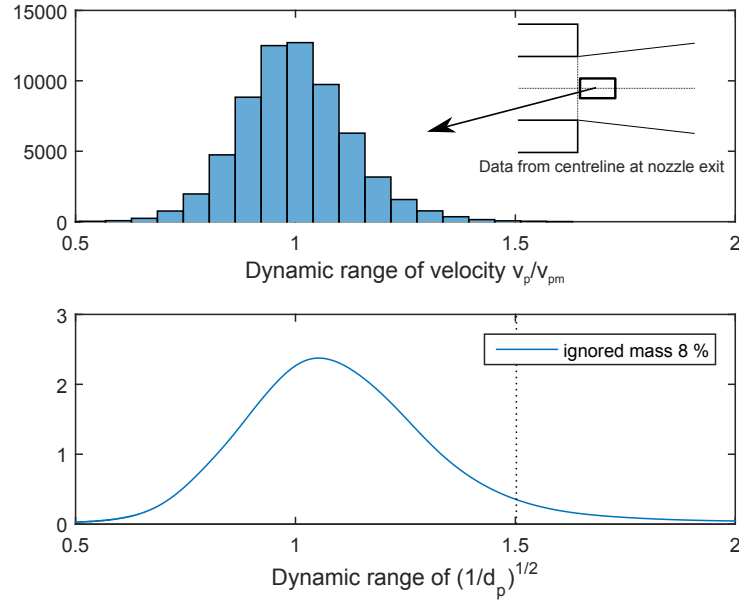


Figure 3.16: Comparison of the normalised dynamic ranges of velocity (top) and particle size function (bottom) for titanium powder to approximate particle size bias.

3.6 Summary

This chapter introduced the experimental novelty of measuring particle velocities and positions inside a Cold Spray nozzle using PTV. The experimental rig was described and the nozzle design reported in detail, emphasising the critical aspects of geometry, material, imaging and handling. The parameter settings of the experimental tests were introduced, including a discussion of the powder materials and an outline of the data processing methods. Finally, the measurement technique was described in detail and evaluated in terms of its precision. In sum, the chapter can be used for future reference and the here-defined terminology is going to be used in the analysis chapters to follow.

Chapter 4

Experimental results: Indirect jet flow characterisation

4.1 Outline of the results chapters

After extracting from the literature review in chapter 2 that gas-particle phase interaction mechanisms are still uncertain, a strategy to clarify them was defined through the research objectives (section 2.5):

1. Clarify loading effect by particle velocity measurements in the jet of an application-tested CS nozzle and extend current knowledge on the importance of gas-particle momentum exchange and on the particle dispersion.
2. For direct investigation of the particles during acceleration in addition to indirect jet characterisations, develop an experimental set-up for nozzle-internal measurements.
3. Measure and analyse particle behaviour within the nozzle and suggest physical mechanisms that explain the novel observations.
4. Based on this, develop a holistic computational model and analyse the present nozzle flow computationally, determine the limits of the model details and derive means for further model improvement.

While the methodology chapter 3 introduced the experimental procedures and thereby reported on the second objective to develop a nozzle-internal measurement, the remaining

objectives will be discussed in the following results chapters 4-6.

Chapter 4:

The gas-particle phase coupling was seen to affect the jet properties in some studies [21, 83, 85, 118], but the findings are inconclusive. Although measurements of particle velocity in the jet were used, in particular, the material of particles was never varied within the same system, such that the role of volume and mass loading could never be inferred. Before measuring inside the nozzle, the first objective is to extend analysis of the effect that particle loading has on the jet by experiments with an application-tested CS nozzle. This is used to infer the importance of gas-particle momentum exchange, of the particle dispersion, and of the influence of mass loading. Based on a wider range of operation conditions and materials than previously reported, it is a goal to suggest nozzle-internal mechanisms, which contribute to the particle behaviour in the jet. This is discussed in chapter 4.

Chapter 5:

Momentum and energy exchange occur within the nozzle, such that measurements at the impact zone do not provide understanding of how the particles reach their state. The previous chapter analyses how the coupling between the phases changes the jet of an application nozzle at increased particle loading. Since these changes are a result of the nozzle-internal behaviour, the goal of this chapter is to characterise the particle motion inside the nozzle experimentally. In the specifically developed transparent nozzle, the changes in velocity and dispersion are measured, when feed rate, material, pressure, and injector location are varied. The observations within the nozzle are connected to those in the jet, suggesting influential physical mechanisms.

Chapter 6:

Finally, these novel experimental findings are used to derive an advanced computational model. This is the final end of the present work, it is not yet possible to computationally guide researchers through challenges in CS applications that are connected to the nozzle-internal gas-particle flow: the deposition efficiency cannot be estimated based on CFD, the particle beam in the jet cannot yet be controlled, and in particular, the effect of nozzle clogging cannot be conclusively analysed numerically. A future advanced class of models should enable the assessment of such often-faced challenges. This is pursued in this final

chapter, providing a high quality model with detailed discussion of its capabilities on the basis of the experimental findings.

4.2 Introduction

Objective 1: Clarify loading effect by particle velocity measurements in the jet of an application-tested CS nozzle and extend current knowledge on the importance of gas-particle momentum exchange and on the particle dispersion.

In this context, this first results chapter begins by using the same indirect means of analysis as previous studies, *i.e.* the measurements of the jet, however extending the investigations to more materials and operating conditions. The reviewed literature discussed in chapter 2 paints an ambiguous picture of the influence of particle loading on the in-flight behaviour. Until now, the effect of significant phase coupling and, with it, the requirement for fluid dynamic considerations beyond a drag force, were only reported in implicit or indirect studies. What is meant by this implicit character is the observation focused on particles in the jet, *i.e.* downstream of the nozzle, in which the most important interactions take place. The few studies that address the parameter of particle loading show no conclusive understanding of how strong the inter-dependency of the phases is, and what mechanisms play a role. One particular reason is the lack of variations of feedstock materials and operating pressures. In order to clarify the necessity of deeper two-phase analysis, this chapter extends this implicit approach of measurements in the jet by adding changing feedstock materials and operating pressures to the existing spectrum of results, before the other chapters overcome confinements of this methodology and demonstrate the capabilities of the new approach.

The following sections therefore report the effect of the particle feed rate as a process parameter for CS application nozzles in more detail, experimentally investigating the particle velocity in the jet downstream of the exit of nozzle NZ1. This is done under low pressure and high pressure conditions. Unlike other studies, the feedstock materials are varied and measured under the flow conditions shown in chapter 3.4, aiming to identify possible driving mechanisms of the phase interactions when operating an application-tested nozzle.

4.3 Low stagnation pressure operation of NZ1

This chapter about the low pressure tests is viewed as an introductory part, that should not be reported in all detail, as it is based on a manuscript [173] originally published by the author in the context of discovering, that the models used in many CS calculations are insufficient. Nonetheless, it is the first indication of possible influences of high particle loading and hence forms the basis for the subsequent, high-pressure measurements. In this

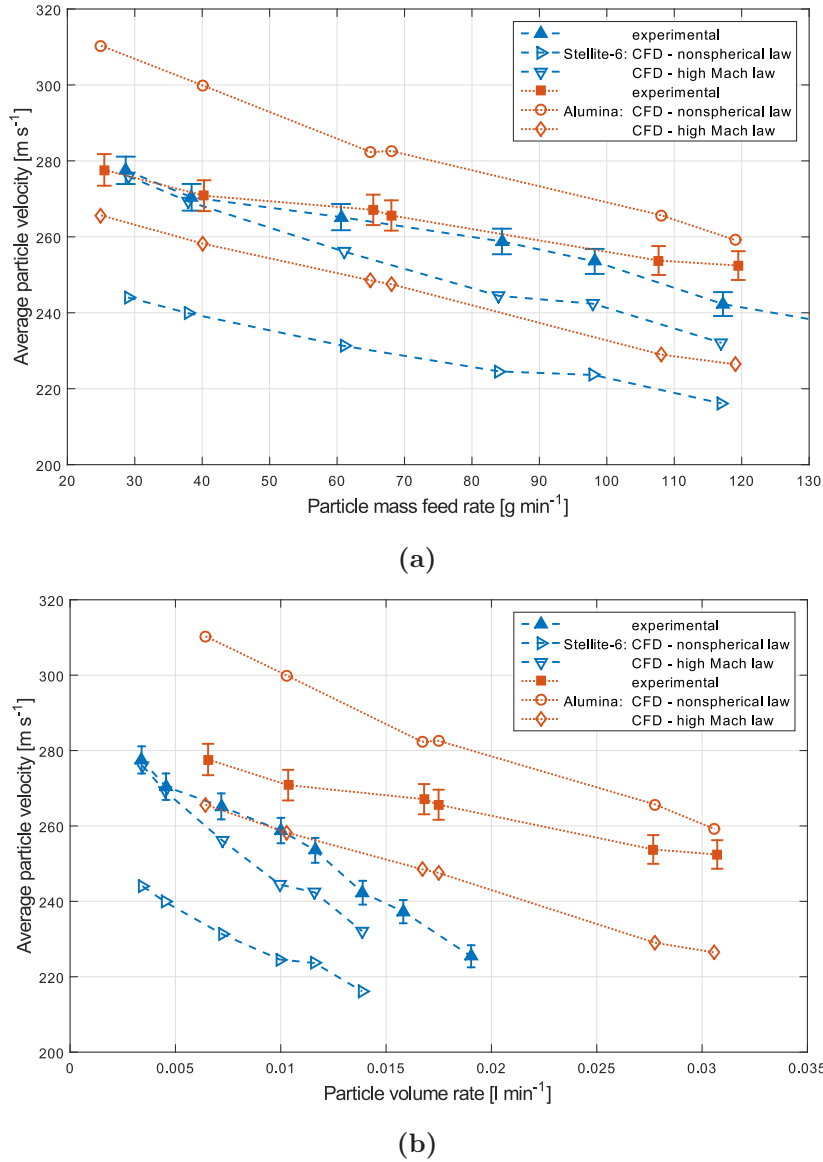


Figure 4.1: Initial low-pressure velocity reduction due to particle feed rate

preliminary study, PTV measurements at the exit of NZ1 with 5.8bar stagnation pressure are compared against respective 2D-axisymmetric Eulerian-Lagrangian simulations with

a two-way coupling procedure, which is not of interest in detail at this point. Stellite is used as the powder, nitrogen as the fluid, and the results are compared to a second measurement using bigger ($60\mu m$), irregularly shaped Alumina (Al_2O_3) powder, in order to distinguish between the influence of the drag-law and of the particle loading.

The measurements reveal that the particle mass loading in fact plays a role for the solid phase acceleration, as shown in figure 4.1a. Higher feed rates of Stellite-6 and Alumina trigger gas-particle interactions that result in lower velocity levels at the nozzle exit. In this case, differences in their material density are equalled out by a bigger size and irregular shape of the alumina powder, such that similar absolute velocities are measured. Differing behaviour is recorded when investigating mass or volume feed rates (figure 4.1b), wherein the effect of volume feed rate is significantly stronger for denser materials, because it corresponds to higher mass loadings. The effect of increasing mass feed rate is similar for the different materials, which means it primarily influences the loss.

As the figures show, these trends of decrease can be approximated using a two-way coupled CFD approach implemented in *Ansys FLUENT v14.5*, but not reconstructed quantitatively. The velocity magnitude depends on the choice of drag model (confirming literature in section 2.4.2): such widely used numerical models are moderately accurate to predict the particle behaviour, here between 5.5-12%, and are particularly sensitive to the shape irregularity. This, in turn, does not cause the discrepancy with respect to particle loading. The study shows further, that similar radial dispersion characteristics can be seen at this low pressure setting for both powders, showing first signs that the radial velocity profile might flatten at high loadings. Such dispersive motion is not reproducible with the model.

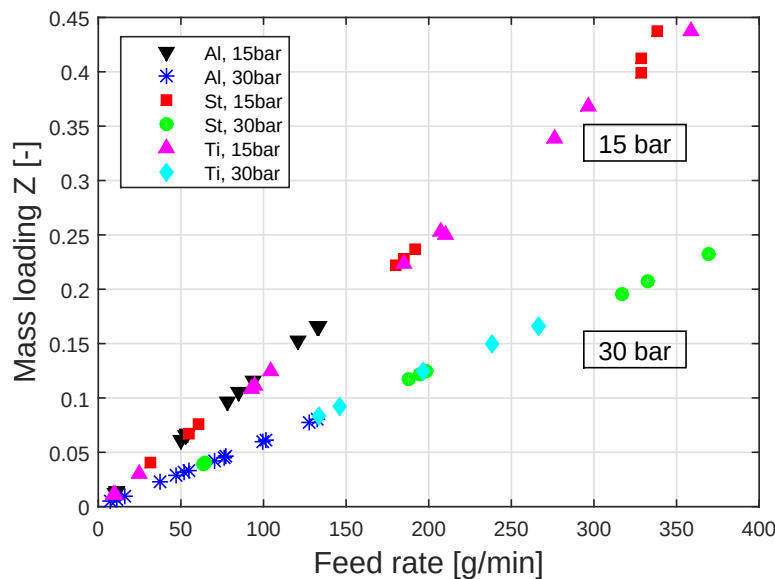
How increasing pressure changes these observations remains unknown, and although mass loading appears to be an important influence, the driving mechanisms are not yet understood. This motivates a more thorough analysis of the adequacy of the methods and leads to the investigation in the following chapter.

4.4 High stagnation pressure operation of NZ1

In order to advance these initial thoughts and learn about the topic in a more differentiated manner, the nozzle NZ1 is operated in the design pressure range: all three material types with similar morphology (Stellite, Aluminium, Titanium) are sprayed at 15 and 30bar under increasing feed rates. Again, particle velocity distributions are measured in the nozzle jet. In the definition of operating conditions in section 3.4.2, this test series is denoted as “Loading effect”.

4.4.1 Flow and loading conditions

Firstly, the feeding and spraying behaviour of the powders should be briefly discussed. As the wheel speed of the powder feeder is increased, the resulting mass feed rate of particles depends on the powder in use and the spray conditions. This results in varying ranges of feed rates for the different materials. For aluminium, having the lowest density, it was possible to increase the mass feed rate to just below 150g/min (corresponding to full wheel speed), whereas titanium could be fed at a maximum of 350g/min. For Stellite-6 even higher feed rates are possible, but lie beyond the scope of interest.



The mass loading Z was introduced in equation 2.15 and is hence calculated with

$$Z = \frac{\dot{m}_p}{\dot{m}_c}, \quad (4.1)$$

where $\dot{m}_p$ is the particle feed rate and $\dot{m}_c$ the overall gas flow rate. For completeness, it is useful to repeat that Z is connected to the volume ratio F by the material densities.

$$F = \frac{\dot{V}_p}{\dot{V}_c} = Z \cdot \frac{\rho_c}{\rho_p} \quad (4.2)$$

The gas flow conditions are kept constant during all measurements for steady operation, yielding ca. 0.0135kg/s at the 15bar and 0.0270kg/s at 30bar inlet pressure settings. A linear connection to the relative loading reveals good reliability of the gas flux, see figure 4.2. It approximately doubles when increasing the pressure from 15 to 30 bar, which results in a steeper slope for the low pressure setting. Loading effects that are related to this relative proportion can thus be expected to be more drastic for the lower pressure.

4.4.2 Velocity distribution and footprint

The chosen set of materials exhibits differences in particle velocity distribution under varying loading conditions. Figure 4.3 shows a comparison of the ensemble-averaged particle distributions in the plane of measurement. On the x-axis, the streamwise distance from the nozzle exit is displayed down to an SoD of 50mm, on the y-axis the radial coordinate. The subfigures represent increasing loading cases for aluminium and Stellite at 15bar (a,b) and at 30bar (c,d) respectively. The positions of detected particles are scattered and coloured by their velocity magnitude. The respective intermediate data for Titanium is evaluated for 15bar and given in the appendix p.234, while the 30bar measurements are disturbed by particle glowing, as introduced in section 3.4.2. Light is continuously emitted from some Titanium particles, particularly occurring at the jet edge when sprayed in nitrogen, or at all locations when air is used. An explanation high frictional forces when impacting onto the nozzle wall cause a beginning reaction with oxygen.

At the nozzle exit, each particle stream has a width of 6mm, as the nozzle exit radius is ± 3 mm. The exiting particles therefore occupy the full nozzle channel after full nozzle-internal dispersion. The plume then slowly widens downstream until it approximately

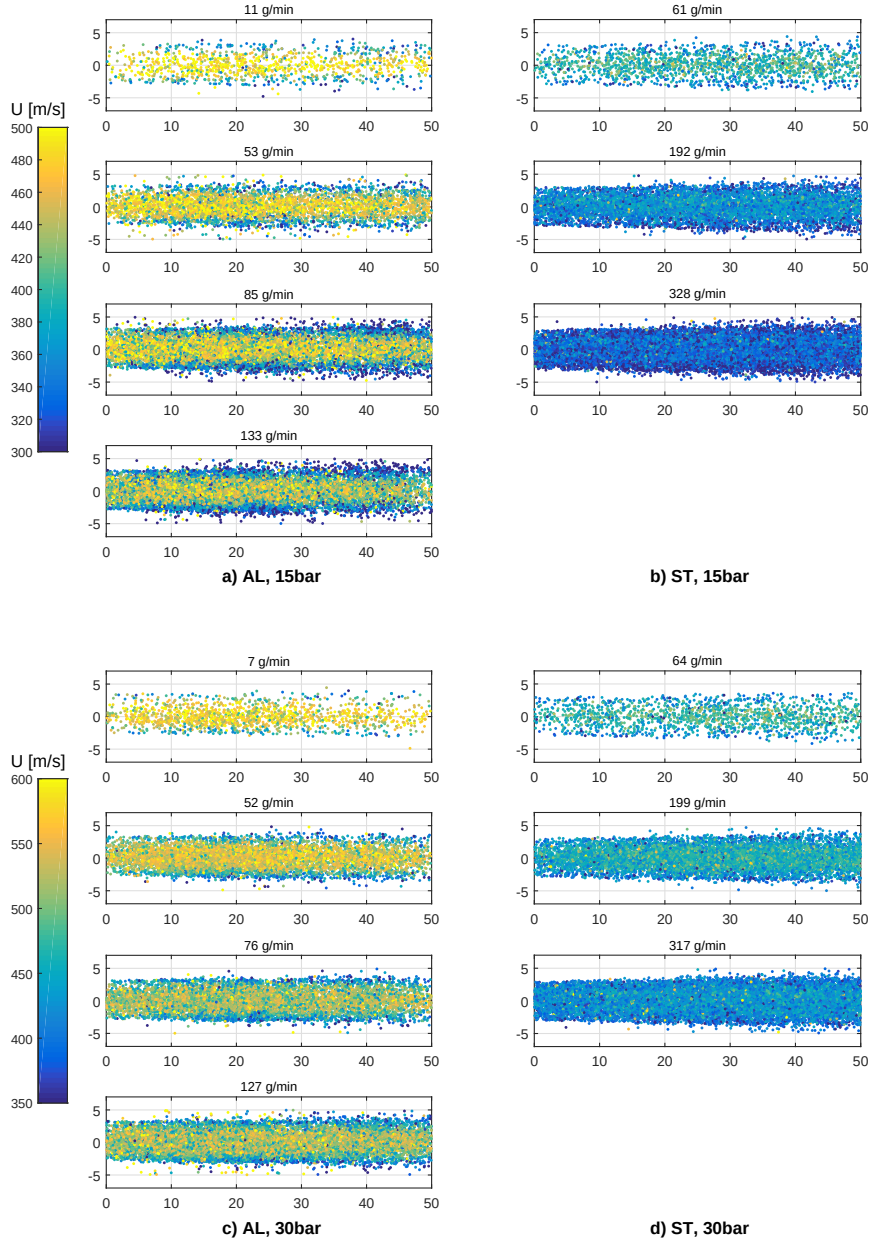


Figure 4.3: Particle velocity distribution in jet of NZ1. Scatter positions in [mm], 0=nozzle exit. Comparison of aluminium and Stellite-6 velocity distribution and particle dispersion for increasing pressure and mass loading

covers a range of ± 5 mm. Due to the high velocity levels, the dispersion over this axial length is moderate in absolute terms. Nevertheless, it determines the footprint and velocity distribution upon impact, and it is therefore important to understand its influences.

Several mechanisms that affect this dispersion can be listed. Particles interact with the turbulence of the gas, and because its level only grows in the jet, it is likely to be

mainly significant at far stand-off distances and for small particles. The particle paths are also deviated by shock waves forming in the gas jet. Again, the effect is stronger for small particles and, due to the over-expanded jet, the first shock tends to act confining rather than dispersing. Moreover, the gas velocity drops across the shear layer, which makes particles at the jet-edge more prone to disperse over long distances. Finally, the trajectories already exhibit a certain randomness as a result from the nozzle-internal motion. Their state at the nozzle exit, is an important starting condition for dispersion in the jet, and it is questionable that the particle paths are straight (as often suggested by CFD models). It can be seen from the measurements that, resulting from the sum of these sources, the dispersion is stronger for high loading cases and for lower pressures. Larger amounts of particles experience sideways forces and are pushed out of the jet core. Aluminium is more prone to this effect than the other materials; the lower material density plays an important role in this respect.

Similarly, this material also shows a more noticeable deceleration with distance from the exit, which generally increases for all materials with loading. It can be explained that the disintegration of the gas jet is stronger at high feed rates, weakening its potential core, and light particles respond more noticeably. A higher overall decrease in velocity (of both the higher velocities in the jet core and the lower velocities at the boundaries) can be seen with growing feed rate.

This has consequences for the spray spot, i.e. the impact velocity footprint. Figure 4.4 investigates the particle velocity profiles in the radial direction at a x-location between 40 and 50mm downstream of the nozzle exit. In this illustration, individual particle speeds are shown and highlighted by curves of Gaussian fits. It should be noted that the purpose of these fits is to guide the eye for ease of comparison and do not directly suggest as a physical trend. Although the gas phase may tend to a self-similar regime that provides such a profile, it will not do so within the observed range, and the particle profiles hence do not have strict physical reason to follow a normal distribution this close to the nozzle exit. However, the near-Gaussian gas velocity distribution is considered as an adequate fitting function.

When opposing the different materials, a weaker curvature of the profiles can be noticed when going from aluminium through titanium to Stellite-6. The maximum velocities of the

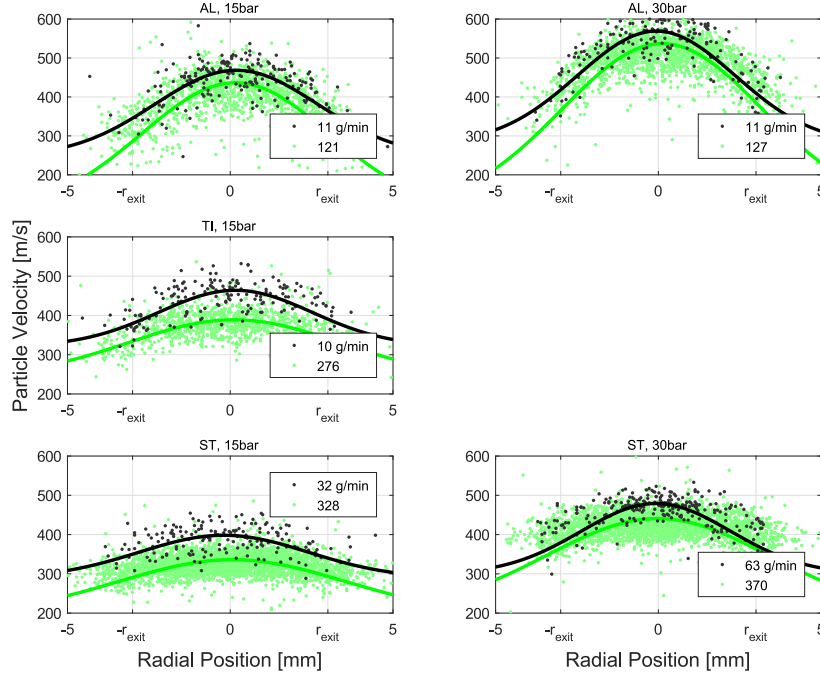


Figure 4.4: Comparison of radial particle velocity profiles at 40-50mm distance from nozzle exit. Data points represent single particle velocities, lines indicate profile trend as Gaussian fit

profiles are located around the centreline, and the radial velocity drop associated with the curvature reaches 200m/s for the light material, while for the alloy, it is only in the order of 50-70m/s. This observation underlines that the light material is affected more than twice as much by the spreading of the gas jet. Note that this may result in lower deposition efficiency for lighter materials. At higher pressure, the curvature is slightly higher than for smaller pressure due to higher core gas velocity, and a shift of the maximum by about 23% for aluminium and 21% for Stellite-6 can be registered. This is approximately the amount by which the velocities reduce when finally comparing the low feed to high feed cases. In this case, the slowest particles in the dilute cases are as fast as the fastest in the dense flow case. This reduction in maximal velocity is stronger for titanium and Stellite-6, where the velocity profile flattens strongly. In that sense, the particle phase experiences a significant equalisation due to loading of high Stokes number particles. Consequently, locations further away from the centreline are increasingly occupied by relatively fast particles. Physically, particles close to the centre exhibit lower velocities at high loadings because the gas core is weaker, while particles that are pushed outwards retain a higher portion of their speed because of their inertia. For aluminium, the curvature changes only

little with loading, since their momentum cannot be maintained well in the shear layer in either case. The low feed results show particles mainly within the region of the nozzle exit diameter ($\pm r_{exit}$), whereas the high feed indicates a significantly stronger dispersion in the realm of $\pm 4\text{mm}$ to $\pm 5\text{mm}$, which quantitatively supports earlier findings from the initial low pressure test. It is interesting to finally note that the aggravation of dispersion due to higher feed rates is weaker the heavier the material. This suggests that not only the mere mass of particles but also their volume influence the dispersion.

4.4.3 Mean velocity change and phase interaction drivers

The change in average particle exit velocity can quantify the susceptibility of a material to the increase in loading. Figure 4.5 shows respective results. The line segments are least-square linear fits. The velocity error bars comprise the inherent uncertainty associated with the optical analysis. As analysed in section 3.5.3, the imaging error is conservatively estimated to 0.2px per particle displacement. This error is added to the standard deviation of the experiment repetitions.

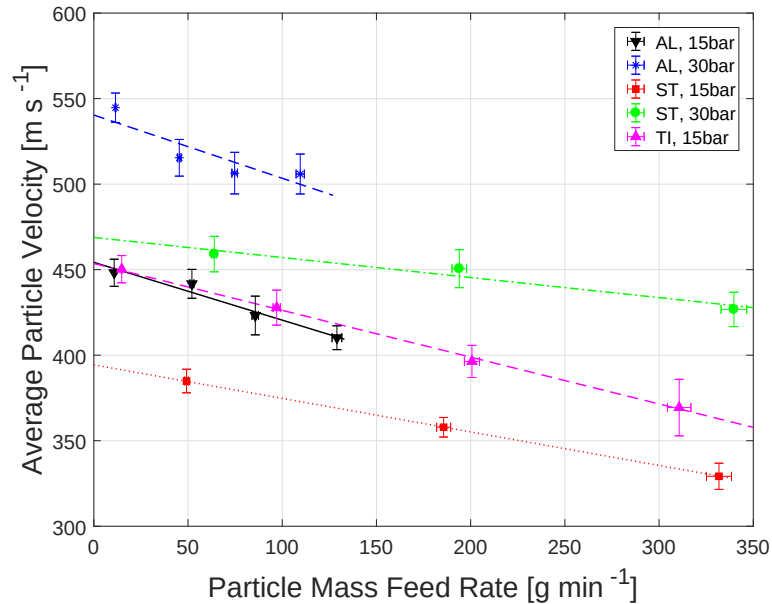


Figure 4.5: Average particle exit velocity trends with increasing particle feed rate

With focus on the velocity values at low pressure and very low feed rates, it can be seen that due to the different material densities, the velocity levels differ. Stellite-6 exhibits the

lowest velocity, ca. 55m/s lower than aluminium. As the pressure level is increased, this velocity level at low-feed rates increases by about 20% for both Stellite-6 and aluminium, or by 75 and 90m/s respectively. Increasing the feed rate clearly provokes a decrease in average velocity for all materials. This trend appears to be approximately linear within the given range of feed rates and slightly less severe at higher pressure. It is essential to note, that the loss in particle velocity is different from case to case; it can be as high as 10% for Aluminium, and up to 16 and 20% for Stellite-6 and titanium respectively. Interestingly, this implies that the same amount of additional particle mass injected in the nozzle does not induce the same velocity loss. More precisely, the slope for Stellite-6 is less steep than for titanium, and the reaction of aluminium is the most severe. According to this, the effect becomes more significant with decreasing density. Based on the conservation of momentum and energy, it was previously argued that the velocity of the discrete phase must drop because its mass increased by a specific amount. However, for the different materials, the given mass feed rate corresponds to different numbers of particles and volumetric feed rates, and therefore to different volume fractions. For example, 100g/min feed rate corresponds to volume feed rates of 0.0118 and 0.037 litres/min for Stellite-6 and aluminium respectively. Moreover, the gas consumption and the resulting mass and volume flux of gas change with pressure setting. If the effects are more complex, the additional influence on the average particle dynamics with a change in relative mass loading Z (eq. 4.1) and in volume fraction F (eq. 4.2) must be investigated.

In this regard, the quantities were non-dimensionalised in the following way. The particle velocity v_p is normalised by the very low-feed limit of each case v_{p0} , which can be interpreted as the particle velocity obtainable without any feeding effect. The feed rate and the measured gas flux were used to calculate firstly the relative mass loading Z , and secondly the volume fraction of particles F with respect to gas conditions in the nozzle throat. The susceptibility was then quantified as the gradient of this normalised velocity with increments of mass loading and volume fraction.

$$Gr_{Z,n} = \frac{d}{dZ} \left(\frac{v_p}{v_{p0}} \right), \quad Gr_{F,n} = \frac{d}{dF} \left(\frac{v_p}{v_{p0}} \right). \quad (4.3)$$

Figure 4.6 presents these gradients, wherein subfigure (a) shows the gradient with mass loading $Gr_{Z,n}$ and subfigure (b) with volume fraction $Gr_{F,n}$. Since the velocity in the jet

decreases with increasing particle feed, these gradients are negative. The different bars indicate the changing particle velocity parameter PVP at the different pressure levels. The PVP is a means to quantify the inertia of the particles, so a high PVP stands for a heavy material, while a low PVP represents a light one. It is interesting to note that high PVP particles also have high Stokes numbers and vice versa, but not information about the local gas phase is required. This figure is interpreted according to the following observations:

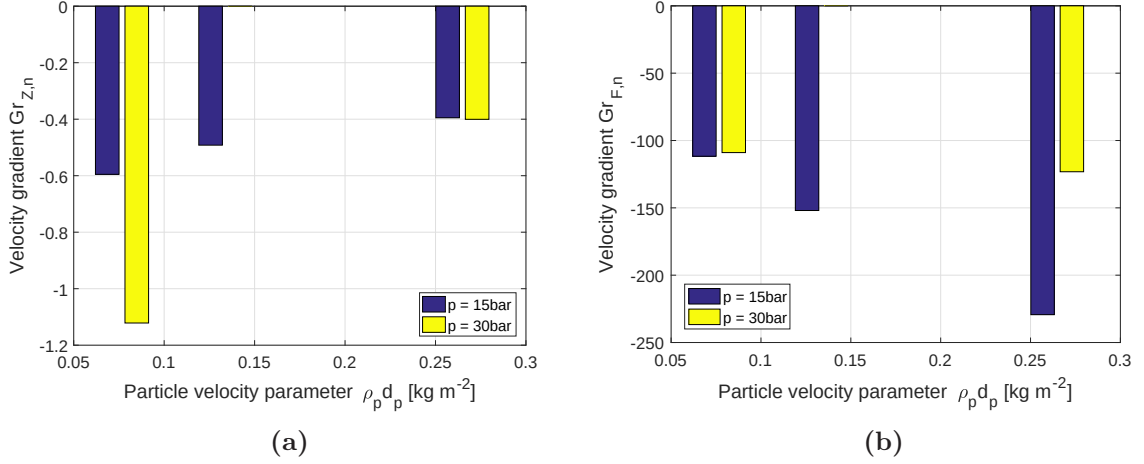


Figure 4.6: Gradient of normalised particle velocity with mass loading (a) and with volume fraction (b) as a susceptibility indicator displayed over the particle velocity parameter

- Observation 1: The heavier the material, the smaller the gradient with Z .

Subfigure (a) shows that, upon increasing mass loading, heavier materials are less prone to drop in velocity than lighter ones. The same raise in aluminium mass corresponds to a much higher volume increase than in case of Stellite. This particle phase volume therefore plays a role for these light materials.

- Observation 2: The heavier the material, the less its reaction to Z increases with pressure.

Subfigure (a) shows that, when increasing the pressure level, lighter materials become much more susceptible to mass loading changes, while heavier materials react similarly for both pressures. Since the raise in mass loading means a high volumetric increase for light materials, it is plausible that the higher pressure aggravates volume-related loss effects.

- Observation 3: The heavier the material, the higher the gradient with F .

Subfigure (b) shows that, upon increasing volume fraction, heavier materials are more prone to drop in velocity than lighter ones. The same raise in aluminium volume corresponds to a much lower mass increase, than in case of Stellite. This additional particle mass is therefore key to the velocity loss of the heavy material.

- Observation 4: The heavier the material, the more its reaction to F reduces with pressure.

Subfigure (b) shows that, when increasing the pressure level, heavier materials become much less susceptible to volume fraction changes, while lighter materials react similarly for both pressures. Since the heavy material loses its velocity predominantly due to the mass increase that comes with higher volume, it is plausible that the higher pressure helps reducing the gas-momentum loss.

Combining these observations, figure 4.6 gives an idea of possible nozzle-internal “driving factors” for velocity losses:

1. Heavy materials: particle mass-related loss through gas momentum reduction, taking place where relative velocities are high.
2. Light materials: particle mass-related loss and additional volume-related loss, e.g. through collisions in the nozzle, taking place where volume-fractions are high.

All materials, when fed at increasing rates, lose a portion of their velocity, because the additional particle mass has to be accelerated by the gas, which reduces the gases momentum flux. For heavy materials, this is the predominant mechanism, because they exhibit particularly high masses, even with moderate amounts of particles. When the pressure and hence the gas momentum flux are increased, this mechanism is dampened. Because this transfer acts through the momentum exchange of the drag relation, it is mainly taking place where relative velocity and acceleration are high. Within the operational range of a CS nozzle, these heavy powders do not noticeably reach a loading level, in which volume-effects play a role. For light materials, the mass-related mechanism is likewise present, however the additional consequence to overly increase the volume of the particles causes volume-related losses as well. Such additional mechanisms could be related to collisions

in the region where the volume fraction is high. Increasing the pressure could cause more frequent or more energetic particle-particle interactions, or reduce the nozzle efficiency by a deceleration of the dense gas-particle mixture in front of the nozzle restriction, which depends on the specifics of the injection. Moreover, a larger volume of particles will cause stronger turbulence modulation, which also depends on the micro-flow around particles and hence on the pressure level, although this aspect is still fully unclear for CS. Linking the losses not only to nozzle-internal particle motion, but also to different zones of the nozzle, it is an obvious consequence, that direct measurements as presented in the subsequent chapters are necessary for deeper understanding of the particle behaviour and for the development of adequate models.

4.5 Summary

Previous fluid dynamic investigations in CS rarely consider the particle feed rate (*e.g.* [44, 92, 107, 188]). However, higher particle loadings can induce noticeable effects on the particle dynamics. This chapter therefore experimentally investigated the particle velocities in the supersonic jet of NZ1, an application-tested CS nozzle, at low and high pressure levels with changing materials, Stellite, aluminium and titanium, by varying their feed rates. Herein, it is a novel finding that not only does the particle velocity decrease at high loadings, but also deceleration within the jet is enhanced. The radial velocity footprint becomes more flat in such a case. Aluminium, the lightest material, is found to be more prone to radial velocity decrease also for high feed rates, but all materials show intensified particle dispersion at increasing distances from the nozzle exit. Moreover, the study finds that the average particle velocity is reduced in every case when the particle loading is increased, in part as high as 20%. The materials behave differently when changing particle feed, depending on their average inertia. The relative drop in velocity can be influenced by both the relative volume fraction and the relative mass loading. For powders with heavy particles, (high particle velocity parameters and Stokes numbers), the driving mechanism is dominated by mass loading through the gas-momentum loss. For light materials (low velocity parameter and Stokes number), the volume fraction is an increasingly important driver. The responsible mechanisms are found to take place

within the nozzle and, in order to understand the particle dynamics further and improve modelling methods, one requires direct nozzle-internal measurements, as reported in the following chapters.

Chapter 5

Experimental results: Direct nozzle-internal measurements

5.1 Introduction

Objective 3: Measure and analyse particle behaviour within the nozzle and suggest physical mechanisms that explain the novel observations.

This chapter transitions to the second phase of extending the knowledge about gas-particle flow in CS. After employing the approach of particle tracking in the jet, it is clear, that such indirect measurements are insufficient and that deeper understanding of the process requires more insight in the actual nozzle-internal behaviour. With the insight that can be generated this way, computational models can be amended to cover not yet predictable application challenges, such as nozzle clogging and the statistical dispersion of particles. In this context, the following sections aim to overcome the confinement of jet characterisation by analysing direct nozzle-internal particle measurements, before addressing their value for a shift in the state of the art of numerical predictions in the subsequent chapter.

This chapter firstly presents an analysis of the gas-particle mixture, that undergoes the extreme changes characteristic for CS: the injection plume with volume fractions up to 1% at particle Stokes numbers between 10 and 1000, the internal behaviour under increasing feed rate, the acceleration throughout the nozzle and finally the consequences in the jet. The basic concept of this chapter is to analyse the statistical behaviour of particles under

such realistic conditions with respect to the increasing loading and changing injection configurations.

The present study is the very first report of a quantitative measurement of a nozzle-internal particulate flow, and therefore represents a very distinct novelty to the field of fluid mechanical measurements in CS. It suggests physical explanations based on a statistical evaluation of the observed data and forms the basis for future work on the understanding of CS fluid mechanics. Apart from this, nozzle-internal measurements have a variety of additional advantages on the application level. For instance, they reveal the injection plume and the respective dispersion of particles in the low-speed section of the nozzle, which could not be experimentally analysed previously. Operational challenges are often faced in CS applications, that are connected to the nozzle-internal particle behaviour, and one of the most important of such currently is the phenomenon of nozzle clogging. The methods presented in the chapter demonstrate their value in this respect by addressing a possibility to experimentally investigate the extent of unwanted particle-wall interactions.

5.2 Injection-independent particle motion

As a reference case for the observations inside nozzle NZTR, injector 0 (placed at 80mm or $40d_{inj}$ upstream of the nozzle inlet) is used. In the definition of configurations and operating conditions in section 3.4.2, this test series is denoted “Internal measurements 1”. With this injector, the particles are released sufficiently far upstream to be dispersed evenly when entering the nozzle flow channel and the influence of the injection plume is negligible. Under these circumstances, it is possible to observe the effect of particle mass loading on the particle velocity without any influence of the injector design or position. This section aims to indicate that there is a discrepancy between the established assumptions for mathematical modelling and particle motion in reality. For this purpose, the particle velocity in FoV1 and FoV4 using injector 0 is analysed for particles of Stellite.

At the nozzle inlet, the particle motion is found to have the expected spatially uniform character, however exhibiting significant statistical variances, as is going to be discussed in sections to follow. At this point, a comparison of the axial velocity distribution of particles within this flow region is shown in Figure 5.1, comprising results at (a) 5.0 and

(b) 8.0bar, each at the lowest and highest feeder wheel speed setting. The lines represent a moving average and the point-markers indicate the statistical deviation from this mean by virtue of the standard deviation of the local interval.

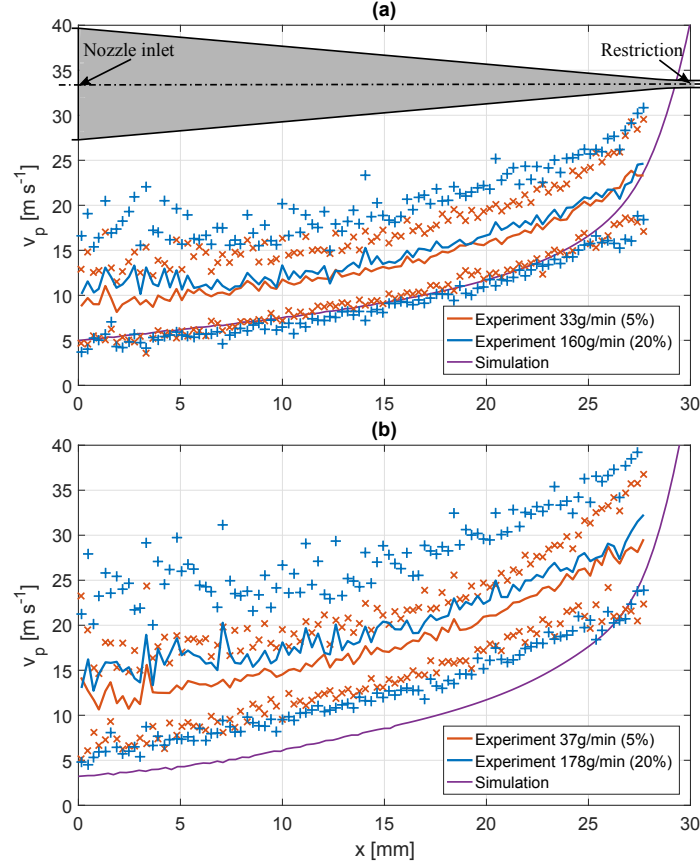


Figure 5.1: Longitudinal profile of particle injection velocity in converging section of NZTR for Inj.0 at (a) 5bar and (b) 8bar, moving average (lines) and corresponding standard deviation (markers).

The average velocity at low pressures is similar for both loadings. Interestingly, while increasing only the particle feed rate, the statistical distribution widens. This suggests that the higher loading results in a stronger mixing and a more unordered flow of particles. At higher pressures, this effect is drastically aggravated, as higher density and more momentum within the flow result in a stronger dispersion. It is uncertain from this data what mechanism causes the effect: an increased turbulence level and respective dissipative losses are possible, because bigger particles range up to more than 10% of the estimated integral length scales, while particles from the smaller end of the size range may rather contribute to dissipative effects [189]. It is unclear if the overall resulting effect would be enhancing or damping, and there is no experimental way to verify this. However, the

turbulence level within the most part of the nozzle is low, because the gas is streaming at low velocities. The large particle response times make the influence of these turbulent fluctuations on the majority of particles relatively small. Although the volume fractions are moderate, in combination with the large response times, particle-particle interactions can be a plausible explanation. Logically, such interactions should be highest close to the injector and decline slightly as the particles disperse, and finally increase again as the channel converges. Because the particle phase is homogeneous, the respective gain in momentum goes hand in hand with a more randomised direction of the particle velocity vectors. This increases the likelihood of collisions and increases losses.

In fact, it can be seen in Figure 5.1 that the particle velocity range spreads more towards higher values than towards lower values, slightly increasing the average velocity with loading. This asymmetry can be caused by more frequent collisions of particles during their acceleration by the gas. A colliding pair of particles results in one accelerated and one decelerated particle, which would widen the observed particle velocity range in both directions roughly by the same amount. Since both particles are accelerated by the gas flow at the same time, the slower particle gains more momentum from the gas than the faster one and appears less decelerated than expected. Consistently, the total velocity increase over the converging section is similar for both loadings, because the gas drags the mixture along continuously in either case. The polydispersity of the powders may play an additional role, since the effect of larger particles during collisions is more significant than of small ones. Since the powder contains more small particles that tend to correspond to the fast velocity detections, this may add to the asymmetry.

Also shown are the average velocity profiles of particles from a computational model, representing an established standard approach for CS analysis (Fluent v16.0, 3D steady-state RANS, with a realizable k - ε -turbulence model, Lagrangian discrete phase model with high-Mach number drag law, Rosin-Rammler particle size distribution). A brief comparison with computed data shows a strong discrepancy. Because the model ignores two-way phase-coupling effects, the particle volume, and particle collisions, it delivers significantly lower particle velocities than measured. The average acceleration is in reasonable agreement for about two thirds of the shown distance.

Hence, the particle motion within the low-velocity section of the nozzle may signifi-

cantly depend on the particle packing and may be characterised not only by gas-particle, but also by particle-particle interactions. This makes the process dependant on both volume and mass fraction of the discrete phase. Downstream of the converging section, the gas expansion leads to a rapid particle acceleration, that increases the average distance between particles. In the diverging section, the primary force on particles therefore certainly becomes the drag force. Nonetheless, this line of thought suggests that particle loading effects in cold spray are a result of the combined effects of particle statistics in the converging section and the gas phase momentum loss within the diverging section. A result can be seen from additional data from FoV4. While the average velocity slightly increased with particle loading at the nozzle inlet, within the jet, the contrary is the case. These trends can again be approximated by linear functions, and figure 5.2 shows the gradients of the average particle velocity with mass loading Z (a) and volume fraction F (b), as defined in eq. 4.3 in chapter 4.4. Again, the velocity was previously normalised by the average velocity of particles at close to zero loading.

Focusing on the gradient with mass loading (a), it should be explained first that the positive gradients for the converging section represent a gain in particle momentum as explained above, possibly an outcome of strong mixing, particle collisions and increased turbulence level, but resulting in more dissipation. At low pressures, this gradient is small. With higher pressure, more momentum (and hence kinetic energy) is gained by particles, which is plausible with stronger collisions. The negative gradients represent behaviour at the nozzle exit and stand for a loss of particle momentum (and energy) due to mass loading. At low pressures, this effect is strong, as the gas flux is rather weak and a unit increment of mass loading can withdraw a significant proportion of kinetic energy from the gas flux during the acceleration within the nozzle. At higher pressures, the gas flow is much more energetic, and consequently, a smaller part of this access momentum flux is transferred to the particle phase. At the same time, collisional losses may be stronger, but the direct effect of this cannot be seen isolated from the trends. Although these observations remain similar with respect to volume fraction, the relative change of the gradient magnitude has interesting implications. While the mass is important for the momentum exchange, the volume fraction is directly connected to the displacement of fluid and the geometric distance of particles. The difference between mass and volume parameters Z and F goes

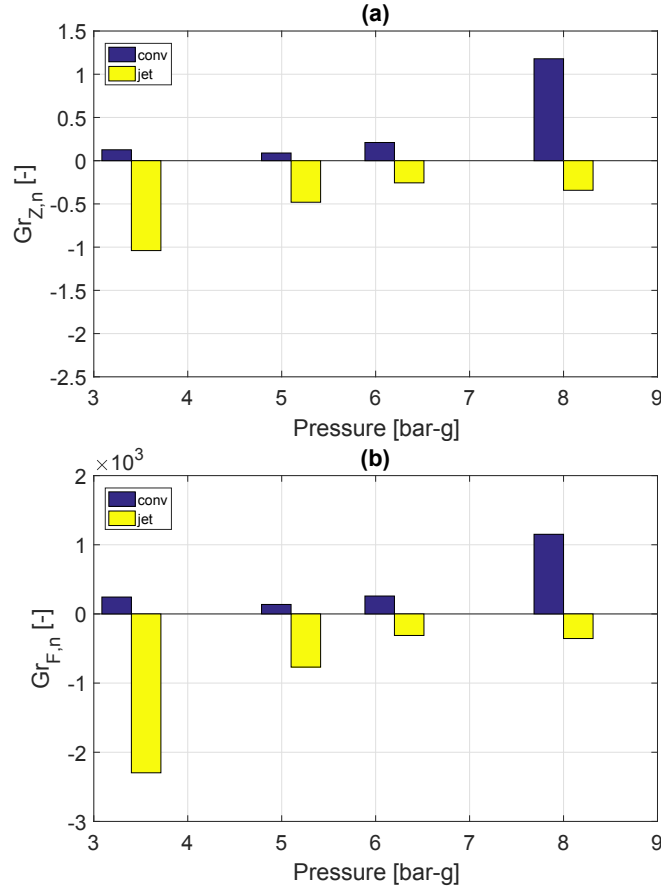


Figure 5.2: Gradients of average normalised particle velocity at nozzle inlet (conv.) and nozzle exit (jet) with mass loading Z (a) and volume fraction F (b)

back to the gas density. This density causes the differences between the two sub-figures (a) and (b). While keeping the gradients at high pressures fixed, the gradients at low pressures scale up. In a volume-fraction-driven loss mechanism, one would expect that the extent of energy and momentum exchange between phases should not be very gas density-dependent. In this case, however, particularly in the jet, this is the case for low pressures, implying that the observed effects are predominantly momentum-driven. On the other hand, an increasing dependency could be inferred for the higher pressure, where the trend in the jet begins to reverse and the gradient with respect to volume fraction remains almost constant. In combination with the high positive gradient within the nozzle for such pressures, it appears logical that importance of the volume fraction begins to rise.

5.3 Injection-dependent particle motion

Further analysis leads towards a full resolution of the velocity distribution inside the nozzle. Moreover, the interactions in such a homogeneous mixture of gas and particles as discussed in section 5.2 certainly differ from those in a still dispersing injection plume. At a shorter distance from the injector, the direction of the particles will be changed. In order to observe the respective consequences, injectors 1-3 are used to inject particles within the nozzle with reducing distance from the restriction. In the definition of configurations and operating conditions in section 3.4.2, this test series is denoted “Internal measurements 2 & 3”.

Some effects in the following section are connected to the fact that the present powders are poly-disperse. As a reminder, it should be noted, that the simultaneous presence of the wide range of particle sizes results in a Stokes number ($Sk = \tau_p/\tau_f$) range. Based on the convective properties of the injection plume, titanium yields a range of $Sk_{Ti}=11-400$ and Stellite-21 a range of $Sk_{St}=21-760$.

5.3.1 Effect of loading on velocity decrease

As described above, the particle mass loading has shown to result in a decrease of the average particle velocity, while the mechanisms behind this are not well understood. Figure 5.3 shows a summarising plot of these declining average velocity trends with mass loading Z for the nozzle internal injections 1-3. It can be seen that the heavier material tends to show a weaker dependence, particularly at higher pressures. Because of the smaller density, the same mass loading of titanium corresponds to a larger volume of particles, which can account for an aggravated influence of volume-dependent losses: particle-particle collisions and turbulence modulation. However, there is no general trend that can be determined to originate from the injector position. The phase-interaction mechanisms can therefore not be significantly influenced by the length of the injection plume in the subsonic section.

5.3.2 Particle acceleration from injection to jet

The full-length study of particle velocities throughout the nozzle is not feasible for all configurations and materials, because the high particle velocities deteriorate the surface

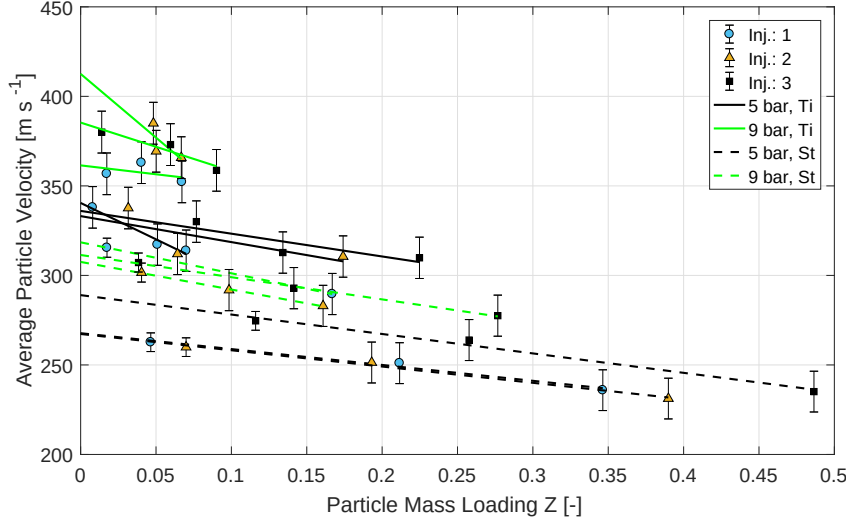


Figure 5.3: Average particle velocity at nozzle exit of NZTR for varying mass loading Z using injector 1-3

quality towards the end of the nozzle. Although replacing nozzle parts is possible in terms of design, such repairs are limited. Therefore, with Stellite, only the main material of investigation is tested for an analysis of the whole field, which leads to several novel findings.

During the steady state operation of the nozzle, the average velocity at a given point does not change, although particles at different times do not generally occupy the same space. This way, an ensemble (or time) average of the overall velocity distribution can be obtained by simply superposing individual measured instances. The local statistical fluctuations of the data then represents the average deviation from this mean and is therefore also an ensemble average of the statistics. At 5, 7 and 9 bar inlet pressure, Stellite particles injected through injector 1, are scattered with respect to position in Figure 5.4. This data summary integrates all measured FoV (1-4) and pulse separation times. The nozzle inlet is at $x=0$, the throat is located at $x=30mm$, and the nozzle exit is at $x=150mm$.

It can be seen that the particles disperse approximately linearly in a subsonic “jet” or plume after injection. The three shown sets exhibit particles dispersing at more or less the same rate and confine into the throat only due to their interactions with the wall (not because of the gas stream). The following section, the nozzle throat, is characterised by a high velocity gradient, evidence of the rapid gas expansion to supersonic velocities. The

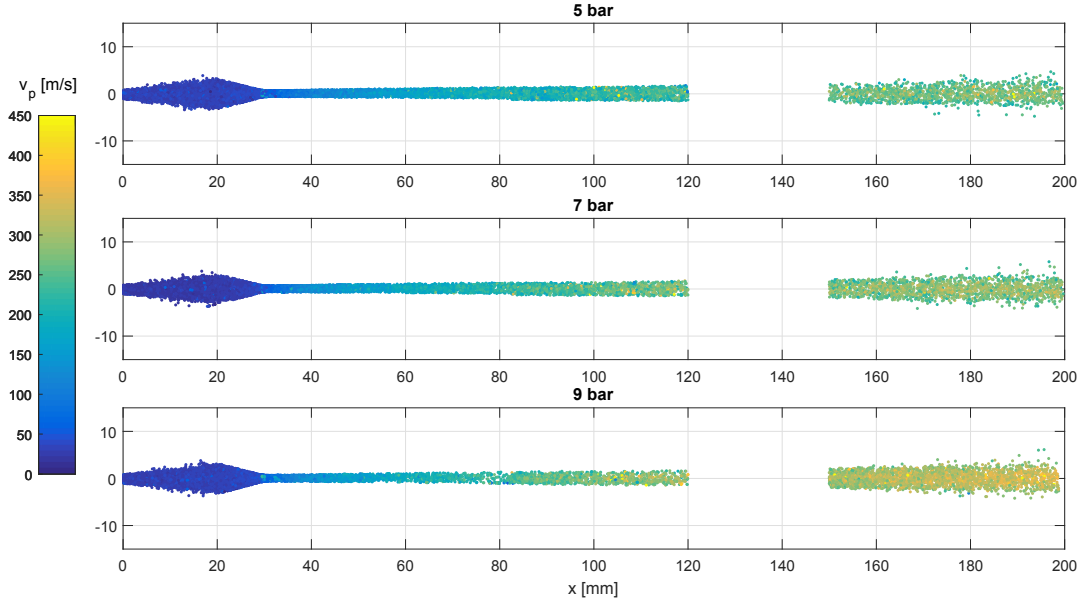


Figure 5.4: Spatial evolution of particle velocity for Stellite-21 with increasing pressure within the full nozzle, using injector 1

maximal gradient is difficult to quantify due to the uncertainty invoked in differentiation of dynamic (*i.e.* noisy) data. Over a span of 5mm within the restriction, where the gradient is highest, the spatial velocity gradients can nonetheless be determined accurately. Based on the substantial derivative (appendix p.236), the particle accelerations within this region can be inferred to be 4.4 , 6.3 , and $8.7 \cdot 10^5 \text{m/s}^2$ for the increasing pressure, as shown in figure 5.5. Due to the span over which these values are deduced, it should be noted that the peak accelerations may be slightly higher. However, as this is the first report of such accelerations, and hence implicit forces, within the restriction of a cold spray nozzle, future studies can aim to refine the values and use the insights to amend empirical drag laws. The figure also displays the analytical solution by Grujicic *et al.* [79] for a $30\mu\text{m}$ particle,

$$v_p = v_c \sqrt{\frac{3C_D \rho_c x}{2d_p \rho_p}} \quad (5.1)$$

$$\frac{dv_p}{dt} = \frac{v_c}{2} \sqrt{\frac{3C_D \rho_c}{2d_p \rho_p x}} \cdot v_p, \quad (5.2)$$

which is derived on the assumption of constant gas properties and a constant drag coeffi-

cient. Since, in fact, all quantities undergo significant change, the calculation is inadequate and it can be seen that the changes in the gas phase are directly reflected in the particle velocity, which leads to excessive values. Moreover, the local change in drag coefficient plays a key role in the particle acceleration. The improvement of this discrepancy through more elaborate CFD is shown in chapter 6.3.

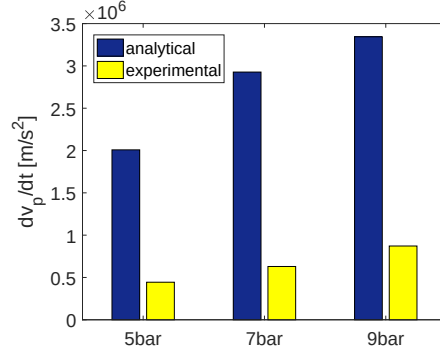


Figure 5.5: Average peak acceleration of Stellite-21 particles at increasing pressure within the nozzle throat, derived for $x=30$ - 35mm , in comparison to analytical model Grujicic *et al.* [79]

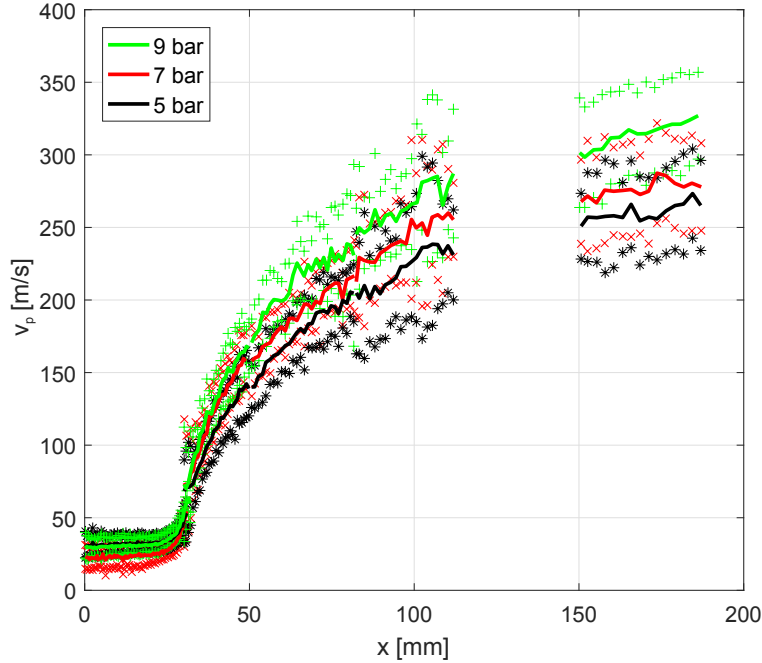


Figure 5.6: Internal particle velocity evolution along the nozzle and jet (Stellite-21, Inj.1)

Following this peak acceleration, the velocity has a regressive trend, of which the last quarter of the length (not measured due to the location of bolts) contributes the least.

As the particles accelerate, the particle Reynolds number and hence the drag force on the particle reduce, leading to the profiles in figure 5.6, where solid lines display the local average and the scatters the local standard deviation of particle speeds.

This drag reduction is, as expected, most prominent at 5bar pressure. Despite this and the strongest over-expansion, the gas does not slow down the particles measurably over a long distance in the jet ($17h_e$). At the higher pressures, particles even continue to accelerate and reach their maximal velocities at 40-50mm ($13-17h_e$) SoD. For this to occur, the gas velocity must still be well above the particle velocity because of a prolonged supersonic potential core. While the velocity increases within the nozzle, so does the local range of velocities, because the initial unordered particle motion is amplified during acceleration. Interestingly, from the final nozzle section through the jet, the velocity range remains approximately constant. Favourable effects of the particle-shock interactions can be one reason: as the particles pass into the high pressure region, smaller particles that potentially contribute significantly to the widened dynamic range are synchronised with the mean flow.

5.3.3 Effect of injection plume on particle motion

From previous aspects we could see only small changes in the dispersion of the injection plume with pressure level. In more detail, the particles disperse in the subsonic part in the following way. Since the gas accelerates in the converging section, the injection plume is not a mere free jet in a co-flow, but a highly constrained jet in close vicinity to adjacent walls. Therefore, it would not follow the relatively well understood physics of such a free jet. Trends that have been reported in fundamental studies [71] can however be used as guides. With increasing Sk , the particles in a free jet would shift the approach to a self-similar region significantly downstream. The high Stokes numbers of present particles and the accelerating co-flow prevent any deceleration and development of similarity. Nevertheless, the three shown sets exhibit particles dispersing after injection at more or less the same rate. Interestingly, this spreading rate is still comparable with a free jet, as we find the plume width to grow about 3 times over a distance of $10d_{inj}$. A visualisation of this can be referred to in figure 5.7. The undisturbed spreading of the particle stream prior to wall collisions indicate that its confinement towards the centreline takes place due to

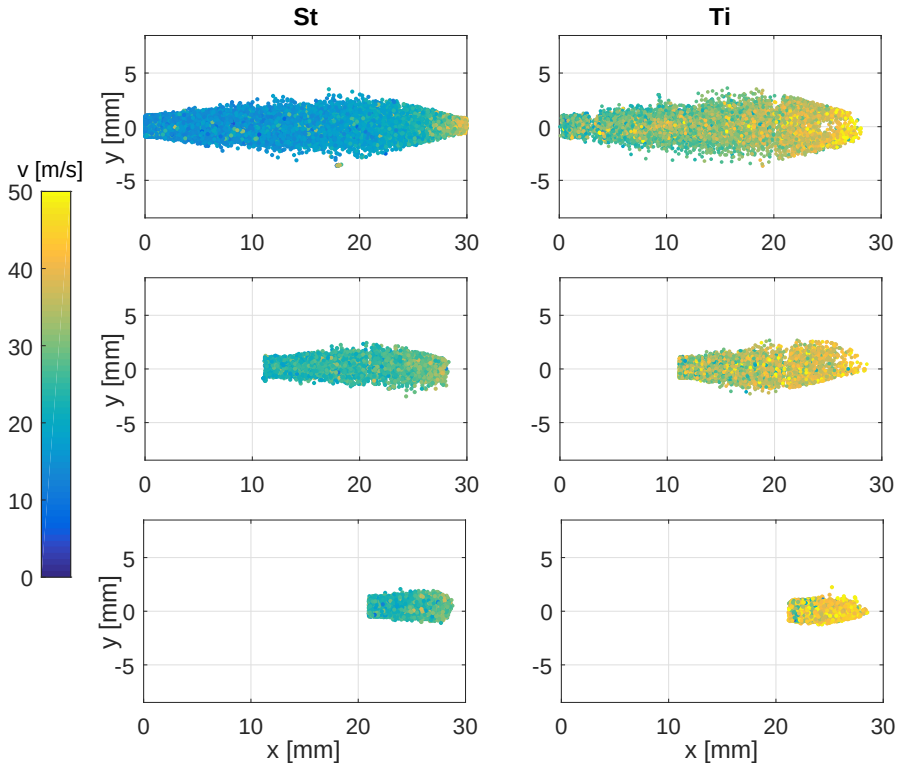


Figure 5.7: Comparison of the injection plumes of injector 1-3 using Stellite-21 and titanium ($p_0=5\text{bar}$)

the interactions with the nozzle wall (an important aspect for clogging risk, see section 5.5), and not due to the main-line flow that entrains the plume towards the centreline. Reducing the distance of the injector exit to the nozzle throat reduces dispersion, because it leaves less space for the spreading, and with a straight injection into the throat, the plume only comes into contact with the fast part of the main line flow. Injector 2, within the intermediate part of the converging section, exhibits some tendency of asymmetric plumes. Titanium exhibits significantly higher injection velocities than Stellite-21 (up to factor of 2), which corresponds well to their material density ratio. It also shows stronger spreading (up to factor of 1.2). In all cases the injection velocity increases with decreasing distance from the restriction, due to a drop in pressure and hence excess pressure in the feeder line. When increasing the loading, the dispersion increases slightly (respective velocity scatters are appended p.7.2). The increase in loading also changes the velocities, which is analysed in longitudinal and cross-sectional velocity profiles in the paragraphs to follow.

The cross-sectional particle velocity profiles of the injection reveal some interesting

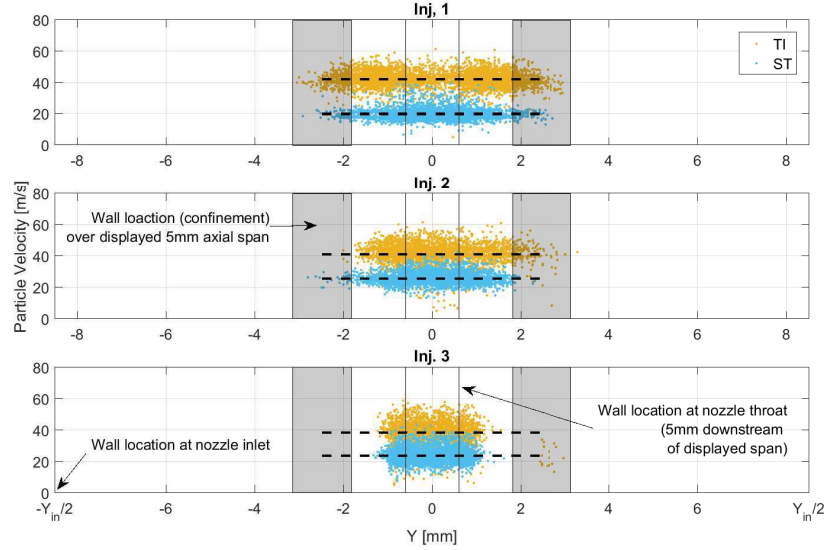


Figure 5.8: Particle velocity profiles in the cross-sectional direction y for injectors 1-3. The profiles display particles between 10-5mm upstream of the nozzle throat, the dashed lines are averages of these profiles. The greyed out area depicts the nozzle wall location over this span, the solid lines display nozzle throat width for comparison

characteristics about the distribution of particles, as displayed in figure 5.8. Here, the cross-sectional profiles of particles located between 10 and 5mm upstream of the nozzle throat are shown. The nozzle internal injections 1-3 are given separately. Injector 1 is located the furthest upstream ($10d_{inj}$), while injector 3 is located just upstream of the shown particles. In this illustration, the cross-sectional y -coordinate is greyed out where the nozzle wall is located over the given span, and the nozzle throat width is displayed for reference. This indicates that the majority of particles undergoes wall interactions in case of upstream injection, while only half of the particles can interact with the nozzle in case of injector 3.

The figure confirms the distinct velocity levels for the two materials, adding flat particle velocity profiles. At the injector needle, the profile is almost constant over the cross-section with no quantifiable gradients at the needle wall. The shortest length of the needles' constant diameter section is $25d_{inj}$. A fully developed flow of the gas phase can hence be assumed in all cases. In spite of this, the particles cannot be meaningfully represented by a power law. The high Stokes numbers of particles explain exactly this effect of flattening profiles [71], as increasing Sk cause decreasing wall gradients and increasing overall lag

to the gas velocity profile. In the present poly-disperse stream of a wide range of Sk , the trend seems to be continued and dominated by the high Sk -particles from the range. Thirdly, this flat profile is maintained throughout the converging section, as figure 5.8 shows profiles that remain close to constant for all three injectors.

It is important to state that a necessary condition for this effect to take place is a low change in velocity lag of particles at the plume edge, implying a very weak shear layer of the gas phase. This means that the co-flow and plume velocity are very similar. For process conditions, this confirms that the ratio of main and feeder line pressures and flow rates are well set. As the purpose of the feed line flow is to inject the particles, while the purpose of the main line flow is to deliver the majority of the process gas, similar velocity levels depend on the flow rate ratio of these two lines (bleed ratio), which is regulated to be 0.1 in all cases.

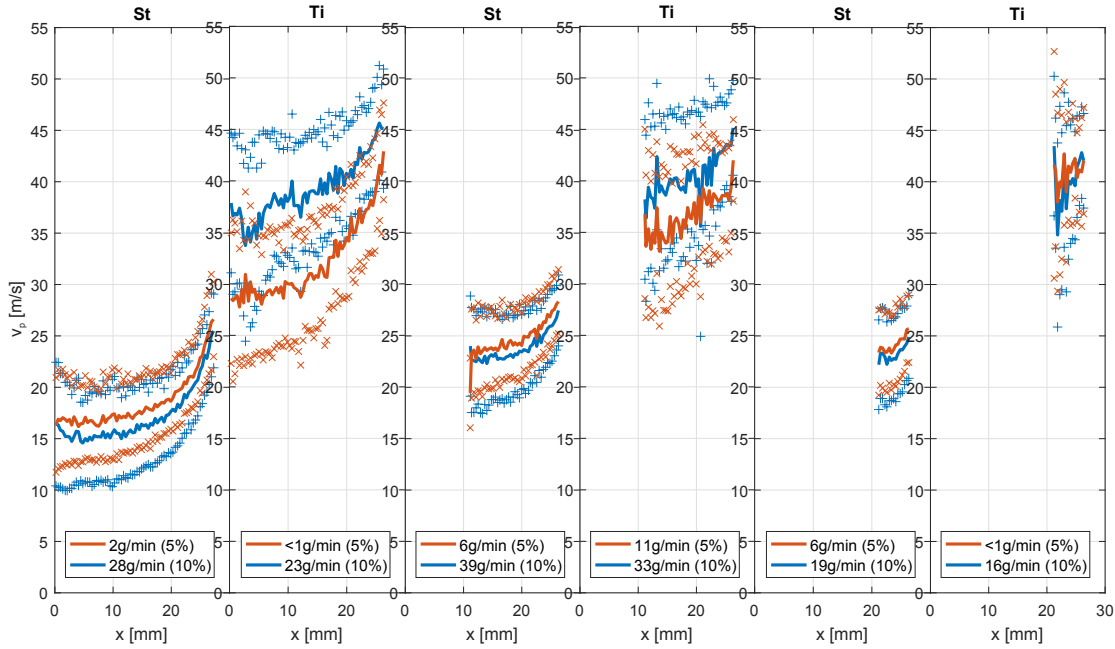


Figure 5.9: Longitudinal particle velocity profiles at injection, comparison of Stellite-21 and titanium with injection position at increasing feed rates. Solid lines are moving average, markers indicate local standard deviations

Figure 5.9 shows a collection of the axial velocity profiles in the converging section for the three injectors and both materials. Moreover, two loading cases are reported to compare the effect of high particle mass in some more detail. Again, the solid line is the moving average of the particle velocity magnitude, while the markers indicate the local

standard deviations from this mean.

The lighter material always exhibits stronger fluctuations, but both materials are injected at increasing velocities with the downstream shift of the injection location as observed before. Here, it can be seen in addition that this increase due to lower static pressure of the gas is not more than 30%.

The injection shift also causes a difference in the local velocity gradient. Injecting the particles further upstream provides a better mix of main process gas and injection plume, which leads to a more continuously increasing velocity. Consequently, the acceleration appears to shift downstream slightly with increasing injection location. The most interesting point is the strong difference in loading effect. Similar to observations of Stellite in the homogeneous mixture case, where the high Sk -particles have sufficient time to interact (see section 5.2), such an velocity increase due to loading can also be observed at smaller injection distances. This is however confined to particular cases: on Stellite, the increased loading has a small effect, expressed by a slight drop in average velocity and a slight increase in dynamic range. This heavy material appears to be less prone to enhance its random motion when injected into the nozzle, as compared to when fully mixed with the main process gas. In contrast, titanium shows massive loading effects of this character and has significantly increased average velocities under higher feed rate. This decreases with a downstream shift in injection point: roughly 20% for injector 1, 10% for injector 2 and no noticeable difference for injector 3. Physical explanation for this is analogue; volumetric effects cause a randomisation of particle motion, and particles of the rather low end of the Sk -range are overly activated, causing an increase in both variance and average of the velocity. Consequently, materials under different Sk -ranges predominantly inject differently due to their material density, and secondly under increasing loading. The third most important aspect is the injection location, and finally pressure plays a subordinate role in this respect (if gas conditions remain well-set).

Integration of the velocity data along the x-axis leads to the time that particles statistically require to travel through the low-velocity section. It is an interesting quantity, as this residence time directly corresponds to the particle heating time when gas heating is employed. In many applications, this particle pre-heating is a crucial point and injection location is adjusted accordingly. Figure 5.10 shows a comparison of the statistical

residence times with both a mean value and a range. The three injector locations are compared, which plausibly cause the residence time to decrease. In addition, a low and a high loading case are shown. As explained above, the velocity distribution is affected by this parameter and, as a result, the residence time changes accordingly. While the feed increases, particles of heavy materials may be heated more, whereas light materials may receive less thermal input. At this point, the validity of models in the computational field should be referenced (see chapter 6). Because there is no solution to particle in-flight temperature measurements, all thermal aspects in CS are fully modelled and rely more than any other aspect on the model adequacy.

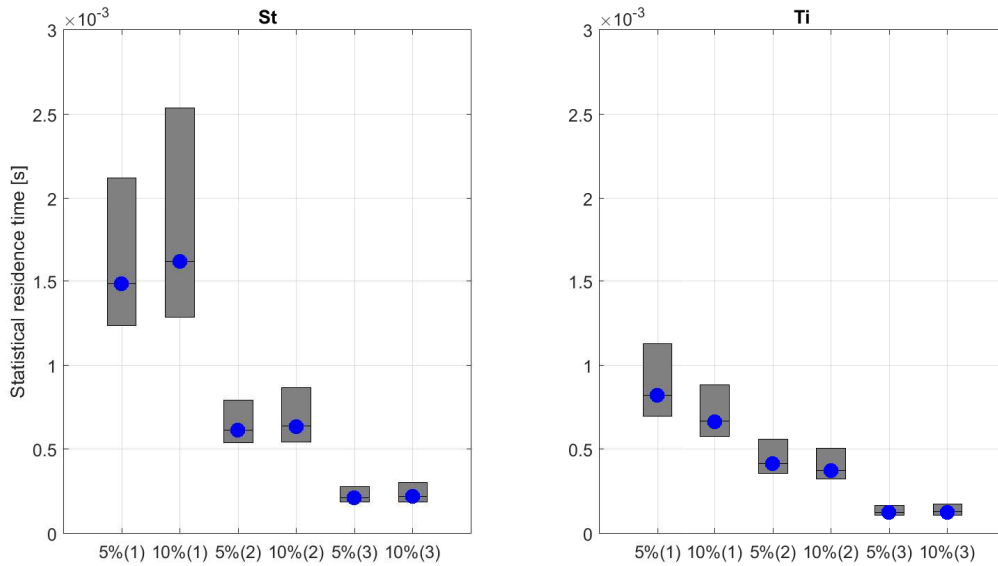


Figure 5.10: Statistical residence times of particles within the low-velocity section using injectors 1-3 at low and high feed rate

A similar comparison of the residence time under changing pressure, as finally displayed in figure 5.11, reveals that increasing pressure causes a slightly higher velocity, but this is sufficient over the long distance in case of injector 1 to significantly decrease the particle residence time. As the injector is prolonged, this effect diminishes quickly.

5.4 Statistical analysis of particle properties

Aiming for a better understanding of this particle behaviour, the statistics of the data set is analysed in more detail. It is important to note that the introduced concepts are

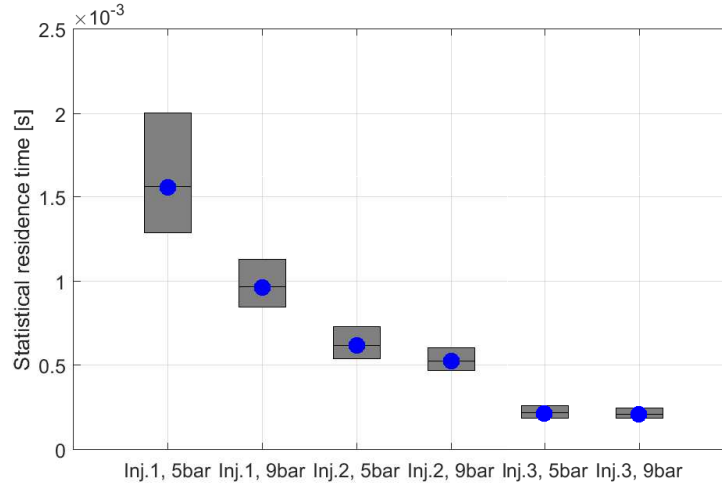


Figure 5.11: Statistical residence times of particles within the low-velocity section using injectors 1-3 at increasing pressure

novel and were never done for any CS flow. As such, physical explanations for observed phenomena have the character of attempts. Future studies in the field are required to verify (or falsify) these concepts through various studies.

In this section, we use the fact that the particle profiles are flat and the statistics do not vary strongly with y-position. The influences of the injector location, the material and pressure are subsequently investigated. In addition to the particle velocity, the role of the trajectory direction is addressed probabilistically. By statistical behaviour, we denote the time-invariant deviation from the average. The local standard deviation, as included in above analysis, does not contain all extractable information, as it does not include how deviations from the mean are distributed within the present range. A useful approach is the derivation of normalised probability density functions (pdf) of the particle quantities. At the example of the particle velocity magnitude, this should be explained here.

The goal is to observe the relative distribution of data among the present values in a local region of the nozzle. Therefore, the data in this region is isolated, and then non-dimensionalised by its average value, in order to obtain a data range with respect to the overall average level. The normal pdf now represents a distribution of the amount of data that occupies a specific value within the dynamic range (ratio of maximal and minimal value of the data). This probability density is calculated relative to the overall amount of data (normalised) such that the dependence on the exact number of identified particles is eliminated. The results can, in principle, take any shape, but specific cases can be

identified as guides. For particle trajectory angles, one can expect a normal distribution about zero, since the particles go straight on average, but have symmetrical, statistically distributed inclinations. Similarly, with the assumption that the drag force is the predominant influence on the particle velocity, one can expect a log-normal distribution around one for its statistics, since any difference from the average velocity would be caused by the particle size, which is by itself log-normally distributed.

5.4.1 Velocity magnitude

Figure 5.12 shows the pdf of the velocity magnitude of particles exiting from the injector needles at 9bar. The lines indicate a log-normal fit for each set, which is based on the aforementioned theoretical case.

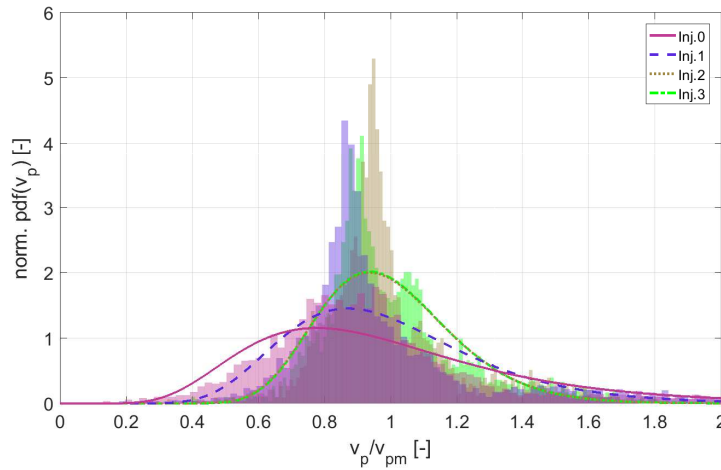


Figure 5.12: Probability density functions (pdf) of normalised particle velocities at injection point for inj.1-3 and nozzle inlet at inj.0, lines are log-normal fits.

Generally, the actual probability distributions are narrower than their log-normal fits. In other words, a first interesting observation is that the observed particles tend to have more similar velocities than they had due to drag alone. Peaks indicate that an increased number of particles occupy similar velocities, which cannot be explained solely by particle size and mean drag. A peak forms if slow particles are accelerated more than due to drag and comparatively fast ones are slowed down accordingly. A possible interpretation of this behaviour is a particular kind of collisional motion, in which particles are directed in the same direction and hence tend to influence each other longitudinally. This can be the case

within the injector for example, or close to the centre line when the trajectory angles are mostly close to zero. Another kind of interaction may cause a flatter probability density distribution, namely if particle directions are also more random and get re-directed more in case of collisions. This latter can be observed in case of injector 0, where the particles have dispersed and occupy the space in the nozzle channel evenly. As a result, this case has a more flat and stretched out distribution than the other injectors. Nevertheless, also in this case the velocities accumulate in the centre below the average. The other cases exhibit higher peaks at similar location with a tendency to approach the average value for longer injector needles. This also means that the particle phase exits the needle with a relatively narrow distribution and that dispersion of the particle phase stretches it accordingly.

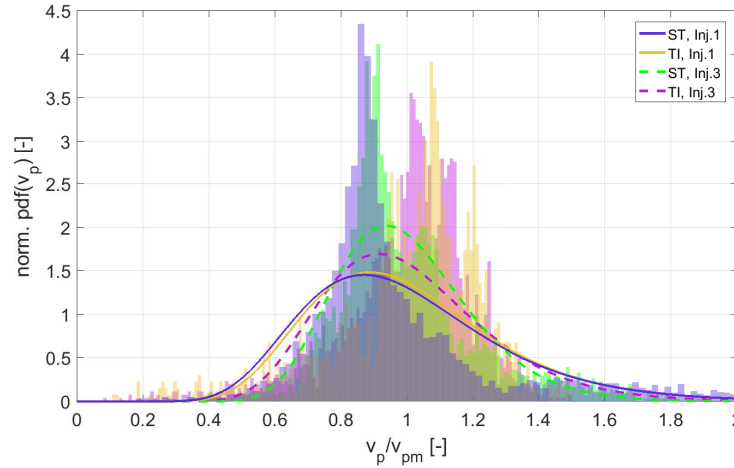


Figure 5.13: Pdf of particle velocities at injection point for inj.1 and 3 for Stellite-21 and Titanium.

Comparing the materials analogously, as in figure 5.13, we can note a significant difference. While peaks of the heavier material lie under the average, because the data falls off quickly towards the low velocity end, the light material has a different character. It exhibits a much higher level and rather shallow slope of low velocities, with a peak at the high velocity end above the average. It therefore departs more strongly from the log-normal notion and is increasingly influenced by more complex mechanisms, as attempted to be explained above; particles of Titanium appear to experience longitudinal collisions more strongly.

Figure 5.14 shows the pdf of the velocity magnitude of both materials at the injection port and when entering the throat (just prior to their strong acceleration). Apart from

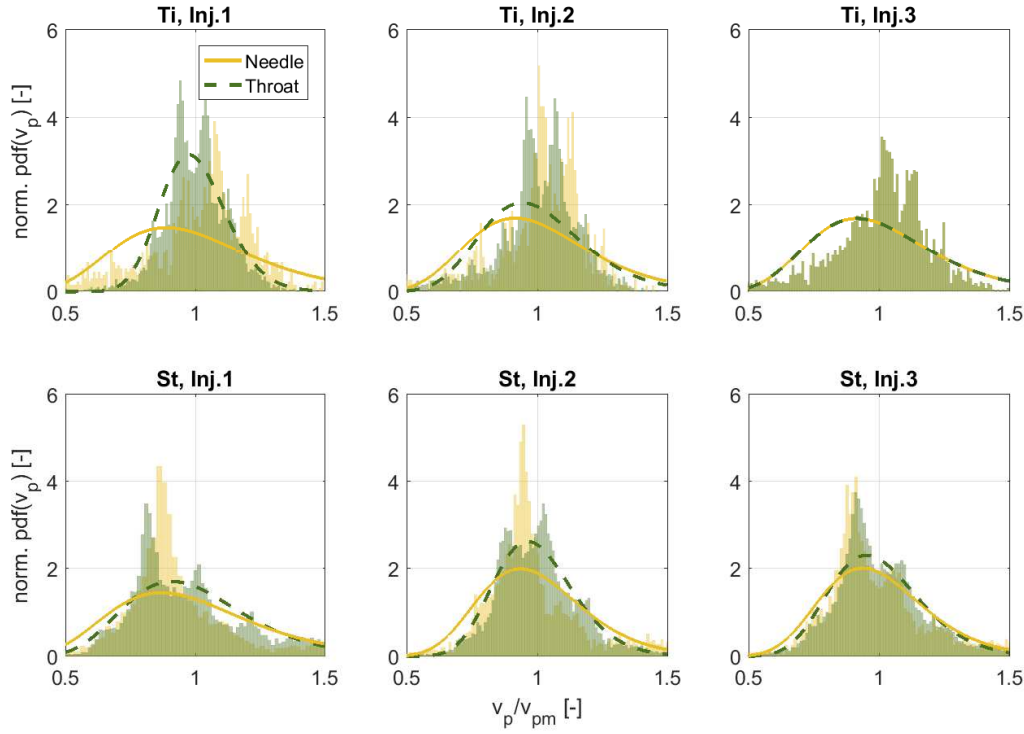


Figure 5.14: Comparison of Stellite-21 and Titanium velocity pdf change from injection to throat.

a more general confirmation of the previous findings, it can be seen that the peaks in the probability density function remain of similar order while the particles move through the low-velocity section. This indicates that the longitudinal interactions are maintained, and not significantly eliminated. While the particles are spreading, the interactions relax and the peaks hereby slightly shift towards lower fractions of the average velocity. This plausibly reduces with smaller injection distance from the throat.

Figure 5.15 aims to summarise the influence of the injection on the statistics for the different pressure levels. It gives data for the throat, representing the point where drag becomes the prominent interaction mechanism.

It can be seen that the shift of the injection in the downstream direction, causes the dynamic range of the velocity to reduce. This means that particles deviate more from the mean in relative terms, if they are injected sufficiently far upstream to have time to experience noticeable non-drag effects within the nozzle. The influence of increasing pressure is smaller, but noticeable for injector 1: more particles have close-to-average velocities at higher operating pressure but a similar normalised span. This is less for

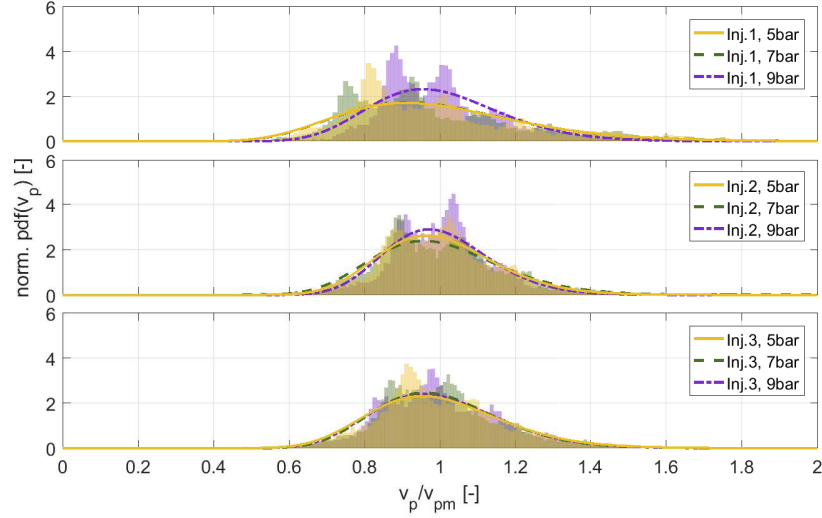


Figure 5.15: Probability density functions of normalised particle velocities at nozzle throat, comparison of Stellite-21 through injectors 1-3 with increasing pressures, lines are log-normal fits.

injector 2, and absent for injector 3. If the injection takes place just upstream of the throat, particles do not disperse strongly and the character of the velocity range does not depend on the pressure level. In this respect, the higher pressure affects the phase-interactions mostly during dispersion, not noticeably inside injector needle.

5.4.2 Trajectory angle

These velocity magnitude observations go hand in hand with information about the trajectory angle. Here, this refers to the instantaneous trajectory direction with respect to the the x-axis. The average of such angles is approximately zero as the process is overall symmetric about the nozzle centreline.

Figure 5.16 is a summary of the normalised angle pdf, at 5 and 9bar, at the injection point just downstream of the needle, at the entry to the restriction, and at the nozzle exit in the supersonic jet. The fit functions are normal distributions around the origin for this variable. As injector 0 is located upstream of FoV1, this case is again evaluated at the nozzle inlet. For this reference flow, we can see that the trajectory angle is broadly randomised. As the flat distribution shows, trajectories at zero angle are not much more likely than those with noticeable inclination. In fact, the distribution is sufficiently wide to cross the maximal angle that could be evaluated by the cross-correlation (due to the

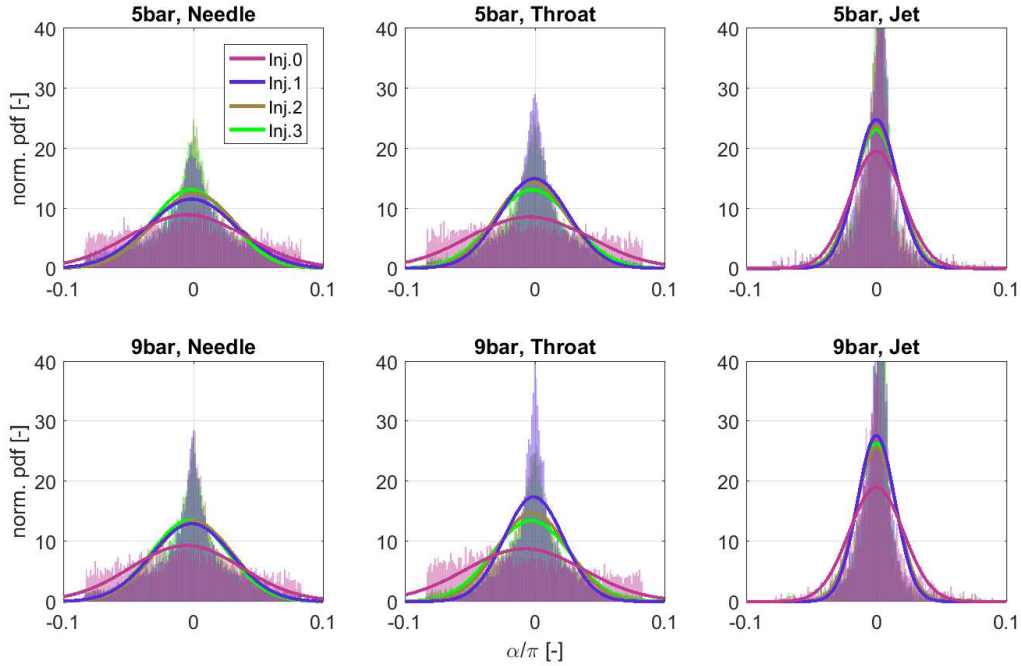


Figure 5.16: Pdf of particle trajectory angle of Stellite-21 at injection, throat and nozzle exit, comparison of injectors 0-3 with increasing pressures

window pre-shift). This broad character is reduced downstream towards the throat and supersonic jet, but remains noticeable. This injection type, allowing for full mixing of the main and feeder flow, thus has a detrimental influence on the directionality of the flow and consequently on the particle dispersion. This effect may also contribute to the velocity losses observed for this configuration due to increased collision probabilities. Apart from this, the nozzle internal injections 1-3 show a very similar form of angle pdf, slightly declining for injector 3. The peak around zero is high as compared to the Gaussian fit, which signals that a large fraction of the particles are directed along the longitudinal direction. Ejection from the long needles clusters the majority of particles well aligned with the centreline, which substantiates previous explanations about interactions. It seems possible as an explanation for this behaviour that particles are much more likely to interact with each other in the longitudinal direction and this way “synchronise” their velocity. Moreover, it can be seen that the pressure statistically straightens the flow of particles, despite the relatively similar velocity pdf, which has a positive influence on dispersion. The downstream evolution to the throat and jet reveals a similar effect. Due to the strong acceleration, the particles are increasingly aligned along the longitudinal direction.

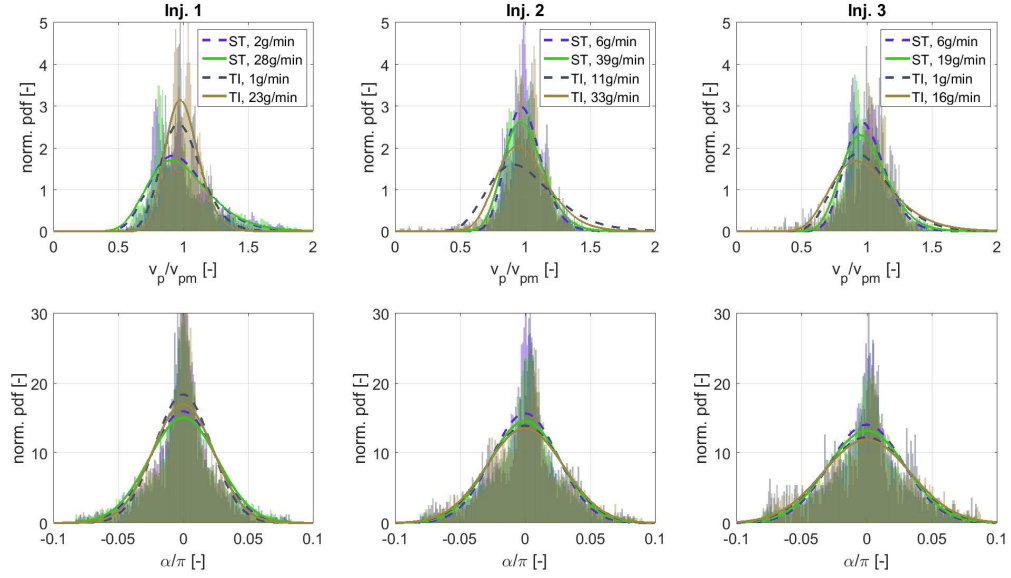


Figure 5.17: Probability density functions of particle velocities and angles at nozzle throat, comparison of injectors 1-3 with increasing feed rates

Figure 5.17 brings together velocity and trajectory angle pdf under varying loading conditions for particles entering into the throat. Here, it can be seen, how the two quantities are connected to the amount of fed particles. As reported above, when shifting the injection point (1-3), the trajectory direction becomes more randomised, while the velocity magnitude tends to be more log-normal. When additionally increasing the particle feed rate, the velocity of Titanium is most strongly affected at injector 1 and the least at injector 3, while the Stellite velocity reacts similarly in all injections. This is reflected in the trajectory angle, as the amount of small values reduces and the amount of larger values increases when feed rate is increased, however more of such inclined trajectories are found for Titanium. This is an additional indicator that the path direction is in general more randomised when loading is increased, but the lower *Sk*-range material is more prone to the “synchronisation” of particle velocities due to particle-particle collisions, which depends on injection location. For a deeper understanding of this effect, future studies could aim for a finer resolution of feed rates and a wider selection of materials and use this analysis as a starting point.

5.5 Discussion of particle statistics in the context of nozzle clogging

The methods used in sections 5.2-5.4 have lead to some new ideas about the multi-phase character of the flow. It is evident that this opens doors to a vast selection of new possible analysis and insights. Clearly, it is important to transfer the findings to applications whenever possible, *e.g.* by using them for numerical models. It is, in addition, very useful to directly address current operational challenges in CS, that are related to the unknown aspects of the nozzle-internal particle behaviour. Because nozzle clogging is the most prominent example of such challenges and widely encountered, it is addressed separately in some more detail in this section.

5.5.1 Wall normal velocity

As summarised in chapter 2.4.2, literature on nozzle clogging indicates that the main influences are thermal softening and the dispersion of particles. The latter is mostly said to be important due to the wall normal velocity component of the particles. This velocity tends to stick particles to the nozzle wall, similarly to the effect during coating, only in a more sparse manner. This is a process that small particles may be more prone to, as they soften easier and reach higher velocities sooner. The wall normal velocity naturally remains relatively small. The present nozzle is a very specific design with respect to surface properties and material, and operating conditions yield comparatively small speeds. Therefore, the absolute velocity levels may be difficult to transfer to other studies. However, the general trends lead to indicators of the particle behaviour, which may be useful as guidelines.

In this respect, the observed nozzle-internal particle velocities are filtered according to their location and decomposed with respect to the wall. Particles in the wall-proximity of about 0.5mm are considered close enough to reach the wall within short distances. The velocities of these particles are decomposed into a wall-normal and a wall-tangential component, $v_{n,w}$ and $v_{t,w}$. The normal components are scattered in figure 5.18 along the nozzle wall, complemented by the sliding average (lines) for increasing pressures.

Particles emerging from injector 1 start interacting with the nozzle wall about 19mm

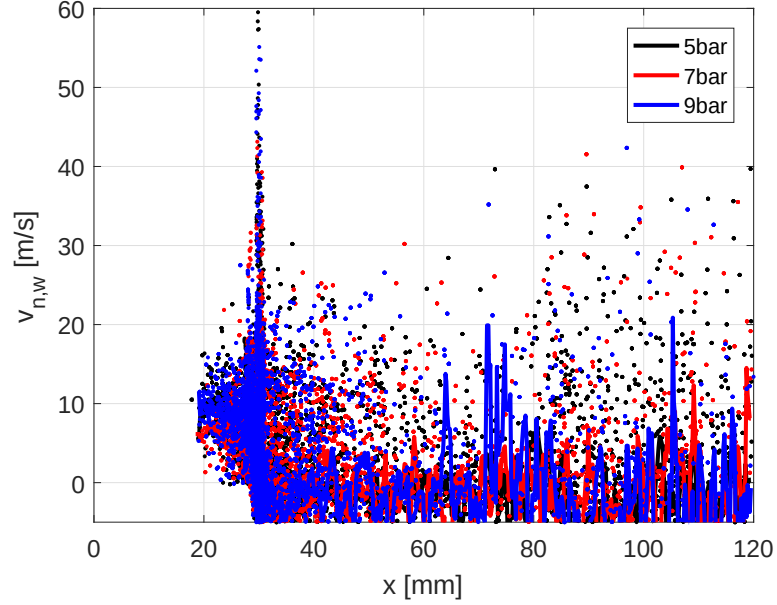


Figure 5.18: Wall normal velocity components of particles in wall vicinity throughout the nozzle with increasing pressure

downstream of the nozzle inlet. The figure depicts that the majority of particles begin impacting the nozzle wall at about 10m/s normal speed. This relatively high with respect to the overall speed level in this section, as the nozzle wall is steeply inclined inwards, while particles tend to disperse and therefore have a positive angle. This dispersive angle, in combination with the beginning acceleration leads to a brief increase of $v_{n,w}$ at the nozzle throat, that certainly contributes to material build-up in cases where clogging was observed within the throat. The velocities then sharply decline to an overall negative average level, as the wall contour now diverges outwards. Nevertheless, the acceleration and dispersion of particles leads to a significant amount of particles with similar or even higher wall-normal velocity than at the throat. This tends to increase towards the exit due to the overall acceleration of the particles. In cases of nozzle clogging in the diverging section, this fact may have played a role, but it seems unlikely as a full explanation, because the normal component does not overly increase while temperatures drop.

Figure 5.19 shows an analogous average for the converging section of the nozzle for several injector/feedstock material combinations. The upstream injection (inj.0) leads to an even and relatively low velocity level of wall collisions, which increases towards the throat. Because the particles are homogeneously distributed, their average impact angle is dominated by the randomness of the particle direction, not the dispersive motion.

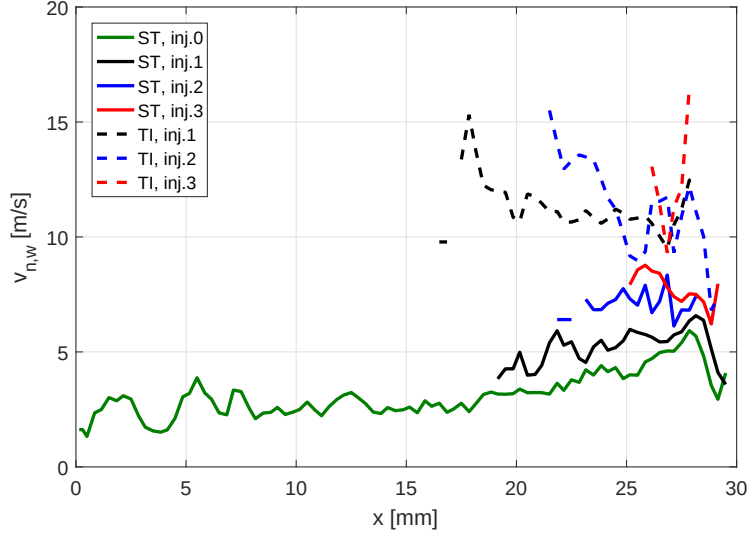


Figure 5.19: Clogging comparison: wall normal velocity of particles in converging section using different injectors and materials

Therefore the impact is less drastic as in cases of other injectors. In fact, shifting the injection point downstream leads to a trade-off between two opposing trends: the values of $v_{n,w}$ increase due to this effect, while the wall interactions start further downstream. A justified assumption would be, that the velocity level is more important and hence injection far upstream is favourable against nozzle clogging. However, according to the particle dynamics analysis in section 5.3, it can be seen that the main differences are caused by the materials, titanium and Stellite. The wall-normal velocity of the lighter material is significantly higher in this region, and due to the stronger dispersion, wall interactions start further upstream. Conceptually, the higher injection velocity is clearly disadvantageous for clogging risk.

5.5.2 Tangential velocity

Reported cases of nozzle clogging in the diverging section are not fully explainable with thermal softening and high wall-normal velocities in the throat. The previous section showed that also in the diverging section, the wall normal component is not likely to significantly surpass the values at the restriction. In combination with lower temperatures, the nozzle-internal deposition of particles further downstream seems unlikely. However, the tangential components naturally make up the most significant part of the velocities as shown in figure 5.20.

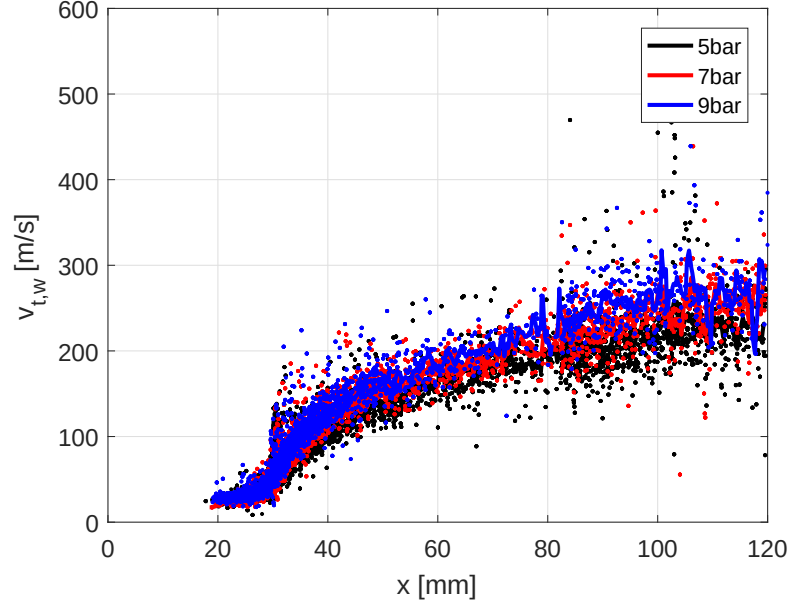


Figure 5.20: Wall tangential velocity components of particles in wall vicinity throughout the nozzle with increasing pressure

As analysed in previous sections, the velocity and the dispersion increase within the diverging section. The notion to consider only the wall-normal component is based on the similarity to impact onto the substrate. It however neglects the fact that the microscopic interaction between nozzle wall and particle is very different, when considering such steep impact angles, and the particle deformation may have a very different character in such cases. Without attempting explanations of this particle deformation at this stage, attention should be raised to a potential field of study of different particle-wall-interaction types. Some studies in non-CS contexts have shown that projectile-target impact is completely different under high angled contact [190], in which in particular shear plays a distinguished role. In the context of clogging towards the exit section of the nozzle, further investigations are required to connect possible shear deformation patterns to the low angle, high velocity impacts. It seems likely that the ratio of particle size and surface roughness is of particular importance under such high tangential velocities. This also agrees well with the fact that such a high velocity clog type is usually observed after long spray operations and with relatively hard materials, such that clogging follows a (possible) previous surface roughening procedure. It is not the scope of this study to investigate details of such effects.

5.5.3 Particle-wall collision probability

According to the presented velocity findings, it is useful to distinguish the two main types of nozzle clogging, that are referred to here as low velocity and high velocity clogging. As such, low velocity clogging appears in converging section close to the throat and is connected to higher temperatures and higher average wall-collision angles. High velocity clogging is the oblique wall-collision at the end of the nozzle (potentially with different deformation).

A useful analysis is the derivation of a likelihood of particle-wall interactions, assuming that the risk of clogging is higher if more particles come into contact with the nozzle wall in the two distinct sections. The first step in this line of thought is to check which percentage of particles will collide with the nozzle wall, based on the assumption that their instantaneous trajectory orientation is frozen. In the converging part, this is a good approximation, as particles are not diverted by the gas significantly, as analysed before. In the diverging section, the approximation could be weaker due to the acceleration. However, if all particles within the diverging section are taken into account, the derived numbers should give a good indication of the real probability, since the acceleration reduces strongly towards the end of the nozzle.

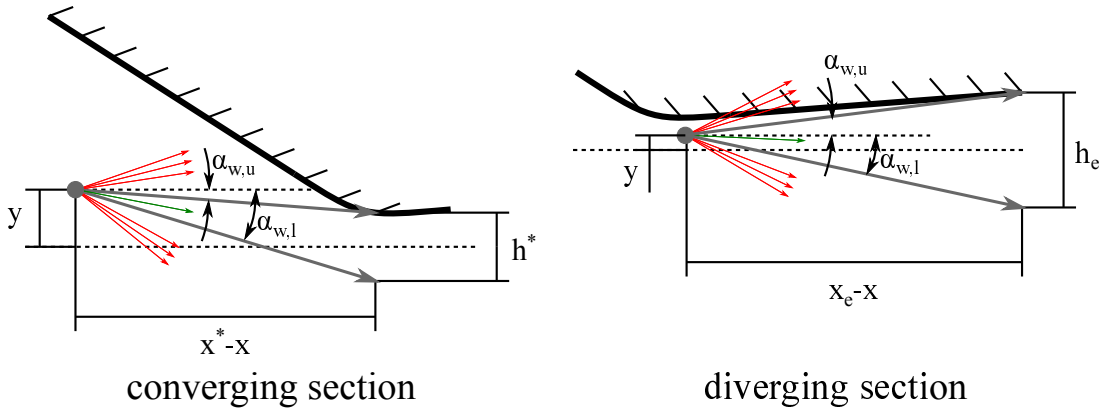


Figure 5.21: Derivation of particles on collision paths with nozzle wall. If trajectory angle is between grey arrows, the particle is assumed to avoid collision. Particles that do not collide in converging section can still collide in diverging section.

In figure 5.21, the derivation of the percentage of affected particles is explained. In the converging section, all particles with a path that directly leads through the nozzle throat are assumed to avoid contact with the nozzle wall, and therefore avoid the low-speed nozzle

clogging risk. Similarly, the particles that have a path direction leading straight through the exit of the nozzle are assumed to avoid wall contact in the diverging section. All particles in the diverging section can potentially interact with the wall, including those, that already impacted on the converging channel. The trajectory angle of each particle α_p has upper and lower boundaries $\alpha_{w,u}$ and $\alpha_{w,l}$ depending on particle position (x,y) . As summarised in eq.5.3-5.5, the particle goes sufficiently straight to avoid the wall, if the orientation is within these boundaries, while outside of the boundaries, the particle will collide either with the upper or the lower wall.

$$\text{Conv. section} \quad \alpha_{w,u} = \text{atan} \left(\frac{\frac{h^*}{2} - y}{x^* - x} \right) \quad \alpha_{w,l} = \text{atan} \left(\frac{-\frac{h^*}{2} - y}{x^* - x} \right) \quad (5.3)$$

$$\text{Div. section} \quad \alpha_{w,u} = \text{atan} \left(\frac{\frac{h_e}{2} - y}{x_e - x} \right) \quad \alpha_{w,l} = \text{atan} \left(\frac{-\frac{h_e}{2} - y}{x_e - x} \right) \quad (5.4)$$

$$\text{Wall collision path} \quad \boxed{\alpha_{w,l} > \alpha_p > \alpha_{w,u}} \quad (5.5)$$

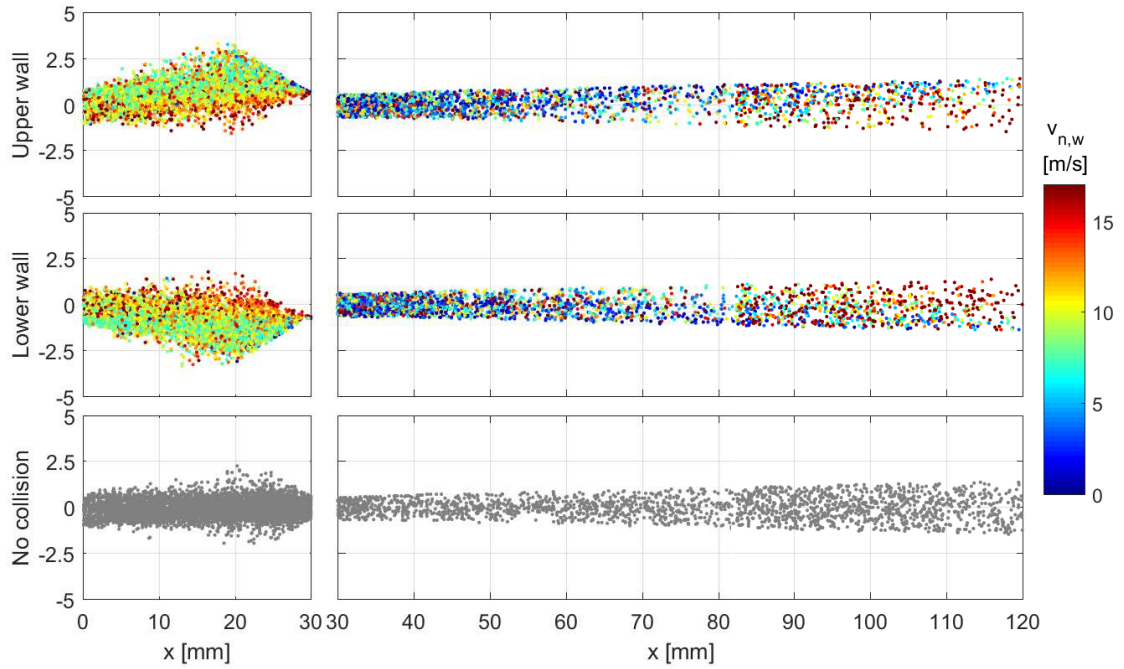


Figure 5.22: Scatter plot of particles contributing to nozzle clogging risk in converging (left) and diverging (right) section, separately shown are: particles on collision path with upper wall (top) with lower wall (centre) and with no collision in the section (bottom), coloured by wall-normal velocity.

Figure 5.22 shows displays those particles (injected with inj.1) that are on a collision path with the upper wall and lower wall in both nozzle sections separately, along with particles that do not collide in the respective section of the nozzle. The intensity of the colour corresponds to the wall-normal velocity component, and therefore indicates the strength of the particles contribution to clogging risk. A second factor that determines this risk is the amount of particles that impact onto the wall. The figure shows that the particles in the central region of the converging section impact onto the walls with highest speed, however, their number decreases towards the centre, as more particles escape the section without collision. Naturally, this is increasingly noticeable when approaching the throat. The outer regions of the injection plume exhibit more moderate speeds, but all particles in these regions collide eventually. The amount of particles that enter the diverging section on a collision path is very high, and both upper and lower wall are affected by particles across the channel. Here, impact velocities are still low, however, grow towards the nozzle exit, where wall collision becomes more sparse but stronger. Moreover, impacting particles tend to originate more from the wall vicinity, rather than any lateral position. At the same time, the particles that avoid wall interaction are distributed more evenly across the nozzle in the final section, while, just downstream of the throat, mostly particles on the centreline avoid wall contact.

This analysis shows that the number of particles that interact with the walls is surprisingly high for both sections. In the converging section, approximately 65% of particles collide with the wall. Because of the length of the diverging section, even higher values up to 81% can be inferred here, despite the widening walls. Note that this implies that almost all particles - up to 93% - are probably interacting with the wall at some stage during their flight.

	5bar	7bar	9bar
Conv. section	65.3%	62.6%	65.0%
Div. section	74.9%	77.0%	81.2%
Overall	91.3%	91.4%	93.4%

Table 5.1: Percentage of particles that is on a collision path with the nozzle wall in converging and diverging section with pressure

Table 5.1 shows the percentages for increasing pressure. The low speed collision probability does not noticeably depend on pressure level, while the likelihood of contact on the

high-speed side increases with higher pressure. This can be explained by the fact, that the injection plume is the main influence for wall contact in the converging section, while the increasingly unordered particle motion and the stronger acceleration define the wall collisions in the diverging section. Consequently, this means that the low-speed clogging risk can be predominantly influenced by the injector, while the high-speed clogging risk results from the process parameters that are required for a specific application and require more complex measures to control (*e.g.* specific nozzle designs).

	Inj.0	Inj.1	Inj.2	Inj.3
N_{coll}/N	75.1%	65.0%	57.5%	38.6%
avg. $\alpha_{w,i}$	10.8°	12.2°	12.8°	14.2°
avg. $v_{p,n}$ [m/s]	4.5	6.7	6.8	7.7
x^*-x_{inj} [mm]	-80	-30	-20	-10
$l_{influence}$ [mm]	-8.6	-13.2	-18.1	-57.3

Table 5.2: Particle-wall collision summary with changing injector position.

Searching for a beneficial configuration, it is useful to analyse the percentage of particles on a collision path for the different injectors. Table 5.2 consolidates these probabilities and other representative quantities. Two of those, the normal impact velocity and the impact angle, are discussed in detail in the sections above. It is evident, that the wall collision probability strongly reduces with decreasing distance between injector tip and nozzle throat, due to the smaller dispersion and the smaller possible contact region. At the same time, the impact angle and velocity increase, meaning that collisions get stronger although fewer. In terms of clogging risk, these trends are opposed: more impacting particles are similarly problematic as high velocities and angles. This indicates that there is an optimum distance for an injection point for clogging risk reduction.

A possible way to find this is to infer it from the properties of a particle that represents the “average” particle-wall contact. If this characteristic particle is released from a beneficially close zone, clogging risk is small, and if it is released from further away, the risk is high. Representing the average particle-wall collision, the particle evaluates the injection distance based on the respective average impact conditions of that configuration. It is the particle, which comes into wall contact just at the nozzle throat, having average impact angle and average wall-normal velocity, as illustrated geometrically in figure 5.23.

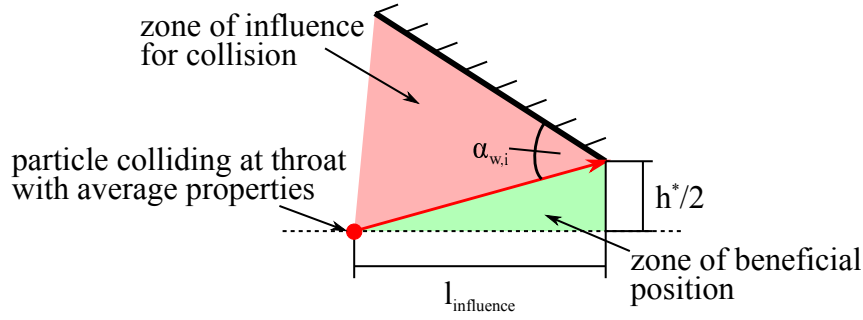


Figure 5.23: Definition of two zones: an average particle with positive wall normal angle defines length of influence $l_{influence}$ in the converging section, representing a beneficial distance of release

A length of influence $l_{influence}$ can be inferred; a distance, at which this average particle is released from the centreline in order to just reach the wall at the throat. It divides the volume in an upstream zone of wall collision influence, and a downstream zone that is beneficial.

Resulting from the average particle properties, the length of influence increases with reducing injection distance, which is also listed in above table 5.2. According to this trend, it is reasonable to consider an injection location as optimal, where the length of influence and the actual injection distance are approximately equal. In the present case, this would represent an injection just millimetres downstream of inj.2, approximately at 20mm ($10d_{inj}$ and $20h^*$) distance to the throat.

Despite being an idealised representation that does not account for the curvature of trajectories and may differ for other nozzle geometries, it is a useful guide to estimate the average wall collisions. In potential future clogging analysis, these findings could be tested experimentally and computationally, as nozzle clogging is an important issue in CS and future studies should attempt to assess this issue more frequently and more thoroughly. At this stage, it is useful to suggest a set of design aspects that ought to be considered for minimal clogging risk nozzles.

1. If particle heating is not crucial, injection into the diverging section is favourable, as it avoids particle impact onto the throat walls [151].
2. The wall temperature influence should be minimised, *e.g.* by cooling or high conductivity materials [106].
3. In order to reconcile the amount of impacting particles and the strength of their

impact, the injection distance from the restriction should be moderate. For heavy materials, 20 times the restriction size can be used as a first estimate.

4. Due to stronger spreading of the injection plume, the value should be reduced for light powder materials, *e.g.* for half the material density by 20%.
5. Low speed clogging results from a small number of fast particles from the centre and a large number of slower particles from the outer regions of the injection plume. The latter can be reduced by shielding the wall, *e.g.* by an angled aperture.
6. The angle of wall impact can be reduced by an elongated convergent section, which is favourable if not in conflict with space limitations. Alternatively, the wall angle can be steep prior to injection and shallow where particles can impact.
7. High speed clogging originates from particles of all lateral positions in the restriction and is difficult to influence. A minimum length nozzle contour with a linearly widening extension would quickly widen the channel, potentially reducing the number of impacting particles.

5.6 Consequences for properties at impact location

5.6.1 Particle velocity footprint

In addition to their influence on nozzle clogging, the statistical trends from section 5.4 influence the velocity distributions and particle dispersion in the supersonic jet. The resulting profiles at the locations, at which substrates are typically placed, are crucial for the achievable conditions upon impact. Typically, these locations would be at about 30-50mm stand-off distance from the nozzle exit. At this point, the dispersion defines the spray spot size, and the particle velocity and angle determine deposition efficiency. The final observations in this experimental chapter are hence from data of FoV4. The theoretical substrate location is assumed to be 40mm-50mm, which corresponds to 13-17 times the nozzle exit height h_e .

Figure 5.24 is a collection of cross-sectional velocity profiles of the particle velocities at this stand-off distance. Results at 5 and 9 bar are shown for all injectors and both

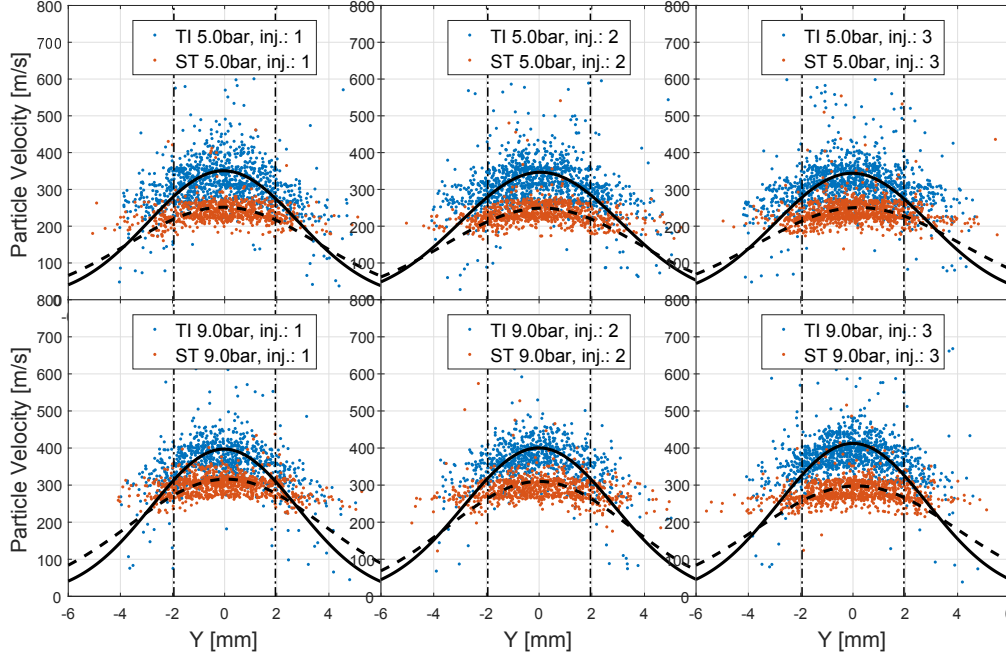


Figure 5.24: Cross-sectional particle velocity profiles at 40mm stand-off distance. Comparison of materials, injectors and pressure

materials. The solid line is a Gaussian fit for Titanium, the dashed line for Stellite particles respectively. Moreover, the nozzle exit width is shown to indicate the dimensions of particle jet widening. As illustrated in this way, the lighter material, besides reaching higher levels of velocity (ca. $1/3$ at 5bar, $1/4$ at 9bar), exhibit a stronger gradient at the jet edge and a wider dispersion. This is a confirmation of the results in chapter 4 with nozzle NZ1. The footprint is about $1.5h_e$ for Stellite and $2h_e$ for Titanium. A higher velocity gradient can be explained by the lower level of Stokes numbers, that cause the particles to decelerate more drastically when entering the shear layer of the gas phase. The stronger dispersion in fact confirms expectations from above statistical analysis. Similarly confirmative is the broader range of velocities of the lighter material, being about 100m/s for Stellite and more than 200m/s for Titanium. Consequently, the change in Sk causes most of the characteristic changes. Shifting the injector position downstream caused the statistical range of particles to reduce when entering the diverging section. Interestingly, this aspect is reflected for Titanium in the jet, as the dynamic range reduces accordingly, but with a small change. Titanium also experiences slightly higher velocities with the injection close to the throat, however with similarly small differences and mostly at higher

pressure. In case of Stellite, this trend is reversed, as the injector 3 delivers a slightly reduced velocity due to the delayed acceleration. This indicates that the volume-fraction related effects (*e.g.* collisional motion, turbulence) mostly change the particle dispersion, but leave particle velocity relatively untouched in this parameter range. Mass-loading related effects (*e.g.* gas momentum drop) are consequently mostly responsible for the latter. These observations are analogue for the nozzle exit velocities (appendix p.237).

Moreover, it can be read from figure 5.24 that the particles disperse stronger at lower pressures. Partially, this can be explained by the statistical analysis, in which pressure caused some straightening and equalisation of the normalised particle velocities within the nozzle. Stronger acceleration in the diverging section at higher pressures maintains this trend for the diverging section. Additionally, the gas phase experiences a series of stronger shock waves at 5bar than at 9bar. These shock-particle interactions may significantly contribute to increased dispersion in spite of the weaker shear layers due to lower core velocity. Measuring both gas and particle phase during shock-interaction is an unsolved challenge in CS and hence left for future work. Here, it should merely be noted that stronger shocks cause stronger dispersive forces and plausibly increase turbulent fluctuations. The detailed influence of these oscillations on the high Stokes number particles remains unknown, again, because gas phase fluctuations cannot be measured simultaneously with the particulate phase present.

5.6.2 Jet dispersion diagram

Since the measurements found that particle dispersion is one of the most affected aspects when changing Stokes number, feed rate as well as pressure, and even undergoes some small variations when the injector location is altered, a more thorough analysis appears justified. In this respect, dispersion should not only be characterised by the velocity footprint alone. It is useful to obtain an indicator of additional dispersion trends, such as rates and directions. Moreover, using the trajectory angle once more, the effect of particle feed rate on the dispersion can be shown in detail.

Therefore, a summary of the particle trajectory angles at the same stand-off distance, *i.e.* corresponding to the velocities in Figure 5.24, is shown in figure 5.25.

Here, the angles are scattered over the y-coordinate, which is normalised by the nozzle

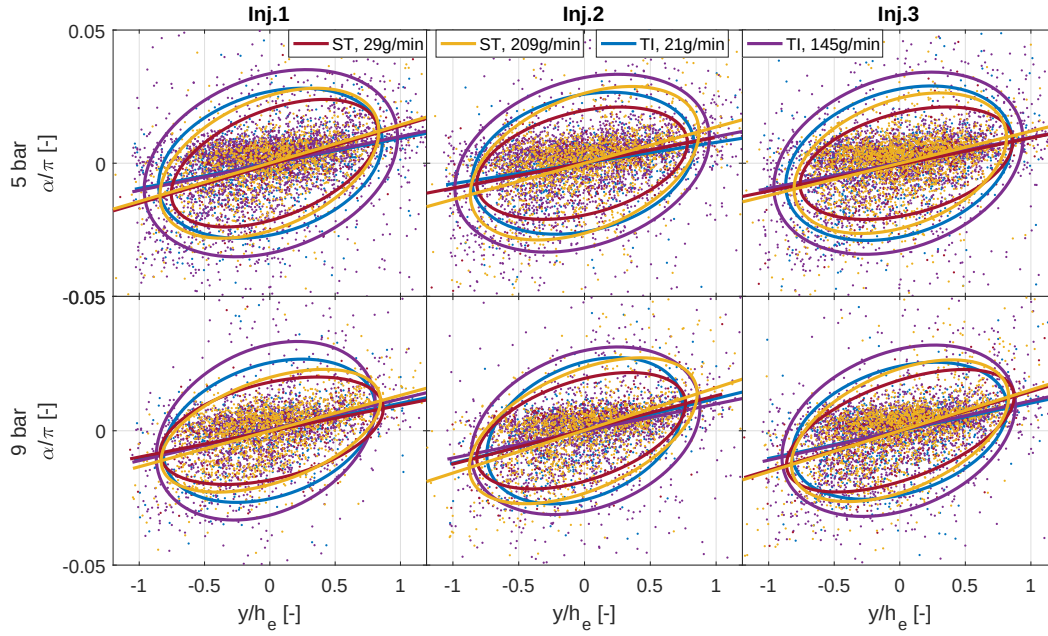


Figure 5.25: Jet dispersion diagram: Regression and 95% confidence ellipse of local trajectory angles with respect to position within a jet cross-section at 40mm stand-off distance. Comparison of materials, injectors, pressures, and feed rate

exit width. Injectors 1-3 are compared at 5 and 9 bar again. Moreover, the plots include high and low loading cases of Stellite and Titanium. Each dataset is supported by two fit plots. The first is a regression line, which indicates a linear function of position. It corresponds to the mean behaviour of particles that are further away from the centreline dispersing outwards at proportionally increasing angles. The second fit is an ellipse, which represents the 95% confidence interval of the scatter, based on the eigenvalues of its covariance matrix. It is a useful way to illustrate the importance of cross-motion. The length of the ellipse indicates the probability of particles to disperse away from the centreline, while the width of the ellipse represents the probability of particles to move back towards the centreline. The same angle, depending on position, is thus either dispersive or concentrative. Following interesting observations can be drawn from this figure in addition to the previous.

The dispersion of Titanium is affected more strongly by feed rate. In this respect, the characteristic change is a similar increase of both axes of the ellipse. This means that particles are enhanced to disperse outwards more, but likewise to fluctuate back inwards, corresponding to a strong mixing. Stellite, in contrast, reacts with a slight

increase of ellipse width and length, but mostly with an increase in the steepness of the ellipse and regression line. This means that under higher feed operation, the heavier material tends to disperse outwards more strongly, but almost maintains its strength of mixing. Due to the increase of particles that move randomly back towards the centreline in case of Titanium, the magnification of the dispersion angle by distance from the centre is effectively reduced. The heavier material does not exhibit this effect as much and therefore shows a less “dampened” dispersion, such that the particle position is increasingly driven outwards. Considering that Titanium has the higher dispersion rate, without this effect, the footprints were even wider. It should be noted that the deposition efficiency may be affected similarly, whether the particle moves outwards or inwards at the same angle.

In all cases, the dispersion is significantly less drastic at the design pressure of 9 bar, confirming previous observation of detrimental low-pressure operation. Moving the injector downstream (1 to 3) was previously shown to have relatively small effects on velocity. In case of low pressures, also the dispersion is only slightly affected by the injection location. At the nozzle design pressure, the dispersion is however statistically changed by the injector position: comparing the average width of all spray spots of injector 1 and 3 with injector 2, it can be seen that injector 1 has a spot by 5.9% wider than injector 2, and injector 3 by 6.4% accordingly. Although the changes are moderate, a mid-injection (2) shows the most favourable results. This injector balances positive (velocity-synchronising) and detrimental (dispersive) consequences of particle-particle and particle-wall interactions within the converging section. It also allows for an intermediate mixing of the process gas and injection plume and limits the acceleration lag. Moreover, the injector has a favourable length to limit the risk of to cause nozzle clogging. However, this dictates the particle residence time and therefore precludes efficient particle heating.

5.7 Summary

Understanding the complexity of the flow mechanisms in CS is important for the advancement of the technology. Isolated investigations that are difficult to integrate are insufficient and the nozzle-internal motion of a simultaneously present wide range of Stokes numbers is key to new insights into the particulate flow physics. As previous chapters have indicated

and started explaining, there is no conclusive understanding of the phase interaction mechanisms in CS, that go beyond the application of high-Mach number drag laws, in particular under increased loading. This chapter started filling this gap by virtue of experimental observation of the particle behaviour within a CS nozzle. Four injectors at different locations were studied with Stellite particles, for a higher range of Stokes numbers, and Titanium, for a lower range.

Injection far upstream with a homogeneous particle phase entering the nozzle, showed an increase in the mean particle velocity with loading in the low speed section. The particle velocity in the jet dropped, which is stronger for low pressures, while high pressures cause a more drastic increase within the nozzle. A more randomised motion due to particle-particle interactions was deemed possible explanation. In this effect, both volume fraction and mass loading play a role.

The injection velocity distribution and the particle acceleration through the nozzle were studied and average accelerations in the throat were found to be as high as $8.7 \cdot 10^5 \text{m/s}^2$. The dispersion of particles at the injection is approximately linear with distance, but cross-sectional velocity profiles do not develop due to the well-set strong co-flow from the main line. At the injection, the Titanium velocity increases with loading while the Stellite remains similar with a tendency to decrease.

Statistical analysis of the normal probability density function of velocity and trajectory angle found that the downstream shift of the injector location reduces the dynamic range of velocities. Increasing pressure aligns particle trajectories with the x-axis, relatively independent of y-position. Titanium particles tend to have a range of velocities that agrees little with the log-normal distribution of a drag-driven motion. Stellite tends to coincide better, which seems to make the lower Stokes number range the driving factor for deviation from this theoretical limit. Consequently, most likely due to collisions in the longitudinal directions, Titanium particles synchronise their normalised velocity more with increasing loading.

The results were used to gather new insights into the nozzle-internal phenomenon of nozzle clogging. Two types of clogging were distinguished: low velocity (throat-) and high velocity (exit-) clogging, differing in wall impingement angle and velocity level. It was suggested that low velocity clogging follows mechanisms similar to deposition on a substrate,

while high-velocity clogging involves the tangential component in a more fundamental way than previously thought, potentially by changing the particle deformation pattern. It was evaluated that injection at an intermediate distance from the throat can reconcile increasing wall collision strength with a reducing percentage of colliding particles. Chances of particles to collide at some stage during their flight with nozzle walls are above 90%.

Within the jet, the volume-fraction effects, shock-particle interactions and turbulence cause a stronger dispersion of Titanium as compared to Stellite. The dispersion tends to follow different mechanisms: Stellite particles mostly disperse outwards, increasingly with distance from the jet axis. Titanium particles have a more randomised motion, and consequently higher probability to move back towards the centreline despite strongly inclined trajectories.

Computational methods, as thoroughly reviewed in 2.4, were never assessed according to these viewpoints. The contrary is true: inter-particle collisions and the role of particle-wall collisions are usually fully ignored. Valid particle velocity and dispersion prediction is hence still a challenge. Even the mass loading of particles, although in few studies accounted for, is not thoroughly computed. Because of this status of validity, CFD is not used for advanced analysis such as for nozzle clogging. Therefore, the final discussion in chapter 6 of the work addresses which of the experimental findings can be represented by the often-used platform *Ansys FLUENT*.

Chapter 6

Computational results: Analysis of a new computational model

6.1 Introduction

Objective 4: Develop a holistic advanced computational model and analyse the present nozzle flow computationally. Based on the experimental findings, determine the limits of the model details and derive means for further model improvement.

This chapter transitions to the third phase of extending the knowledge about gas-particle flow in CS. After employing the established approach of jet measurements, and then extending it by novel measurements inside the nozzle, the potential of the new experimental approach should be demonstrated for a wider use. These experiments open the opportunity to analyse the gas-particle flow computationally in much more detail than previously, and the chance to validate numerical models in a new direct way and in more detail is particularly valuable. Such investigations identify the weaknesses of established models and show the necessity for advanced modelling details. In this, they shift the current state of the art to higher precision models, which are beneficial for future process optimisation, design and analysis work.

Transferring the experimentally observed trends to arbitrary opaque application nozzles is by nature difficult. This leads the argumentation back to the motivation of the study.

Because such experiments are so unique, they mainly serve the purpose of improving numerical techniques, which can then be applied more widely. Therefore, the experimental analysis presented in this work is used to assess often used commercial methods (*Ansys FLUENT*). This final chapter discusses capabilities and limits of such models with the goal of improving their applicability based on the nozzle-internal measurements. After introducing the CFD model, the chapter is thereby sectioned according to main challenges that arise about CS computations:

- Velocity level prediction
- Dispersion estimation
- Residence times (particle heating) and wall-collisions (nozzle clogging)

6.2 Holistic computational model development

In this section, the computational model is introduced. *Ansys FLUENT v16.0* is used as a solution tool, pre-processing is done in *Ansys Workbench* and post-processing is done in *Matlab* as well as *CFD-Post*. Since no solution code was developed in itself, the model description is condensed to the essentials.

6.2.1 Model equations

After having introduced the theoretical aspects and a wide range of model equations in chapter 2.4, a brief summary of the mathematical side is deemed sufficient at this stage. The particulate phase, not being spatially interconnected, and the gaseous, being continuous, are described by two different sets of equations. These conservation laws are called the Eulerian-Lagrangian framework. Table 6.1 summarises the equations described below.

Gas equations

Reynolds-averaged Navier-Stokes equations (equations tab.6.1 1-3) are solved in a steady state for the nitrogen gas phase. For the closure of the Reynolds-averaged equations, additional expressions for the Reynolds-stresses are required, and the eddy-viscosity approach

is chosen to model these stresses with a two-equation model, as the vast majority of researchers does (see section 2.4). The present simulations employ the realizable k - ε -model for the eddy viscosity, solving additional transport equations for the turbulent kinetic energy k and dissipation rate ε , given in equations tab.6.1 4-5. It is chosen due to its strength in capturing the dissipation rate in highly dissipative flows such as axisymmetric jets by imposing a realistic condition of non-negativity on the mean-square vorticity fluctuation. This also provides superior performance for flows involving boundary layers under strong adverse pressure gradients, relevant mostly in the nozzle exit and free jet region. Literature review shows that the only alternative for significantly better performance are Reynolds-Stress-Models, which account for the inhomogeneous character of turbulence in rapidly-strained nozzle and jet flows. These models are beyond the scope of the affordable

Gas phase		
1	Mass	$\nabla \cdot (\rho \vec{v}) = S_m$
2	Momentum	$\nabla \cdot (\rho \vec{v} \vec{v}) = -\nabla p + \nabla \cdot \mu [(\nabla \vec{v} + \nabla \vec{v}^T) - \frac{2}{3} \nabla \cdot \vec{v} I] + \rho \vec{g} + \vec{F}$
3	Energy	$\nabla \cdot (\vec{v}(\rho E + p)) = \nabla \cdot (k_{eff} \nabla T - \sum_j h_j \vec{J}_j + \bar{\tau}_{eff} \cdot \vec{v}) + S_h$
4	Turb. kin. energy	$\frac{\partial}{\partial x_j} (\rho k u_j) = \frac{\partial}{\partial x_j} \left[\left(\mu + \frac{\mu_t}{\sigma_k} \right) \frac{\partial k}{\partial x_j} \right] + G_k + G_b - \rho \varepsilon - Y_M + S_k$
5	Trub. diss. rate	$\frac{\partial}{\partial x_j} (\rho \varepsilon u_j) = \frac{\partial}{\partial x_j} \left[\left(\mu + \frac{\mu_t}{\sigma_\varepsilon} \right) \frac{\partial \varepsilon}{\partial x_j} \right] + \rho C_1 S \varepsilon - \rho C_2 \frac{\varepsilon^2}{k + \sqrt{\nu \varepsilon}} + C_{1\varepsilon} \frac{\varepsilon}{k} C_{3\varepsilon} G_b + S_\varepsilon$ $C_1 = \max \left[0.43, \frac{\eta}{\eta + 5} \right], \quad \eta = S \frac{k}{\varepsilon}, \quad S = \sqrt{2 S_{ij} S_{ij}}, \quad G_k, G_b, Y_M \text{ are constants}$
Particle phase		
6	Momentum	a) $\frac{d\vec{u}_p}{dt} = \frac{1}{\tau_r} (\vec{u}_c - \vec{u}_p) + \frac{\vec{g}(\rho_p - \rho_c)}{\rho_p} + \vec{F}$
		b) $\frac{1}{\tau_r} = \frac{18\mu}{\rho_p d_p^2} \cdot \frac{C_D Re_p}{24}$
		c) $C_D = \frac{24}{Re_p} (1 + 0.15 Re_p^{0.687}) + \frac{0.42}{1 + 4.25 \cdot 10^4 Re_p^{-1.16}}$
7	Energy	a) $m_p c_p \frac{dT_p}{dt} = h_q A_p (T_c - T_p)$
		b) $Nu = \frac{h_q d_p}{k_c} = 2.0 + 0.6 Re_p^{1/2} Pr^{1/3}, \quad Pr = \frac{c_p \mu}{k_c}$

Table 6.1: Overview of model equations with nomenclature according to chapter 2.4

effort for CS applications, in particular for three-dimensional geometries, and cause instabilities in combination with aggressive source terms resulting from the particulate phase in case of two-way coupling. This makes them uninteresting for wide usage and inapplicable to the present case. To account for the transition of importance of molecular to turbulent viscosity as wall distance increases, without an immensely refined mesh, non-equilibrium wall functions are employed. Distinctly from standard wall functions, they are capable to account for the effects of pressure gradients in complex flows, where the mean flow and turbulence are subject to strong pressure gradients and rapid changes [136]. Ideal gas law

is used to model the compressibility and Sutherland's law delivers molecular viscosity.

Particle equations

The particles are solved in a Lagrangian reference frame and connected through coupling laws to the gas phase, that is solved in the Eulerian frame. This way, the motion of a particle can be integrated simply along its trajectory using a force balance, as given by equation tab.6.1 6a-c). Herein, the drag coefficient C_D is calculated according to the law by Clift et al. [129], which is the only available model incorporating compressibility effects. As discussed in section 2.4.2, this model makes use of an empirical correction for particle Mach numbers exceeding 0.4 and can hence be applied to such compressible regimes. Nevertheless, the variation of Mach number is not explicitly accounted for, and gas density variations are only included through the in the Reynolds number. It is therefore applicable to moderate relative Mach numbers, limited by maximum relative Reynolds numbers of 300,000, but may have limited precision in the rate of change in lack of direct representation of the local Mach number. Particles do not have physical volume and are represented as point masses, which imposes a restriction to relatively low volume fractions ($<12\%$) on the discrete phase, a condition which is fulfilled. The question of the requirement and options to incorporate volume-fraction related effects is part of following discussions. Additional fixed body forces are Saffman lift to account for transverse velocity gradients, virtual mass due to massive acceleration, and a longitudinal pressure gradient correction. Local gas velocity fluctuations may impact the particle motion. Turbulent dispersion is one of the aspects discussed below, which, when used, is modelled through a stochastic tracking discrete random walk (DRW) model. An isotropic fluctuating velocity component u' is superposed to the mean gas velocity, based on the turbulence kinetic energy from the turbulence model: $u'=\zeta\sqrt{u'^2}=\zeta\sqrt{2k/3}$, where ζ causes a random normal distribution that is updated after the characteristic life-time of a turbulent eddy.

When collisions of particles are enabled, the stochastic algorithm of O'Rourke [191] is used, for which the velocity outcome $v'_{p,1}$ of a collision for a particle parcel of mass m_1 with a second one of mass m_2 , and the respective probability of the collision $P(n)$ are given in equations 6.1-eq:orourke3. The outcome is calculated from a momentum conservation, which is altered by random number b to account for assumed dissipative losses. Further,

$\bar{n}$ is the average number of collisions of a parcel, with $r_{1,2}$ as particle radii, $v_{p,rel}$ as relative velocity, n_2 as number of particles per parcel, and V as the volume of the grid cell. Each time step calculates the mean number of collisions for all parcels $\bar{n}$ and generates the actual number based on a random number generation within the probability distribution $P(n)$, before updating the velocities according to the outcome.

$$v'_{p,1} = \frac{m_1 v_{p,1} + m_2 v_{p,2}}{m_1 + m_2} + \frac{m_2 (v_{p,1} - v_{p,2})}{m_1 + m_2} \left(\frac{b}{r_1 + r_2} \right) \quad (6.1)$$

$$P(n) = e^{-\bar{n}} \frac{\bar{n}^n}{n!} \quad (6.2)$$

$$\bar{n} = \frac{n_2 \pi (r_1 + r_2)^2 v_{p,rel} \Delta t}{V}, \quad (6.3)$$

Based on small Biot numbers, the temperature difference across a particle is neglected due to negligible internal resistance to heat transfer. The uniform temperature is obtained by equation tab.6.1 7a-b), in which the temperature difference between the local gas phase and the particle phase drives the transfer, neglecting radiation. The heat transfer coefficient h_q is calculated according to the Nusselt number relation of Ranz-Marshall as given.

6.2.2 Geometry and computational grid

Domain

The geometry of the CFD model is composed of three main sections: fluid domain of the nozzle head, the nozzle itself, as well as an expansion chamber. Heat transfer through the nozzle and head walls is not considered, so that the solid parts are not modelled. This can be justified by the short operational times (~ 1 min) and the low temperature gradients due to absence of gas heating. Therefore, the domain represents the fluid domain only, which is presented in figure 6.1.

The main sections and boundaries are identified in this figure, in order to clarify the geometry. As the designed equipment and hence also the fluid domain are inherently three-dimensional, a full 3D model is constructed. The head section is geometrically more complex than nozzle and chamber, with the main-line inlet offset, the conversion from circular to rectangular shape, and the presence of the injector needle. The nozzle section

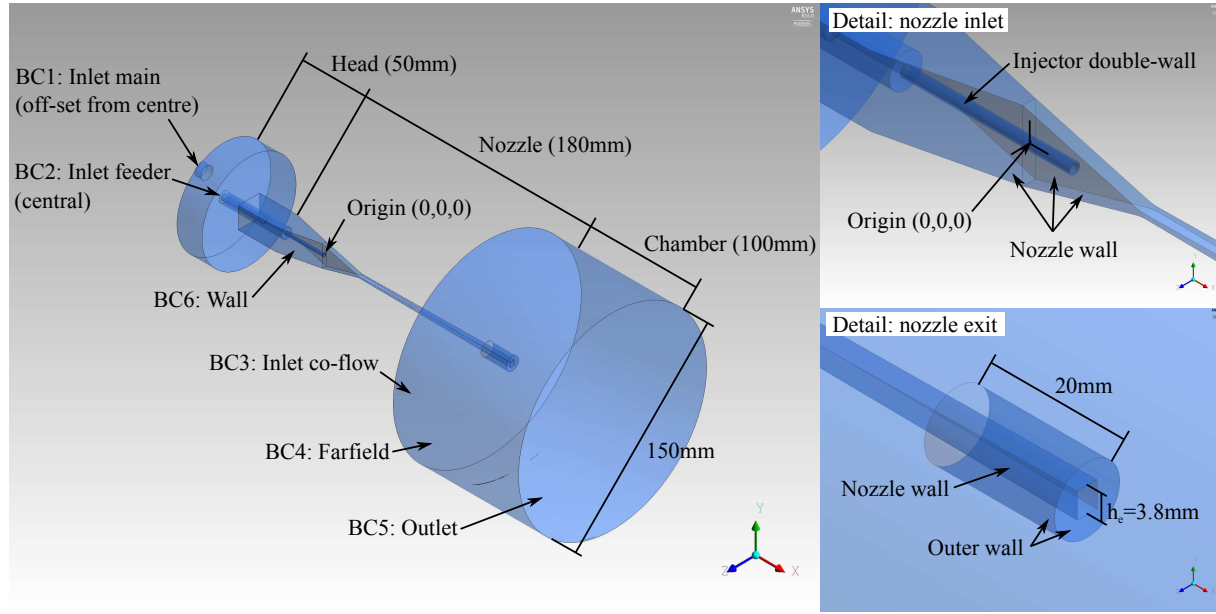


Figure 6.1: Computational domain geometry with nozzle head section, nozzle section, and chamber section, identifying boundary conditions (BC)

details correspond to the experimental design. In agreement with the convention in the experimental analysis, the origin lies at the point that is referred to as nozzle-inlet, meaning the inlet of the measurable part of the nozzle (section with constant width). Just upstream of this, the channel is narrowed from the square cross-section to the nozzle inlet shape. In order to avoid influence of the boundaries on the jet flow, the ambient boundaries in the chamber (BC3-5) are placed with sufficient distance from physically important parts. In this respect, the far-field is placed at a distance of $\sim 20h_e$ away in each direction, the outlet at $\sim 26h_e$ downstream, and the co-flow inlet at $\sim 5h_e$ respectively. An increase of these values (25,30, and $7h_e$) did not influence test-case results of jet velocity distribution.

Mesh

The mesh, shown in figures 6.2a-6.2e below, is constructed using tetrahedron-dominated unstructured cells in the geometrically complex part, namely the head and injector region. In order to optimise the mesh for high velocity nozzle- and jet-flows, structured hexahedron elements are employed for the diverging section and the chamber. Transition between mesh types is set upstream of the nozzle throat, in order to avoid the risk of influences on regions of strong acceleration, but cell sizes are matched upon transition. Each injector needle is meshed according to their individual geometry and the injection location can be seen in

figures 6.2b-6.2d. Elements are coarsened in the nozzle head to reduce the number of cells and refined within the injector and particularly at the tip of the injection needle as well as towards the nozzle throat. Inflation layers in the unstructured mesh are used for wall refinement within the injector needle and the converging part of the nozzle. In case of the structured mesh, wall refinement and jet shear layer resolution, shown in figure 6.2e, are obtained using node spacing functions. The dimensionless wall adjacent cell size is kept within the range of $y^+ = 20-300$, such that wall-function invoked errors in shear stress and wall temperature are avoided, yet always resolving the layers (analysis in appendix p.238). The resulting mesh is summarised in table 6.2. The meshes provide sufficiently mesh-

Mesh	Geometry	Elements	Nodes
1	Inj.0	2,247,269	1,877,797
2	Inj.1	2,449,522	1,869,768
3	Inj.2	2,817,646	1,947,238
4	Inj.3	2,368,263	1,861,962

Table 6.2: Computational mesh details, comparison of the four domains with different injector needles.

independent results for the gas phase at 9bar pressure: up-scaling the mesh of injector 1 to about 2,708,000 elements by refining these critical zones further leads to a change of representative variables (e.g. integral gas exit velocity) below 1% and does not change location of flow patterns. The resolution of shock waves is acceptable; pressure gradients are used on a single test-case to adaptively refine the mesh, which leads to minute changes in the shock thickness, but increases the mesh size significantly. Therefore, the original structured mesh resolution is accepted for present computations.

Boundary conditions

The boundaries as introduced above are treated mathematically in the following way, which is summarised in table 6.3. In terms of the gas phase, boundaries are combinations of Neumann- (gradient-specification) and Dirichlet-type (value-specification) boundaries. All inlets are set as pressure inlet conditions that hence specify the pressure value along with the temperature. Consequently, the velocity is set invariant over the boundary. Similarly, the far-field and the outlet specify the pressure, but extrapolate both velocity

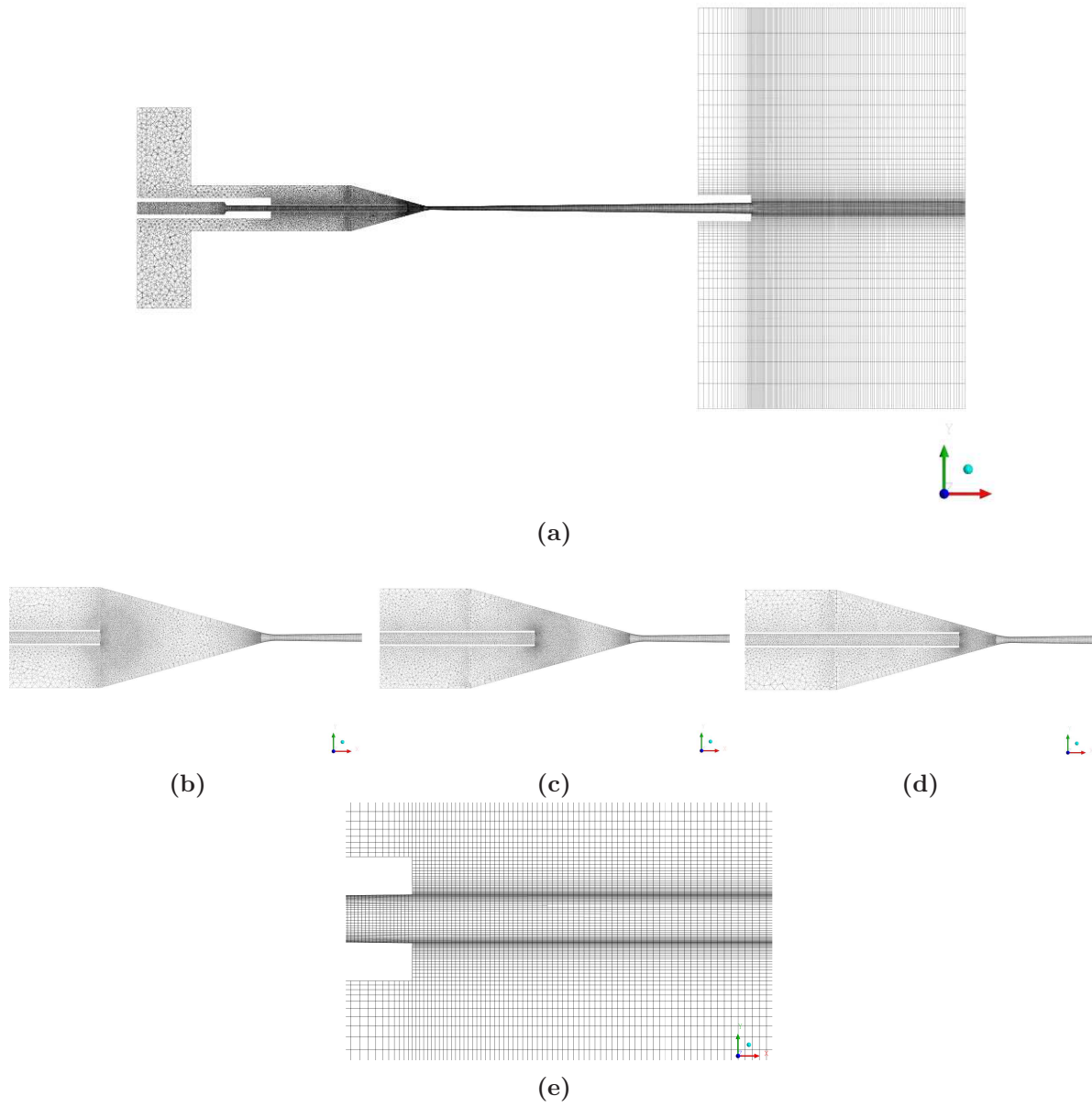


Figure 6.2: Sections of the computational mesh: full domain (a); head and injector resolved by tetrahedrons (b) inj.1 (c) inj.2 and (d) inj.3; nozzle and chamber resolved by hexahedrons (e)

and temperature form the domain. The outlet boundary has the special property to be flexible in Mach number, as it switches to a Neumann boundary for the pressure, if the local velocity is supersonic on the boundary (real characteristics lead to no possible fixed value on the boundary). The wall boundary condition sets the pressure gradient to zero and applies a no-slip condition. It also defines the gradient of temperature to be zero due to the aforementioned assumption of adiabatic walls.

	Name	p_0	$\vec{v}$	T_0	DPM
BC 1	Inlet main	specified	$\frac{\partial \vec{v}}{\partial n} = 0$	specified	reflect
BC 2	Inlet feeder	specified	$\frac{\partial \vec{v}}{\partial n} = 0$	specified	injection
BC 3	Inlet co-flow	specified	$\frac{\partial \vec{v}}{\partial n} = 0$	specified	escape
BC 4	Far-field	specified	$\frac{\partial \vec{v}}{\partial n} = 0$	$\frac{\partial T}{\partial n} = 0$	escape
BC 5	Outlet	specified	$\frac{\partial \vec{v}}{\partial n} = 0$	$\frac{\partial T}{\partial n} = 0$	escape
BC 6	Wall	$\frac{\partial p}{\partial n} = 0$	$\vec{v} = 0$	$\frac{\partial T}{\partial n} = 0$	reflect

Table 6.3: Boundary condition (BC) definition according to fig.6.1

Boundary conditions for the particle phase are defined separately. Particles are injected by the DPM model into the needle to establish realistic internal flow. Particles are forced to reflect off the nozzle walls (and the inlet of the main line, although particles do not reach this boundary). The remaining boundaries in the chamber are all open surfaces for particles and allow them to escape. Set-values for these boundaries depend on the operation point and are summarised in the section to follow.

6.2.3 Case settings and solution strategy

Boundary values

The temperature was set to room temperature in all cases. Ambient pressure, applicable to BC3-5, is set to atmospheric pressure. The pressure values on boundaries BC1 and BC2 vary with operation point. From the experimental values, two representative settings are chosen for each injector, as shown in table 6.4. The parameters under each pressure setting are the particle feed rates for Stellite (St) and Titanium (Ti) from experiments, which are applied to the particle phase inlet boundary. The zero feed-rate cases imply that the respective powder was calculated without two-way coupling. Likewise, the non-zero

values represent coupled simulations with the respective particle mass inflow.

BC3-5:									
Inj.0-3 $T_0=293\text{K}$, $p_0=1.013\text{bar}$									
BC1&2:									
Inj.0-3 $T_0=293\text{K}$, $\dot{m}_p[\frac{g}{min}]=\dots$, for $p_0=\dots$									
		3bar		8bar		5bar		9bar	
		St	Ti	St	Ti	St	Ti	St	Ti
Inj.0		0	-	0	-	Inj.1	0	0	0
		34	-	40	-		38	13	51
			-	180	-		130	47	206
		5bar		9bar		5bar		9bar	
		St	Ti	St	Ti	Inj.3	St	Ti	St
Inj.2		0	0	0	0		0	0	0
		118	115	115	82		141	145	152

Table 6.4: Cases of various boundary parameter settings for computational set-up taken from experimental data

The injection boundary of the particle phase is a crucial boundary for the particle movement within the nozzle and therefore assessed in the evaluations of this result chapter. During set-up, additional input values are required on the Lagrangian boundary, since it is mathematically an initial condition. The release is of surface type, so particles are released from all cells on the respective boundary. By defining the material and a discrete particle size distribution based on the known powder properties, the individual particle properties are set. Using a mono-disperse particle phase with the average particle size is inappropriate for modelling any velocity distribution and dispersive motion, hence not considered in this work. A Rosin-Rammler logarithmic approximation of the known size distribution is set with 10 intermediate values. In addition, the particle temperature (also room temperature) and the injection velocity need to be given. Here, the approximate velocity level of the measured injection is used. A second approach in this work is a user-defined function, programmed to replace the initial velocity setting of the injection. More details of the approach are explained in section 6.5.

Solver settings

The phase coupling is fixed in one direction: drag and heat-exchange laws define the momentum and temperature change of the particle passing through a cell. In case of a coupled simulation, while the particle equations are solved, the gas phase is altered by a negative source term added to the RANS equations (excl. mass conservation), which balances out the transfer onto the particle. This process consequently involves an iterative solution procedure. This iteration is done 3 times per continuous phase iteration step, which again is then solved 3 times. Such a setting is found as reasonable compromise between solution stability and speed (e.g. a setting of 1-1 is the most stable and higher values speed up the particle transport). Another aspect of phase coupling solution settings is the turbulence modulation. Respective source terms are similarly hooked to the equations of the turbulence model, such that the particle movement changes the transport and production of k and ε . In case of included collision modelling, the stochastic collision algorithm is employed.

Because it is particularly suited for highly compressible flows, a steady-state density-based solver with double precision is used to solve all gas equations in a coupled system. The discretisation is implicit in formulation with a ROE-FDS flux splitting scheme. It makes use of a re-combination of the discrete flux elements according to the speeds of the transmission characteristics through a given cell. In this respect, a localised Riemann problem calculates the flux at a given face of the cell, based on the property that changes in a flow can travel only through entropy waves (representing the eigenvalues of the non-linear equation system). A least squares cell method is used to calculate gradients across cells and a second order upwind approximation is chosen for the spatial discretisation of the flow and turbulent quantities. Gas phase convergence is a necessary requirement for subsequent particle phase coupling. The Lagrangian equations of the particle phase is discretised with an implicit Euler scheme and solved by a pseudo-transient method. As the physical problem is steady-state, an artificial time is used to calculate the individual particles. Once the particle phase iterations lead to stable residuals, the system reaches the coupled steady state with each particle following the physics of its pseudo-time dependent trajectory. The pseudo-time step is selected small enough to inject new parcels homogeneously during iteration, and 500,000 steps with a step length factor of 5 are included to avoid aborted

particle trajectories. Parcels summarise particles into clusters as the individual calculation of all particles is computationally impossible. The parcel size is varied depending on the feed rate and material, such that each coupled case leads to approximately 70,000 additional equations.

6.3 Prediction of particle velocity

The introduced model naturally delivers information about a multitude of flow quantities at any location in the 3D field for the gas, and for every particle parcel in the domain. This big advantage over experimental methods is used in all CFD applications, but carries the risk of misinterpretation and invalidity. This section is about evaluating the quantities that are directly measurable, but also to connect them to the properties that remain hidden in the experiments. In that way, the gas phase is discussed in connection to answering the first question that arises in CFD computations in CS: can the particle velocity be predicted accurately? Respective emphasis in the section is the capability of the “full” model to represent an appropriate drag connection and to account for the measured trends with increasing loading. By full model, the introduced domain, mesh and boundary conditions are meant, including:

- Two-way phase coupling
- Turbulence coupling
- Collision modelling
- Injection boundary adaptation by user-defined function

Inclusion of all these aspects results in the most accurate model version, which is capable to deliver some distinguishing results. Following section 6.4 addresses influences and resulting advantages of these model properties in detail by separately excluding them.

6.3.1 Effect of coupling between phases

By reporting some features of the CFD results, the gas phase properties are introduced and its modulation due to the presence of the discrete phase is explained. This is used as a starting point for the particle phase and the validity of the reaction to particle loading.

Gas phase

Figure 6.3 shows the gas velocity profile along the nozzle and jet centreline. It presents a comparison of the uncoupled and coupled (high feed rate) solution of both materials using injector 1 on the left side. In addition, the right shows a comparison of Stellite using all

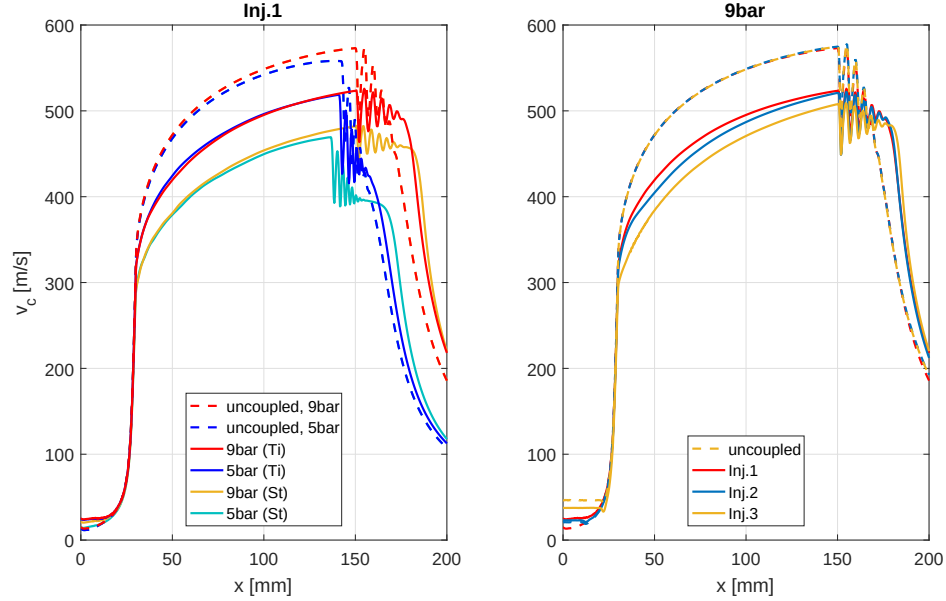


Figure 6.3: Comparison of gas velocity profiles along the nozzle and jet

injectors at 9bar, again compared to the uncoupled solution.

The main points to note are the over-expansion state of the gas, as indicated by the shock-expansion pattern at the nozzle exit, and the phase coupling. The gas Mach number evolution (app. p.239) shows very similar trends, but being widely invariant of the nozzle operation point, it does not substantiate the pressure influence like the velocity. At 5 bar pressure, the shocks begin to move into the nozzle diverging section and lie a small fraction of the diverging section internally. The pressure evolution (app. p.238) explains this adaptation mechanism by the below-ambient pressure level at the exit. Due to stronger shocks at 5bar, the shock angles are steeper, dissipation stronger, such that the compressive oscillations in the supersonic core of the jet are shorter for this pressure. Velocities consequently decline faster, but do not significantly differ within the nozzle (a reason why CS nozzles can in fact be operated below their design points in some cases). In terms of the full coupled model, velocities for both pressures drop significantly within the nozzle. The decrease correlates to the particle feed rate: Stellite, being fed at 2.5 the

maximum rate of Titanium, experiences a drop about 2 times as strong. Note, that for the lower pressure, this results in a stronger upstream shift of the shock position. It can also be seen, that injector 1 has the most undisturbed gas velocity evolution and injector 2 a slightly flattened profile, which is aggravated for injector 3. Therefore, the downstream shift of the injection location causes a stronger velocity loss along the centreline due to particle acceleration, while it has no effect on the uncoupled gas phase.

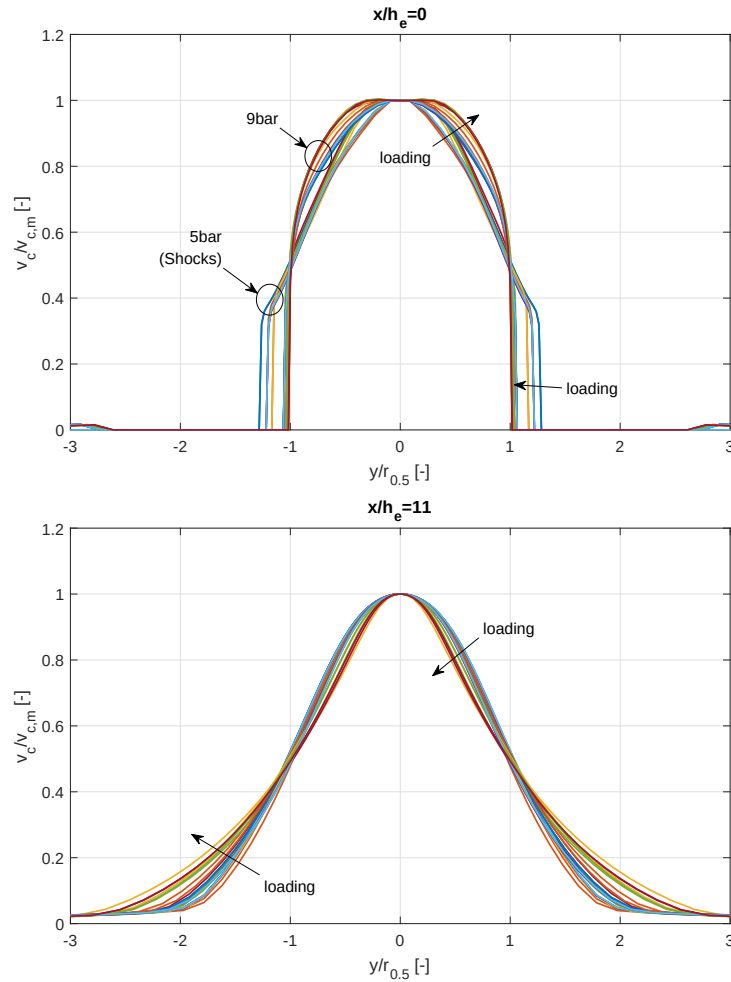


Figure 6.4: Comparison of gas velocity profiles in cross section at the nozzle exit and in the jet

Boundary layers and hence velocity profiles grow three-dimensionally along this described longitudinal evolution. At the nozzle exit, the velocity profiles are just about to reach a fully developed state. This can be seen in figure 6.4 by the small flat section in the centre. This figure shows the velocity profiles, normalised by the above described local centreline velocity, over the y -coordinate, normalised by the velocity half width, at the nozzle exit and at the end of the supersonic core. At the exit, the difference in shock

position can be seen from the oblique profiles on the outer parts for the low pressure. Increasing the feed rate causes the profiles to flatten in the centre and hence result in stronger gradients at the walls. The shock structures are weakened simultaneously. In agreement with literature, the high- Sk particle phase forces the velocity gradients towards the wall. This flattening effect is reversed downstream of the supersonic core, where the jet is no longer interrupted by shocks, but, under high feed rate, is not yet self-similar either. Here, the heavy particle phase begins to drag along the mean flow and reverse the signs of the the phase coupling terms. The higher the feed rate, the stronger the profiles deviate from a Gaussian curve towards a concentration in the centre. This trend regulates itself and leaves a fully self-similar gas phase for all loadings and pressures at $15\text{-}16h_e$ (appendix p.239).

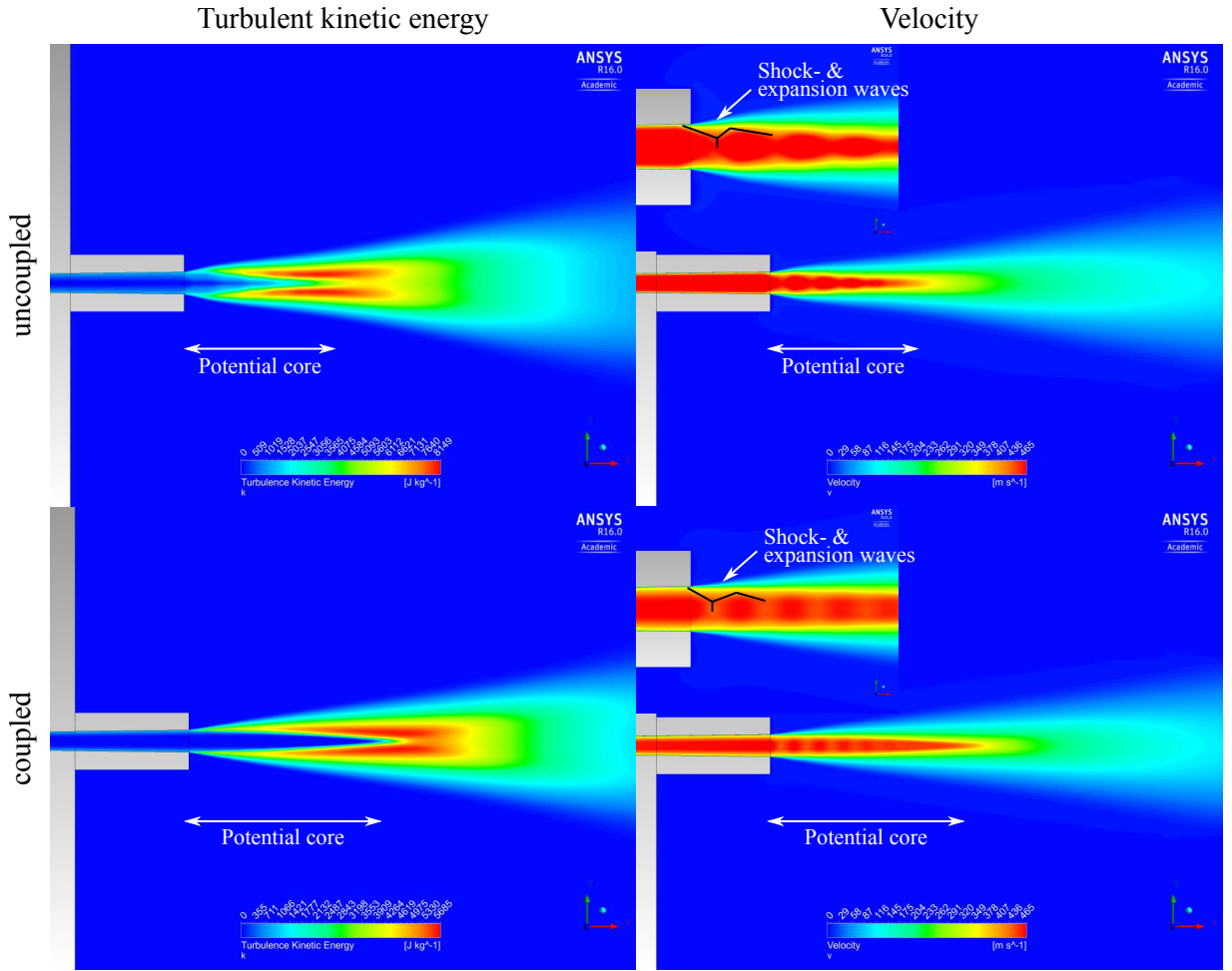


Figure 6.5: Contours of velocity magnitude and turbulent kinetic energy in the jet at 9bar pressure, comparing uncoupled and coupled gas phase under strong Stellite feed (200g/min)

The disintegration of the shock structures and the absolute distribution of the velocity can be seen well in a contour plot, as in figure 6.5. Here, contours of the velocity and turbulent kinetic energy in the jet at 9bar pressure are shown. The figure compares the uncoupled gas phase to the coupled solution under strong Stellite feed. As expected, the shock structures in the uncoupled case show a clearer pattern and compression waves stretch out further than in case of the phase coupling. The latter exhibits blurred discontinuities as the particles penetrate the shocks, carrying momentum and entropy with them. A result of this process is the prolonged potential core of the jet, which in turn correlates to stronger outward spreading as a consequence of the reduction of the velocity gradient in the shear layer. This can also be seen from the turbulent kinetic energy and the production of such in the shear layer (a 3D visualisation appended p.242).

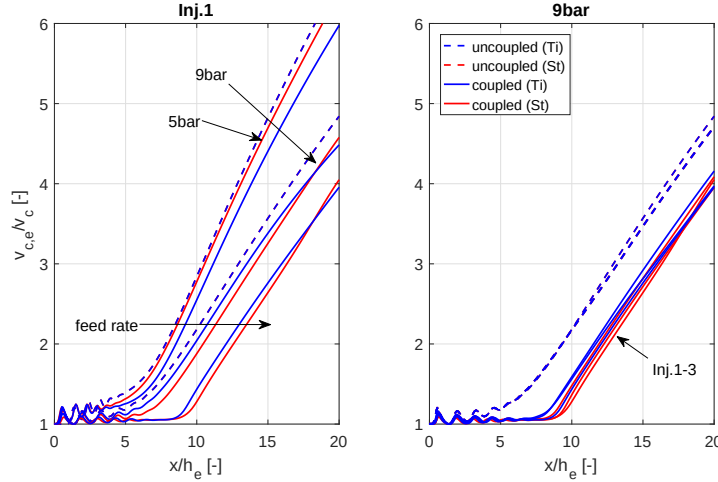


Figure 6.6: Inverse centreline velocity evolution in the jet

These mechanisms cause the jet to reach its continuous disintegration further downstream. In order to substantiate that increasing loading shifts jet self-similarity, the inverse of the centreline velocity can be compared, here normalised by the nozzle exit velocity. Figure 6.6 shows a comparison of the curves associated with injector 1, and those at 9bar for all injectors. Because a jet approaches a state of disintegration inversely proportional to position, this curve should approach a linear function. It can be seen, that both materials modulate the gas phase very similarly in this respect. Increasing feed rate drastically shifts the linear function downstream but maintains the gradient. However, the transition from a nearly constant velocity (core) to the linear regime is more abrupt. This is connected to the different character of disintegration in the first part, where the potential core

is prolonged but the shear layers widen into the surrounding fluid. This spreading state enhances entrainment of outer fluid, so that once the core starts losing momentum, the jet is comparatively far spread and the transition therefore shorter. The injector location does not have any particular influence on this jet property, in spite of the slightly differing nozzle-internal velocity developments.

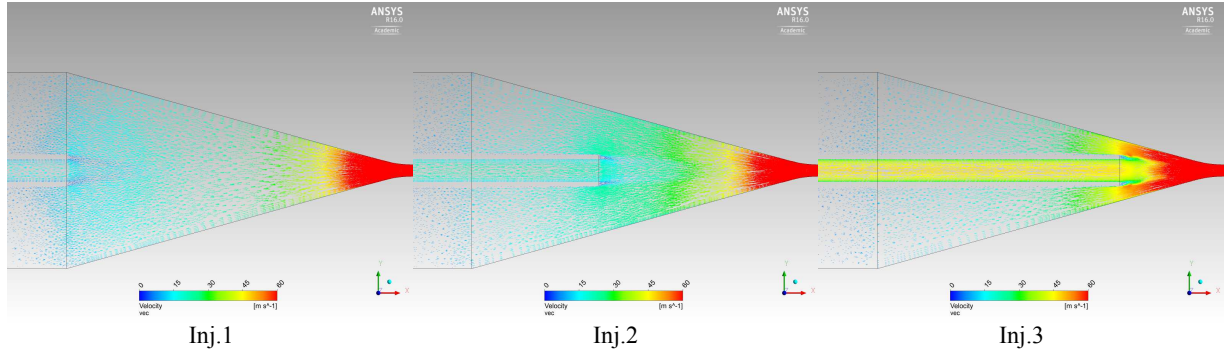


Figure 6.7: Gas velocity vectors at injection, displaying the range from 0 to 60m/s

The question arises how the gas flow alters around and through the injector tip with its location, which is shown in figure 6.7. When entering into the throat, the gas has a rather uniform profile that only slightly varies across the section with a very thin boundary layer (ca. $60\mu m$). This uniform shape is explained by the short distances of merging the main and feeder line. Injector 1 is far upstream in this concern, so that the flow is uniform quickly after. The second position shows a slightly accelerated main flow around the tip, as the process gas experiences a stronger confinement in the gap between needle and wall. Nevertheless, the subsequent acceleration of fluid homogenises this small difference. In case of injector 3, the local pressure at the tip has sufficiently dropped due to gas acceleration to cause stronger suction in the injector needle, which consequently has a higher velocity level throughout. Difference between process and carrier gas is still significant at the tip, which however equalises within millimetres due to the strong decline in pressure.

This inward movement of the main gas is manifested in the velocity profile at the throat, which is very flat and only exhibits small peaks close to the wall (4-5% above centreline velocity), transitioning into the boundary layer. The different injector tips do not cause noticeable differences in this respect. By increasing the particle feed rate, the character of this profile does not change and the velocity level across the entire section decreases, without an enhancement of such peaks. Therefore, the modulation of the gas

phase seems to take place uniformly over the cross-section (app. p.241).

Knowing these aspects of the gas phase, the influences in both directions can be analysed.

Particle phase

Where the gas phase loses momentum through the acceleration of the particle phase, the coupling is strong. In these locations, the gas phase exhibits a negative momentum exchange. If particles drag along the mean gas flow, the source of momentum is consequently positive. Figure 6.8 shows such a source term for the gas phase momentum equations in

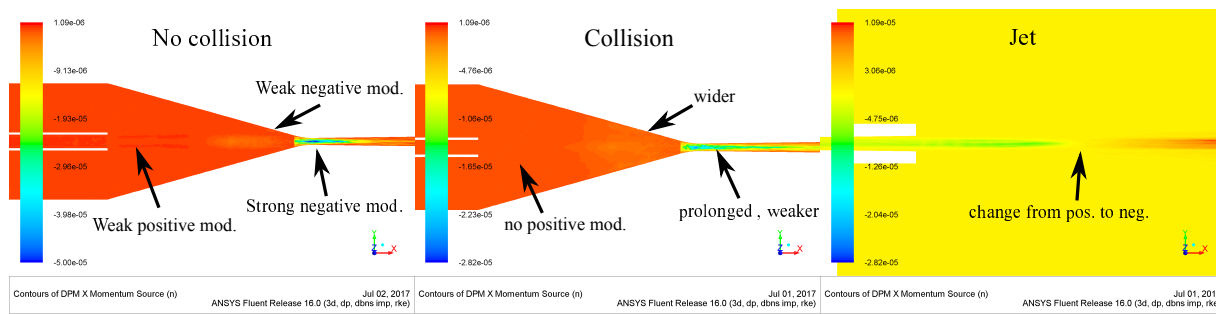


Figure 6.8: Contours of the source term of the discrete phase in the gas phase momentum equations indicating acceleration and deceleration of fluid by particles

the injection region and the jet. The converging section is characterised by a weak negative source, which strongly increases towards the throat, just downstream of which the strongest exchange due to massive particle lag is observable. Interestingly, the momentum source changes from negative to positive in some regions. One is the jet, where the gas velocity drops below the particle speed, such that momentum transfer is reversed. A second, less prominent example is the injector plume, where the gas velocity is particularly slow and particles have very straight paths. This is only observable in reduced models, hence not reproducible with the full model, where mixing is strong enough. In this case, which includes also collisions, the modulation is always negative, spread out through wider regions of the converging section and is significantly prolonged in the throat, while having lower maxima. This is connected to a different concentration through dispersion, such that the aspect of collisions also has a direct influence on the gas phase and in turn an indirect impact on the particle acceleration. Considering that the drag model is a quadratic function of the velocity lag between particle and gas, this illustrates why the investigation

of feed rate effects is conducted with the best possible model, i.e. including collisions.

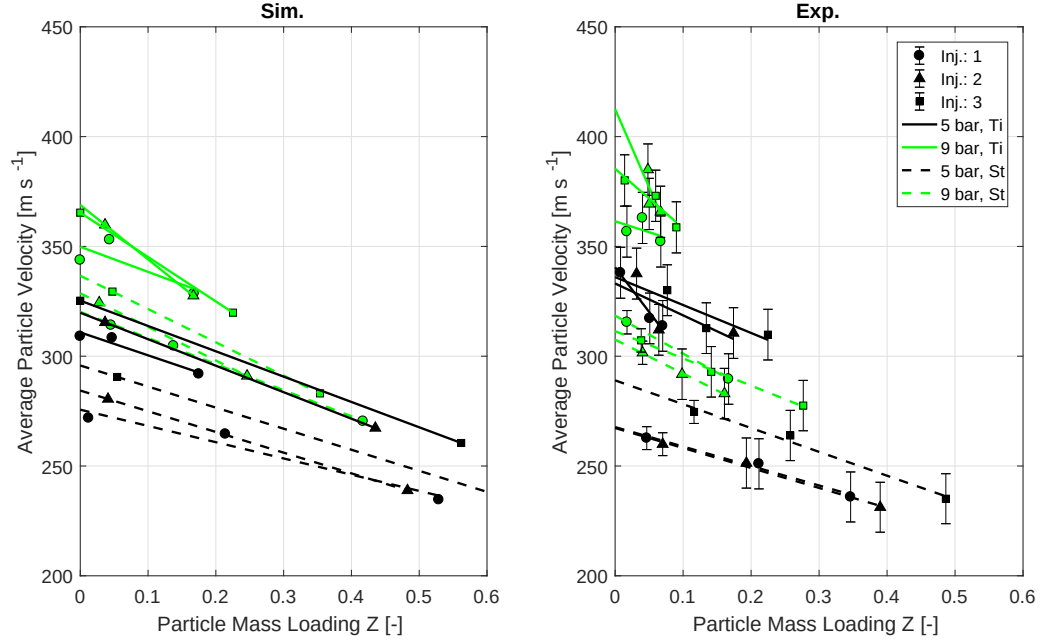


Figure 6.9: Validation of average particle velocity level in the nozzle exit region with increasing mass loading

Figure 6.9 presents a comparison of the computed velocity results of the full model and the experiments discussed in chapter 5. The average velocity at the nozzle exit is shown over increasing loading. Although the absolute gas flow rate differs for the numerical results, such that the data points shift on the x-axis, It can be seen, however, that this contributes to an acceptable validity for the particle velocity levels. At low feed rates, Titanium is under-predicted in velocity by about 5-10% for both pressures, which is out of bounds of the measurement uncertainty. Stellite is well-predicted with an over-estimation of about 3-4%, which is just on the edge of the uncertainty span, therefore a valid result. The small values of Titanium are connected to a flattening of the axial velocity profile in the jet, that is analysed in more detail in sections to follow. In the simulations, the gradients of the linear functions are slightly underestimated, which means that the measured susceptibility to loading is higher than the predicted. This results in slightly over-estimated velocities at higher loadings, but this trend is within the uncertainty bounds. The model is capable of predicting the overall velocity level, including the decreasing effect with particle loading.

From above analysis, it is not possible to state, if the model also captures the evolution

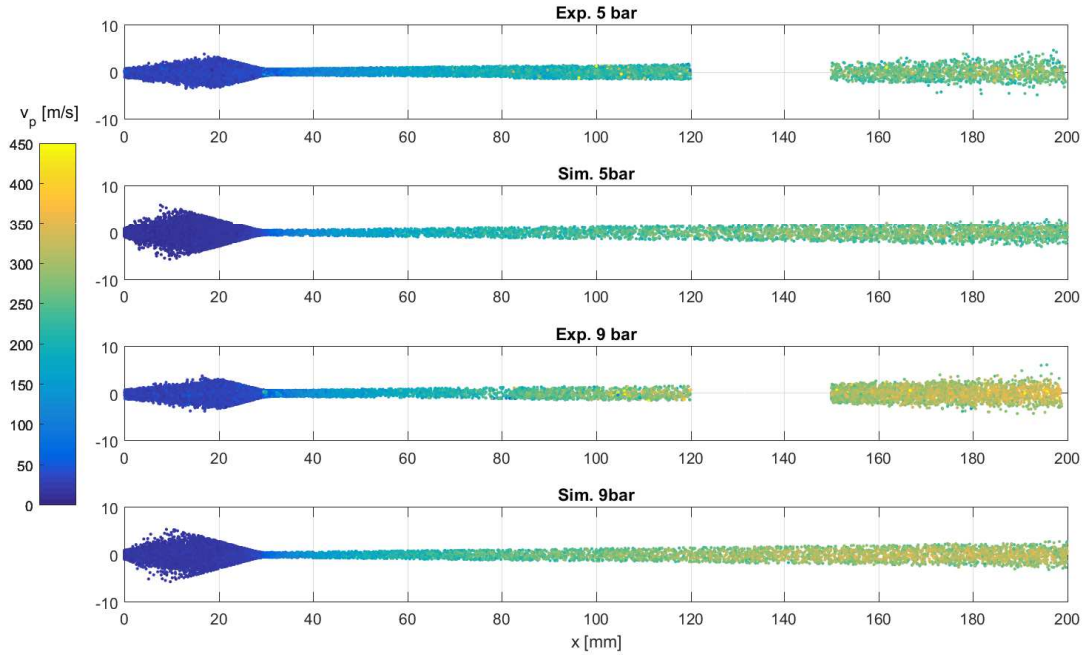


Figure 6.10: Scatter of particle positions and velocities for comparison of nozzle-internal distribution in experiment and simulation

of particle velocities. A model with an invalid acceleration model could “by chance” deliver proper results for specific nozzle-geometries. Therefore, the particle velocity distribution within the nozzle is taken into account, as shown in figure 6.10. It shows the a comparison of nozzle-internal particle velocities in experiment and simulation, at 5 and 9bar. This illustration shows generally good agreement of the evolution within the nozzle. Most obvious difference is the dispersion character both within the nozzle and the jet. The first is apparently over-estimated by this full model, while at the same time, the latter is too low and spreading not adequately calculated. Dispersion is therefore a topic to analyse in more detail and appears as the weakness of the model.

In terms of the acceleration, figure 6.10 does not provide much detailed understanding of the simulation quality yet. In order to do so, the data is evaluated along the x-direction. It is important that derived information is directly comparable to measurements, if analysed statistically as explained in figure 6.11. The local average and statistical properties of particle data must be considered, since individual diameters are indistinguishable in measurements. This means, although the acceleration of particles can be associated with their size in simulations, this cannot be directly verified experimentally. The figure shows

the example of different particle diameters reacting to the gas solution. These trajectories are three-dimensional and influenced by wall and particle collisions, turbulence and shocks in addition to their particle size - resulting in a probabilistic character. Consequently, only the statistical evaluation fully represents the behaviour and is comparable to measurements.

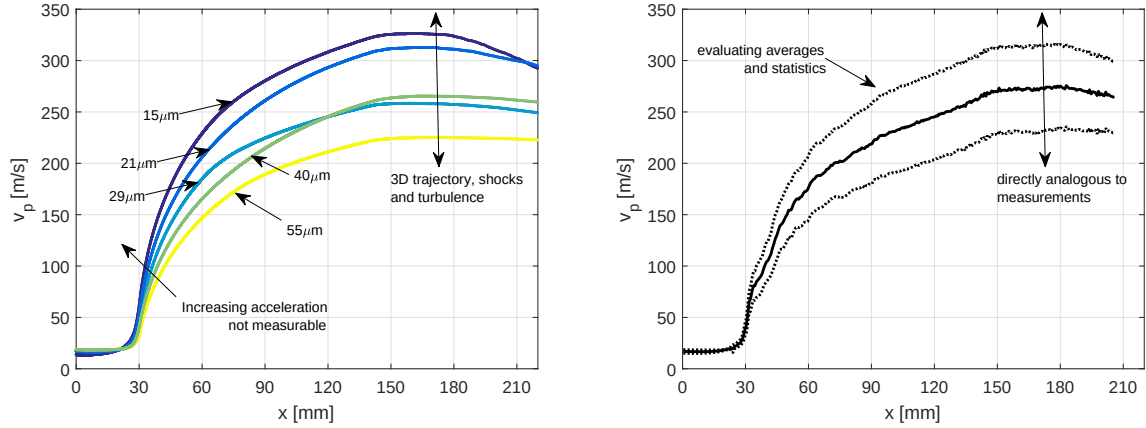


Figure 6.11: Concept of particle phase evaluation: average and statistical particle data is analysed, since individual diameters are indistinguishable in measurements

6.3.2 Adequacy of drag on particles

For the assessment of the drag force, and therefore the adequacy of the drag coefficient, the particle acceleration along the longitudinal direction is key. As explained above, the analysis focuses on two points, the local average and the local statistics. These are displayed in figure 6.12, where the solid lines are the averages and the markers display the standard deviation of particles. The dotted lines are the mean results of the uncoupled simulation.

The simulated velocity curves follow the experimental ones quite closely for both pressure levels. The most profound difference is the dynamic range of velocities, which is underestimated by the full model simulation throughout the field. This difference is smaller for 9bar than for 5bar, mostly because the collision model captures this range better. Moreover, it is smallest in the jet, which is connected to the absence of a noticeable reduction in velocity variance (due to shocks) at the nozzle exit in the model results. The velocity range at the injection is captured relatively well, when using this full model, in-

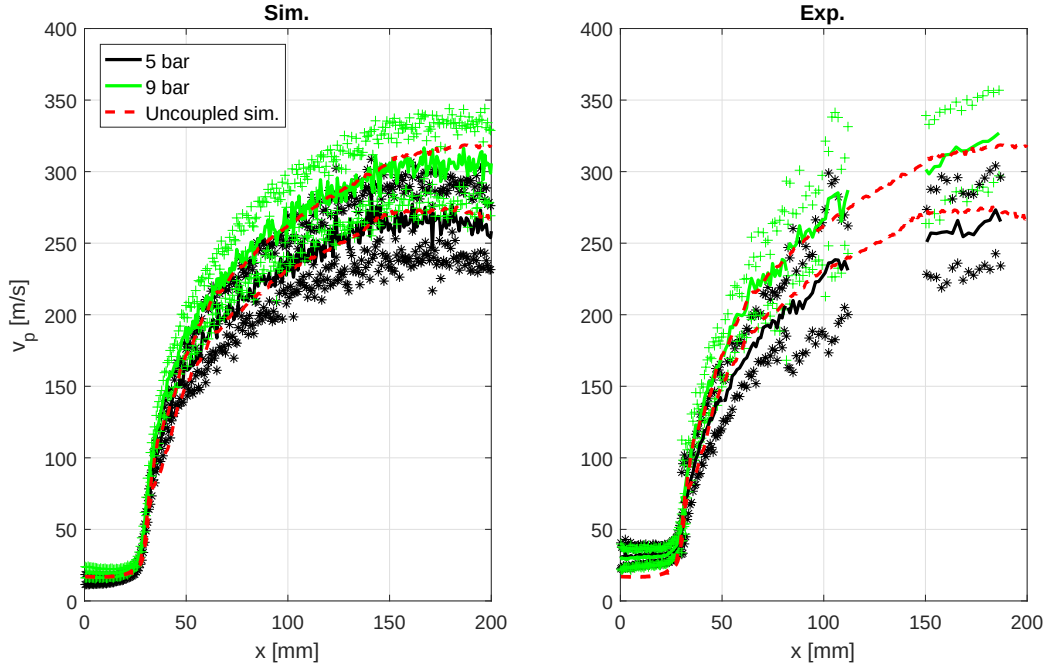


Figure 6.12: Particle velocity evolution along the nozzle and jet, comparison of simulation and experiments

cluding the adapted DPM boundary condition. An uncoupled model would underestimate the velocity level and not predict a meaningful range of velocities, which substantiates that the range is mostly influenced by the sum of coupling effects (appendix p.246).

While the average velocity in figure 6.12 is also slightly under-predicted in the injection region, the simulations tend towards higher exit velocity levels (in particular at 5bar), such that the peak acceleration seems to be too strong in the simulation. Nonetheless, the comparison of the uncoupled and fully coupled solution shows that this discrepancy is small, if the velocity drop due to particle loading is taken into account. Other marginal differences can be noticed; the flatter profile of the experimental values in the diverging section between $x=50-150\text{mm}$, and the deceleration in the jet that is only present in simulations but not observable in reality. In fact, the measurements indicate ongoing acceleration downstream of the exit while the simulations level off quickly. This flattening effect is a result of a too aggressive drag force in the throat. It is stronger for Titanium, such that its average velocity in the jet is miscalculated with previously reported differences. This indicates that the drag law is slightly too responsive at high relative velocities and too forgiving at low relative velocities, which may also contribute to the difference in dynamic range.

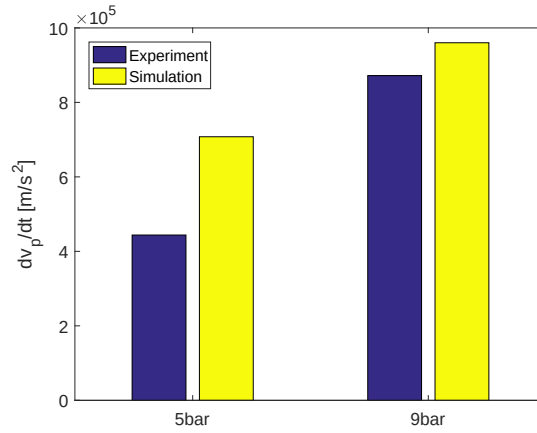


Figure 6.13: Validation of peak acceleration as a representative of the drag law adequacy

It is therefore interesting to derive the peak acceleration values of the average trajectory and check for their validity, to be seen in figure 6.13. Reflecting above analysis in hard numbers, the maximal drag error could be quantified to be as high as 60% at 5bar and only 10% at 9bar. Because the exit velocity nonetheless matches relatively well, the overall acceleration has rather low sensitivity to these differences. The overly strong acceleration in the throat is partly balanced out by the resulting low velocity lag towards the end of the nozzle. In particular at the design point, the solution is accurate. In summary, the drag law of Clift et al. is certainly valid for this application and nozzle geometry, but could be adapted for more geometry-independent reliability and for capturing the dynamic range. The latter depends primarily on the collision model and turbulence, which in turn not only influences the velocity drop analysed above but also determines the dispersion. This influence is hence subject to the following sections.

6.4 Prediction of particle dispersion

At the beginning of section 6.3, the statement is that in order to obtain the most accurate results, the full model is required. In the corresponding paragraphs, the velocity level is found valid, but dispersion appears problematic. Being mostly influenced by the individual advanced sub-models with respect to momentum-, energy- and turbulence-coupling as well as collision modelling, the second question that is to be answered at this point is: can the particle dispersion be predicted accurately?

6.4.1 Influences of advanced sub-models

Turbulence coupling

As could be seen in section 6.3, the turbulence level is low within the nozzle including the diverging section, with the exception of a thin increase in turbulent quantities in the boundary layer towards the wall. Low velocity in the converging part and short length scales contribute to this fact. The region where turbulence is the most influential is the jet (as can be seen in a comparison of k in throat and jet in appendix p.243). The two-way coupling of turbulence therefore causes particle and gas modulation predominantly in those high-turbulence regions, meaning that particles disperse due to turbulence mostly only in the jet. Implicitly, however, a two-way coupling is important, as the turbulent properties vary the development of the flow field (e.g. growth of boundary layers). Such a coupling involves the DRW model, which manipulates particle tracks based on k , and the gas phase source terms, which reflect the presence of the particles in the turbulence model. This source is shown for the nozzle throat and diverging section in figure 6.14.

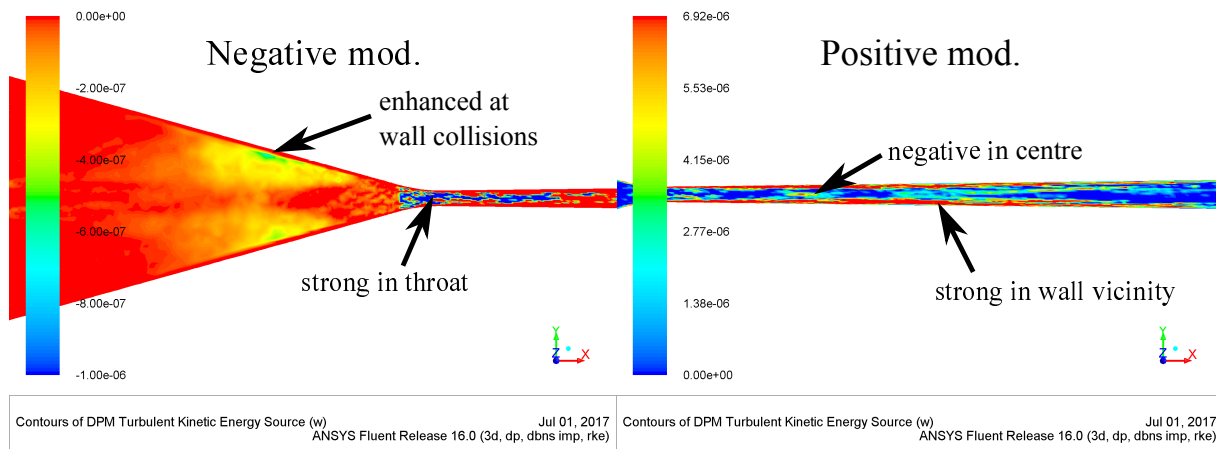


Figure 6.14: Negative and positive turbulence kinetic energy source term for gas phase showing modulation by particles in throat

Because this is the area of low turbulence level, the gas modulation is relatively weak. In order to see the character of it, the negative and positive values are shown separately. Positive modulation occurs only in a narrow band in wall vicinity of the diverging section, where acceleration and pressure gradients are strong. Negative modulation occurs in the remaining regions, in particular where particles concentrate and experience strong acceleration. Surprisingly, this means that the particle phase widely reduces the turbulence

level, hence dampens it. An expected increase in turbulence level is therefore no longer plausible. Explanations can be provided by the two main mechanisms of turbulence modulation, depending on length scales. Particles that are relatively large with respect to the energetic turbulent eddies can add turbulent energy to the gas phase due to wake formation in their relative flow. In contrast, particles that are small in this regard tend to break up eddies of larger sizes which may therefore decay more quickly into the viscous dissipation range of the turbulent spectrum. The approximate transition between the two is set to 10% of the estimated integral length scale [136, 189]. A rough estimate of the integral length scale can be derived from the domain dimensions $l_{turb} = 0.07d_{inj} \approx 140\mu m$, which is 7% of the injector diameter. The particles of the lower end of the size range may hence provide such damping, while larger particles may do the opposite. It is likely, that this underestimates the integral scale slightly, such that a significant part of the size range contributes to damping. Because larger particles become more rare due to the logarithmic distribution, it is plausible that the influence of damping weighs more than the few wakes formed by large particles. Interestingly, this is also true for the jet, where turbulence is much stronger. Here, modulation is fully negative (app. p.242), because the most energetic eddies are of even larger size.

Collision model

From the experiments in chapter 5, collisions were derived as a main candidate for important drivers of nozzle-internal dispersion. The introduced dispersion model [191] calculates possible collision events at every time step in a stochastic approach, since the computational cost to calculate intersections of hundreds of thousands of particle paths does not allow for deterministic approaches. The assumption is that two parcels may collide with an empirical probability, only if they are located in the same continuous-phase cell. The discrete phase volume fraction is therefore the main quantity of influence for this model.

Figure 6.15 shows the injection region for the coupled but collision-free solution and such including the stochastic model at a very high loading (200g/min) of Stellite particles. The highest volume fraction is found in the injector needle and exceeds 1.5%. The value drops in the dispersing injection plume and then increases again to about 0.5% in the restriction. Neglecting particle collisions, the volume fraction remains high for a longer

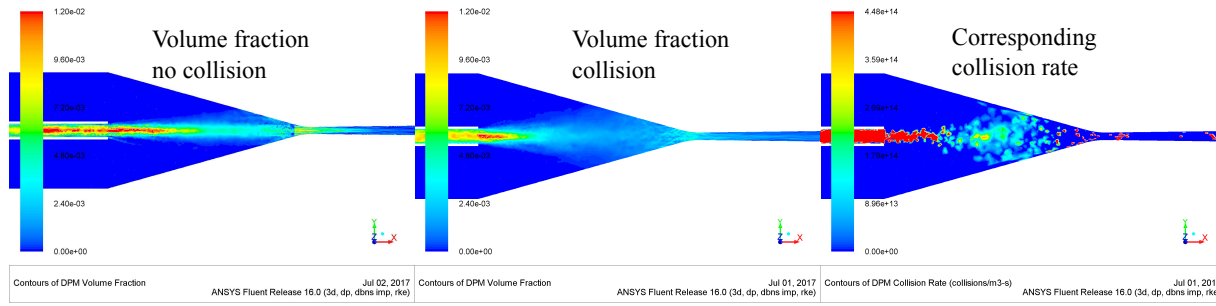


Figure 6.15: Volume fraction of particles at the injection without (left) and with collision (centre), and collision rate distribution (right)

stretch after injection and results in stronger accumulation at the centreline in the throat. These noticeable differences indicate the strong importance of the collision model. The rate of collisions calculated by the model can be additionally seen in the figure. Using the collision model hence produces strong particle-particle interactions in the direct injection region and a noticeable level throughout the converging section. Having declined in the throat, the collision rate is less important here, and negligible from this point downstream. Collision-depending properties of the jet are hence a result of such upstream interactions in the low-velocity part.

Injection Boundary Condition

As figure 6.15 indicates, the collision frequency is most drastic in within the needle. This raises attention to the injection boundary condition, as it determines the state of particles within the needle and, depending on the advanced models, their development down to the tip of the injector. Collisions may cause stronger mixing of the particles with different properties and hence provide a homogeneous phase. Nonetheless, the boundary condition determines which velocity magnitude and trajectory direction the particles have when entering the domain. There is a risk of bias of particle location, velocity and trajectory angle, due to the discrete release of particles over pseudo-time. As a result, the boundary condition can be adapted using a user-defined function (UDF), which makes use of data taken from the measurements. It is therefore a semi-empirical input function. The velocity angle and magnitude spans are known just at the nozzle tip, but not within the needle. The assumption is therefore that the values can be transferred upstream into the injector and that the z -component is analogue to the y -component. In this way, the UDF (code

in app. p.244) does the following:

- Load all particle parcels (all diameters) for a release step
- Define average particle velocity
- Define particle velocity ranges for x - y - and z -direction
- Generate random numbers within the defined ranges
- Superpose average and random fluctuation values
- Write fluctuation values to particle velocity components and release parcels

Resulting in a three-dimensional injection, this function can reduce the trajectory bias towards straight lines and of particle velocities to position. Note, that this function does not account for the pdf of the velocity range, hence does not realistically mimic the probability of each velocity. Such a boundary condition is possible but more challenging to implement and was considered beyond the scope of the analysis.

Comparison of particle movement

These aspects influence the particle movement in various ways. In this paragraph, the individual effects are shown by subsequently adding the model of the next complexity level. Figures 6.16-6.17 display results of measurements and simulations at the nozzle injector tip and in the region downstream of the throat respectively.

As figure 6.16 illustrates in the very lowest plot, the uncoupled model provides full trajectories, while the other models show instantaneous parcel positions, because there is no disturbance of the trajectory in the uncoupled case and it can be calculated instantaneously. It shows that the vast majority of particles follow very straight paths towards the throat as a concentrated bulk stream and only few trajectories diverge slightly outwards. These sparse trajectories result only from wall-collisions within the injector needle (app. p.246). Particles that are released close to the wall are pushed towards it due to Saffman lift and, once bounced off, keep colliding on opposite walls. Coupling the phases does not change much about this character. Although the gas phase is changed and a slightly increased velocity level can be observed, the dispersion of particles still depends only on

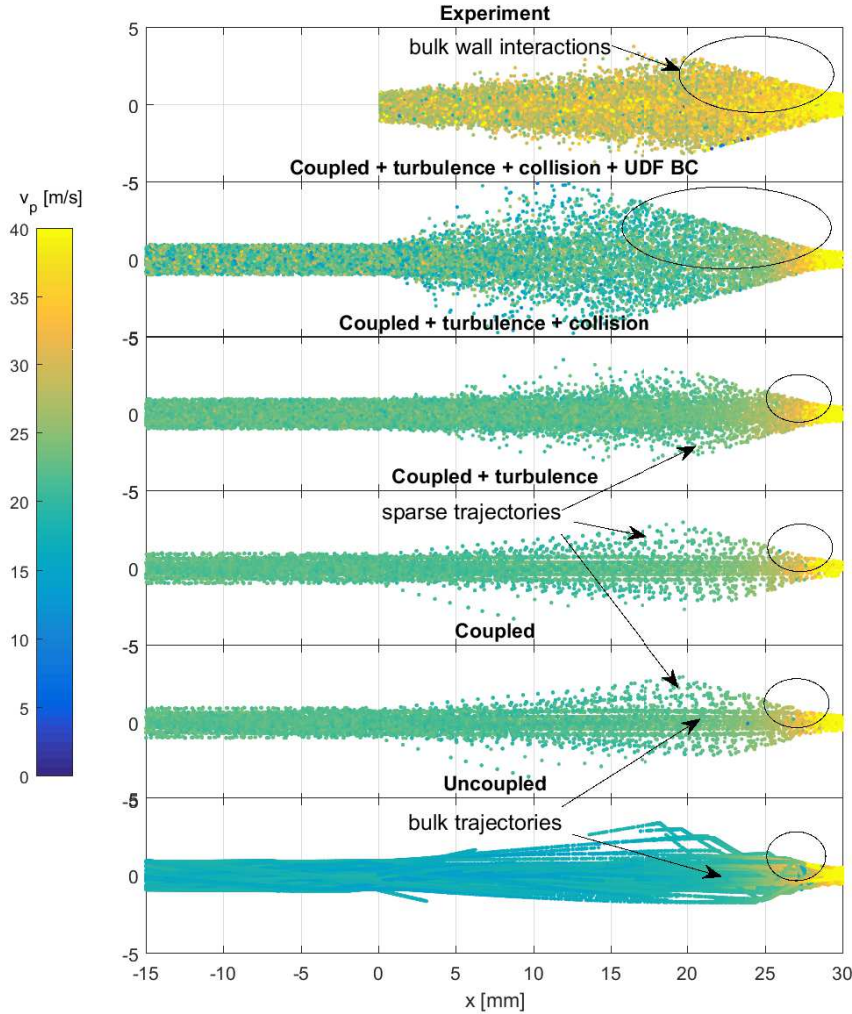


Figure 6.16: Stellite particle dispersion during injection comparing all sub-models

the aforementioned effect. Due to the low turbulence level, even a full turbulence coupling has minute effects on the dispersion. The scatter of the coupled model reveals the position bias, as particles that are released from the same cell with the same properties all follow very similar paths, which results in the unrealistically even spacing. This position bias can be resolved adding randomness to the particle paths using the collision model. As discussed above, this stochastically distributes particles according to the volume fraction, which leads to a much better break-up of parcel clouds after release into the domain. The discrete phase hence exits much more uniformly from the needle and disperses more realistically. This also slightly improves the region, in which particles collide with the wall at throat - range that is much smaller than in the experiments due to the under-predicted

dispersion. However, the collision model by itself does not sufficiently increase dispersion. Outer trajectories are still few and the majority of particles remain in the centre. When finally additionally employing the UDF, the dispersion is greatly enhanced. This comes from a fully homogeneous particulate phase throughout the injector needle that has stronger velocity components in the y - and z -direction. Therefore, needle-internal wall collisions contribute to the particle movement and, when reaching the tip, they are released at a statistically steeper angle. Moreover, the collision rate increases, because the relative particle velocity has a broader spectrum. Increased dispersive motion results in a strongly increased zone of particle-wall collisions in the throat region, now overestimating this aspect with respect to measurements. Nonetheless, the frequency of wall collisions and the width of the dispersion is best represented by this model. The over-estimation of the plume angle goes hand in hand with the smaller concentration close to the centreline, which is a consequence of the statistical distribution through the UDF. Its random number generator causes all velocities in the span to be produced equally often. This results in too frequent extrema and too few smaller deviations. Advancing the injection boundary condition methods is a good opportunity for future work to improve particle dispersion. At the same time, the collision rate in the stochastic model needs to be adjusted: the random number generation based on eq.6.3 should be flattened, such that fewer collisions occur in the dense, and more collisions occur in the dilute zone, while the velocity outcome of collisions should be maintained through factor b in eq.6.1.

Downstream of the throat, the findings discussed above develop some important features, as displayed in 6.17. The position bias can clearly be seen for the less complex models, even including the turbulence coupling. Non-physical concentrations both represented by the distinguishable trajectories and by a clustering of particles close to the centreline are the result. The uncoupled simulations shows a very regular wall-interaction pattern, which cause this central accumulation. Also the solution including the collision model does not fully resolve this issue, because of the character upstream of the throat as discussed above. This is an important point for the employment of the UDF and points out the sensitivity of the dispersion results to the injection boundary condition of the discrete phase. The full model therefore clearly matches the experimental observations most closely. Again, it is important to emphasise that the aspects are connected: invalid pre-

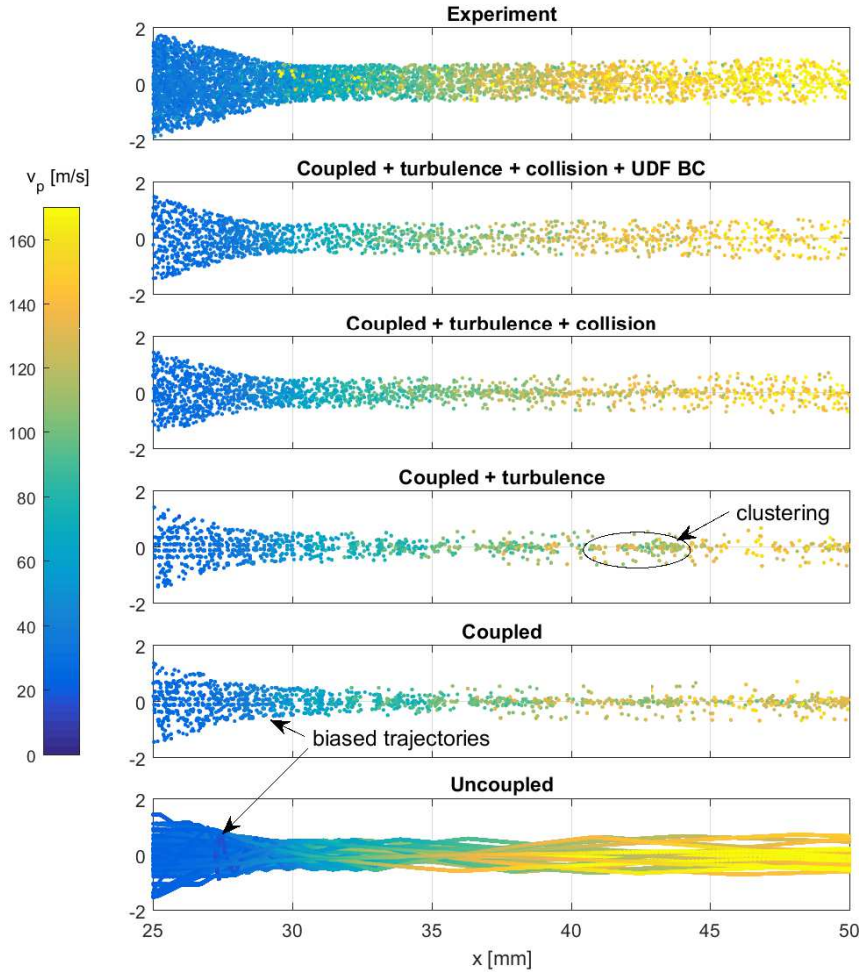


Figure 6.17: Stellite particle dispersion during evolution downstream of nozzle throat comparing all sub-models

diction of the injection dispersion leads to biased paths and nozzle-wall interactions, that, in turn, result in invalid clustering of particles. If such a concentration develops, it directly affects the dispersion in the jet. The accumulated particles follow paths that slowly tend away from the centreline, such that unrealistic concentrations of particles develop symmetrically both inside and outside the nozzle. When the injector location approaches the nozzle throat, the nozzle-internal dispersion prediction of the full model is amended because of the shorter spreading distance (app. p.248). On the other hand, the failure to capture valid trajectories by the simplified models aggravate accordingly. This direct relevance for the dispersion in the jet and the particle velocity footprint at some stand-off distance is analysed in the sections to follow, as it is of utmost interest for the application.

6.4.2 Injection statistics and jet dispersion

Normalised probabilities

Although the full model is the most capable to mimic the measurements, it still has the weakness of inadequate velocity probabilities. Since the materials behave differently in reality and exhibit probability distributions that differ from a log-normal profile, it is useful to assess the expected weakness by the same means. Figure 6.18 shows a comparison of normalised velocity pdf, as introduced in chapter 5.4. Here, the uncoupled and full model are contrasted, and the full model is validated against experiments at nozzle exit and in the throat.

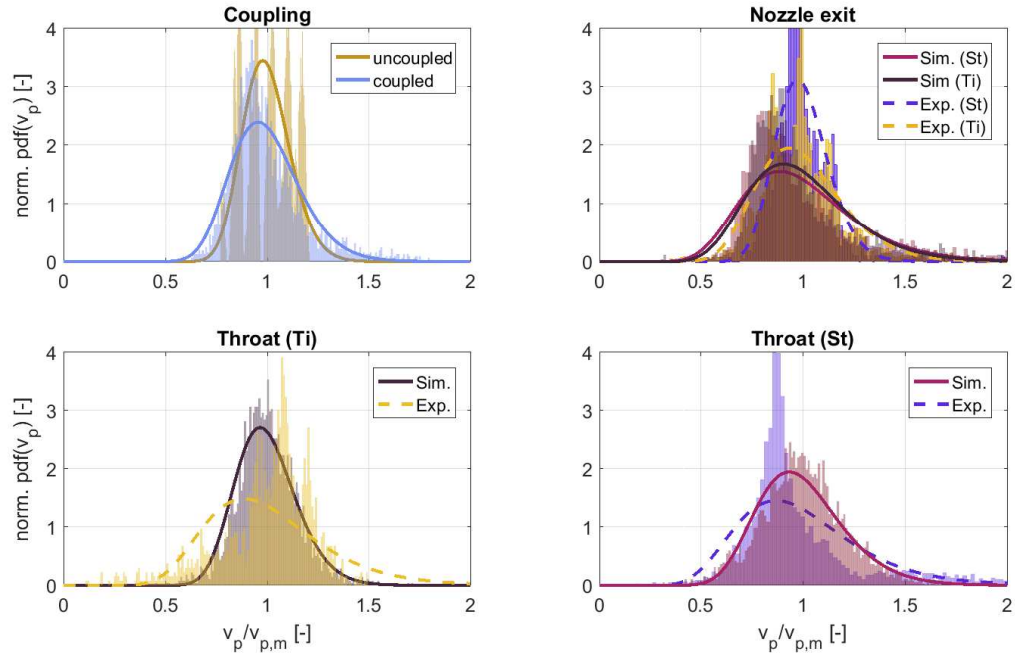


Figure 6.18: Normalised velocity probability density functions, comparison of uncoupled and full model, validation against experiments at exit and in throat

The uncoupled model is incapable of covering this statistical property of the particle phase. Its pdf shows distinct peaks for the different discrete particle sizes recognised in the throat, and their resolution is too low to meaningfully represent a probability density. The full model, in contrast, has a curve that approaches a log-normal profile, but concentrates higher velocities just below the mean, forming a light peak. This is a tendency that can also be seen in the experiments, however, the simulations show it in a relaxed form: when comparing the experimental and simulated results at the nozzle exit, both materials

resemble the log-normal profile too closely and exhibit a peak at too low velocities. In the throat, the simulation of Titanium shows an even bell curve, while the experiments shift to above-average velocity concentrations. In case of Stellite, the same character of simulation curve is contrasted by a downward shift of the peak in the experiment. Consequently, the model predicts too uniform probabilities around the average. This is in part connected to the boundary condition setting, which is too flat and hence equalises the likelihood of all velocities. In addition, it results from the imperfect collision model and the discrete size distribution. In principle, a model with a random size generation could provide a more adequate size distribution.

Velocity footprints

Above analysis indicates, that the full model is required in order to mimic the real dispersion. One point of main interest that this leads to is the particle velocity footprint in the jet at a specific stand-off distance. Figure 6.19 illustrates this by means of the cross-sectional particle velocity profiles of Stellite at a SoD of 40mm. The figure compares the full model as well as the uncoupled model to the experimental data for varying injectors and pressures.

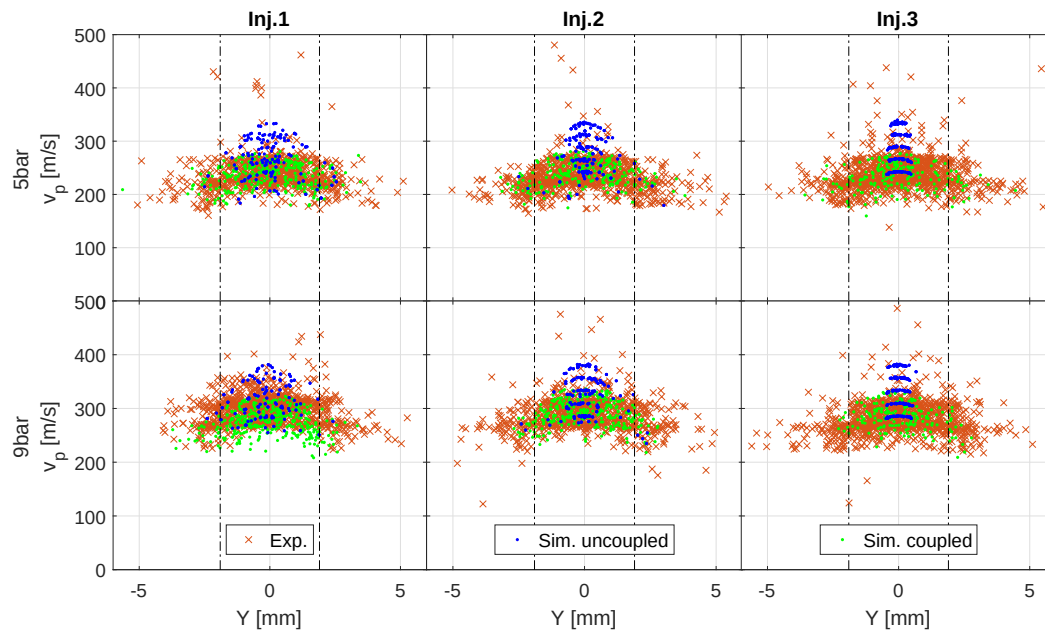


Figure 6.19: Stellite particle velocity footprint validation at 40mm SoD, comparison of pressures, injectors 1-3, full (coupled) and simplified (uncoupled) model

The absolute velocity values delivered by the full model are valid and the span of velocity values is acceptable as well, in spite of a small fraction of particles, representing the extreme values, which are missing in the simulation. The dispersion, however, is underestimated by the model. Although the dispersion in the converging section is over-predicted by the full model due to the boundary condition, in combination with the collision model, this does not result in sufficient sideways-directed forces to disperse particles in the jet. It can be seen that the model covers the most part of the measured particle beam width for 5bar, but remains too straight at 9bar, such that roughly 25% of the track are not in agreement at the design pressure. For injector 2 and 3 this observation gradually aggravates, because the dispersion reduces with decreasing distance between injection port and nozzle throat in the simulation, while in reality, this position has minor influence. Moreover, the uncoupled simulation, due to reasons discussed with respect to the dispersion upon injection, behaves unphysically at the spray spot. Injector 3, which injects directly into the nozzle throat, causes strongly accumulated trajectories on the centreline. Respective data points also show velocity bands biased by the particle size due to lack of dispersive mechanisms. The intermediate injector amends this situation, but the particles still exhibit velocity bands and remain close to the centre. In the model with injector 1, the nozzle exit cross section is almost covered by particles but dispersion beyond is non-existent. This model is therefore invalid in this respect.

The same comparison can be made for Titanium. Having the lower Sk -number range, the particle trajectories may react more strongly to the fluid phase and therefore deliver a different degree of validity. The according data is given with figure 6.20, which has an analogous structure as the previous plots.

The uncoupled model shows the same mismatches as in case of the heavy material. Here, an underestimation of the velocity level by the full model, in agreement with previous analysis in section 6.3, can be seen. More importantly, the calculated span of velocities is not covered either. The experiments show a much wider range, predominantly spread out to higher values. Similarly, the dispersion is not adequate either and differs by almost a full nozzle exit height in all cases. Interestingly, the models as employed are relatively well-set for the higher Sk -range, while they lack dispersive and acceleration forces and in case of the lighter material. Nonetheless, the particle phase reaction to the growing gas

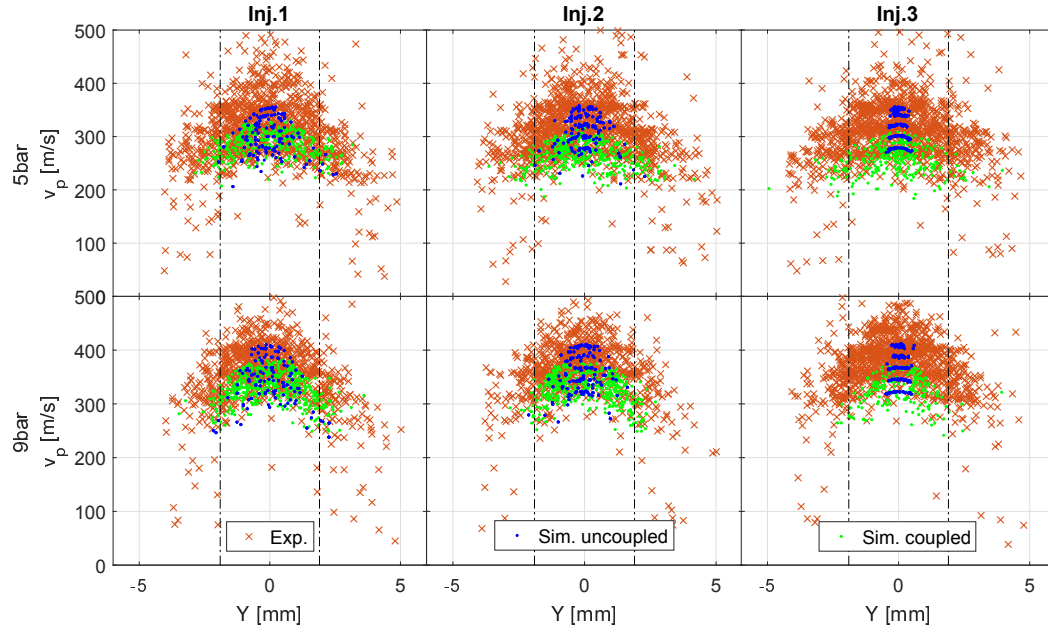


Figure 6.20: Titanium particle velocity footprint validation at 40mm SoD, comparison of pressures, injectors 1-3, full (coupled) and simplified (uncoupled) model

shear layer is captured well, which can be seen by the velocity profile curvature that is stronger for the lighter material and increases with pressure. Table 6.5 summarises these findings by comparing the size of the particle footprints.

	Ti	Inj.1	Inj.2	Inj.3	St	Inj.1	Inj.2	Inj.3
exp.		5.74	5.97	5.82		5.05	5.53	5.05
5bar coup.		4.48	4.59	3.50		4.19	4.51	4.69
uncoup.		3.79	2.38	0.75		4.20	2.26	0.78
exp.		5.67	5.18	5.12		4.18	5.52	5.22
9bar coup.		4.06	3.87	3.20		3.77	4.18	4.09
uncoup.		3.88	1.86	0.87		2.74	2.72	0.86

Table 6.5: Comparison of spray footprint widths at 40mm SoD (3.8mm nozzle exit width, values in [mm]).

As a sidenote, the lack of potential for outward movement of particles is already reflected in the particle velocity profile at the nozzle exit. A very flat particle velocity profile stands for a homogeneous phase that, when released from the nozzle, strives to equalise the positions more strongly. However, the simulations show that the exit profile curvature is too strong in comparison to the measurements (app. p.249). This substantiates above observations, in particular the unrealistically low amount of particles interacting with the

walls of the diverging section.

Dispersion diagrams

In analogy to chapter 5.6.2, the dispersion can be characterised in more detail by a diagram that gives the trajectory angle with respect to position within the cross-section at a given stand-off distance. This dispersion diagram can be extended by a regression fit and a covariance ellipse, in order to visualise the statistical tendency of particles to move straight, or outwards and disperse the plume, or inwards and reduce plume growth in spite of steeper angles. The slope of the regression indicates the tendency to move outwards, while the width of the ellipse stands for the cross-motion, while the spot size can directly be seen from the abscissa. Using this type of diagram, the simulation results can be compared to the experimental data in figure 6.21, where the respective simulation data is given for a SoD of 40mm in scatter and solid lines for Stellite and Titanium at low feed rate (uncoupled model) and high feed rate (full model). It is compared to respective measurements, which are dotted in the background, for varying pressure and injector location.

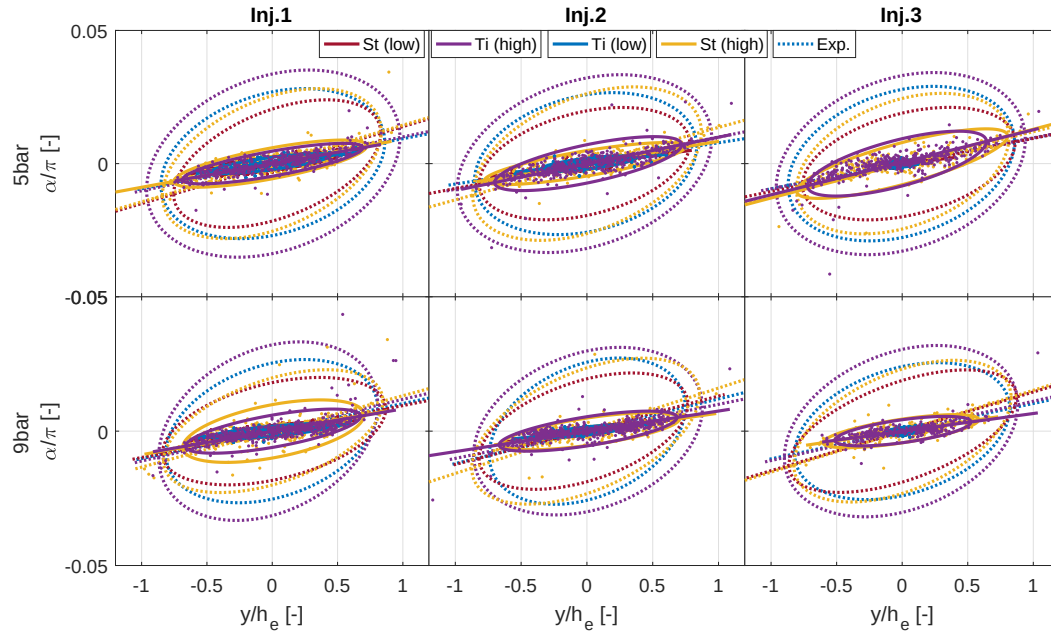


Figure 6.21: Dispersion diagram for the characterisation of particle plume growth for varying pressures and injectors at a stand-off distance of 40mm

The differences between simulations and experiments are unfortunately drastic, and the quality of these differences is similar for all compared cases. The regression lines

are simulated too flat, i.e. the calculation does not predict sufficient outward movement. This is directly connected to the fact that the calculated spray spot is smaller than the experimentally observed width. Moreover, the ellipses are of completely different size, which substantiates two aspects. Firstly, the underestimation of the plume size at this SoD can be seen by the too small ellipse length. Secondly, with regard to the ellipse width, it shows the massively miscalculated cross-motion of particles. The trajectories are by several factors directed to straight and hence unrealistically ordered. This effect is worse for Titanium, for which the unordered motion is stronger. Another point, which was alluded to in the velocity profiles, is the spot reduction with decreasing injector distance from the throat. In particular at 9bar pressure, this is a very strong effect, that is not reflected in the experiments. Models with injector 2 and 3 provide the most drastic underpredictions of the spray spot for the uncoupled (low feed) case. In order to investigate the individual loadings separately, the case of injector 1 is used, such that both the full and the uncoupled model can be assessed by separately showing the two loading states for the two materials in figure 6.22.

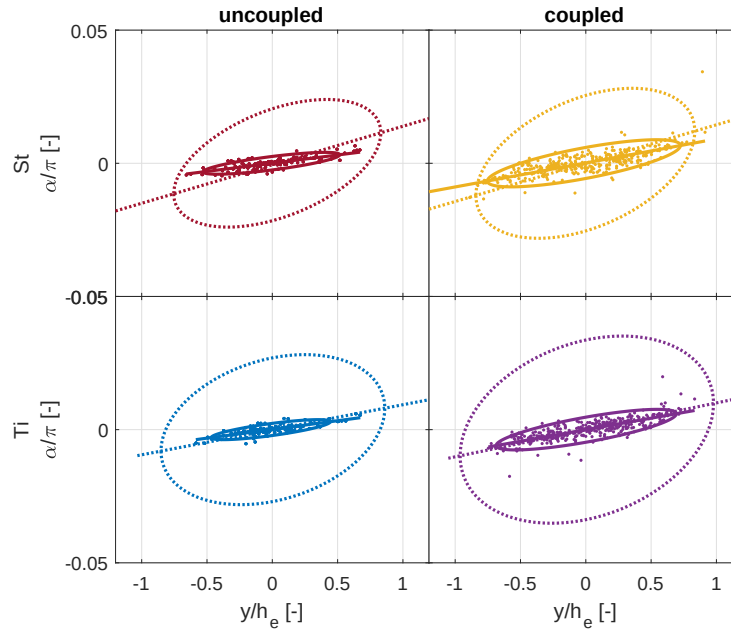


Figure 6.22: Dispersion diagram for uncoupled and coupled model with injector 1 at a stand-off distance of 40mm

Here, it can be seen that the underestimation of the slope, i.e. the outward dispersion, is worse for Stellite than Titanium. The high Sk -numbers prohibit an increase in dispersion

rate while travelling downstream in the jet, such that without a strong random motions within the nozzle, Stellite does not spread outwards sufficiently in the simulations. In contrast, the spreading rate of Titanium approaches a valid state, however too late to provide good spot size results. This is connected to the growing turbulence level in this region of the jet, which more rapidly influences the light material in the simulations. Dispersion due to the nozzle-internal motion is not adequately represented upstream of this, leading to the smaller spot. Both materials show the same differences with respect to the ellipse fit, but the previously found stronger miscalculation of cross-motion in case of Titanium becomes clearer. Randomness of particle motion is clearly more strongly underestimated for this material. The uncoupled simulation performs worse in all aspects than the coupled simulation for obvious reasons. In terms of error values, table 6.6 gives a summary of the underestimation of the dispersion. It gives the spot size error, as well as the outward and cross-motion error for injector 1. While injector 2 and 3 are predicted with similar accuracy with the full model, the uncoupled model deteriorates more heavily to factors up to 5.

	St		Ti	
	uncoupled	coupled	uncoupled	coupled
Spot error (y/h_e)	-37.9%	-10.7%	-49.3%	-26.1%
Outward-motion error (slope)	-57.4%	-37.4%	-30.4%	-14.4%
Cross-motion error (width)	-78.8%	-79.5%	-83.1%	-67.1%

Table 6.6: Error levels of dispersion for injector 1 at 40mm SoD

6.5 Prediction of operational challenges

The previous sections 6.3 and 6.4 discuss two important requirements for a holistic computational model for CS applications, the velocity and dispersion prediction capabilities. Velocity is clearly directly connected to deposition, already answering the question for its relevance. This is different for dispersion, as the relevance is more subtle, but some examples are nonetheless easy to find: the spot size is directly connected to powder consumption and geometric build-up, e.g. crucial in additive manufacturing. Moreover, the trajectory angle is core element of the dispersive motion and determines the impact angle

of the particle onto the substrate. In this study, the nozzle was not designed for deposition itself and no substrates were present in experiment and simulation, so it should merely be emphasised that the dispersive properties of a particle stream can influence the efficiency in the ways analysed above, with special awareness of particle cross-motion.

These two aspects, velocity and dispersion, not only carry value for the application individually. Combined, they can be used to advance the simulation capabilities of heating residence times and nozzle clogging risk. These aspects are addressed in this section as a third main question to answer: can the model be used to assess advanced operational problems with high relevance? For details of the experimental findings, the reader may refer to chapter 5.

6.5.1 Residence times

The particle residence time has relevance for the heating of particles. Although the average particle velocity level can be well-calculated using these models, the particle temperature remains unknown, as measurements of such are not possible. Nevertheless, the particle impact temperature is important for deposition as it contributes to the critical velocity. In practise, these temperatures are obtained computationally. Depending on the injector location, the particle residence time is used as a pre-heating parameter. When particles must pass a longer distance in the low velocity section, they are exposed to the process gas temperature for a longer time. At the same time, the particles disperse internally. Because these aspects go hand in hand, the pre-heating of particles and the dispersion are directly connected. In addition, dispersion is result of the statistical distribution of velocity and trajectory angle, which also determines how fast the particles may pass through the heating section. Model-wise, it is clear that mostly the collision model and the DPM boundary condition influence the adequacy of the model with respect to particle residence times, which makes the employment of the full model necessary. Figure 6.23 shows the axial profile of the injection velocity including the standard deviation intervals of the local dynamic range for injector 1.

It illustrates the influence of the model on the velocity distribution, which defines the particle residence times. The lowest feed rate is represented by the uncoupled model, which does not adequately capture the observable velocity span. The full model amends

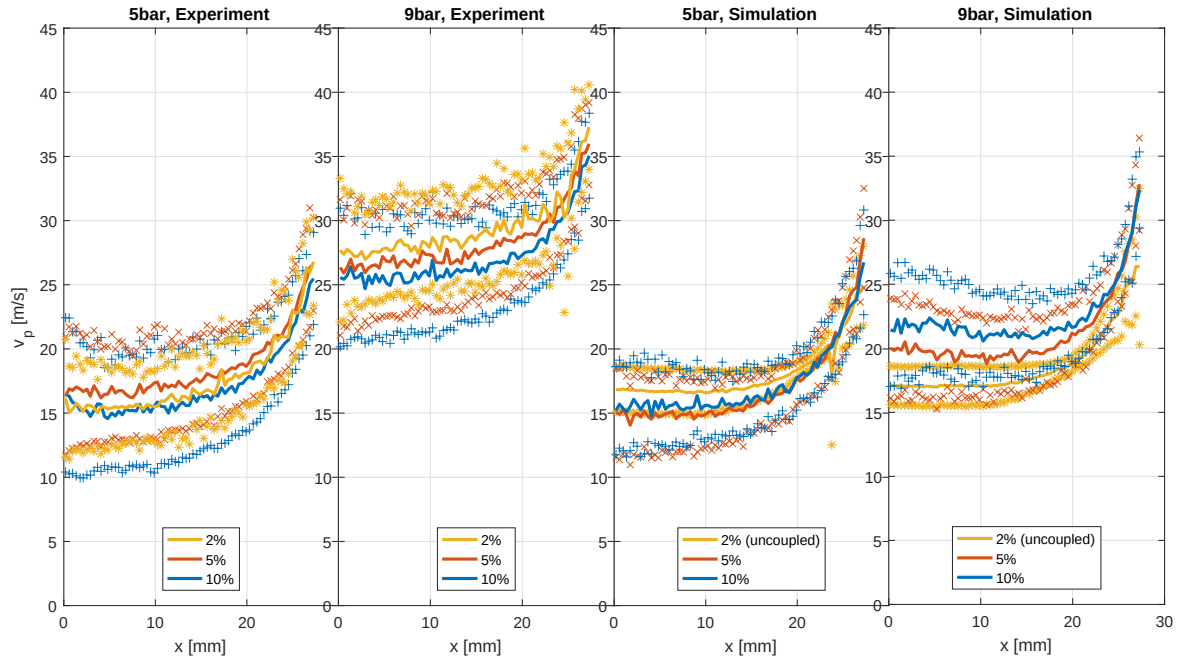


Figure 6.23: Longitudinal velocity evolution (St) in the converging section with injector comparing simulation and experiment

this noticeably, in particular at 5bar pressure. With growing feed rate, the range is increasingly under-predicted. Moreover, at 9bar, the velocity level of the simulation is too low, despite the UDF boundary condition in use, due to a reduced gas flow rate in the injector line. This reflects back onto the pressure-type boundary condition for the gas phase, which is not controlling the gas velocity within the injector. Although the particle phase is injected with a correct velocity level, the gas phase decelerates it slightly before emitting it into the nozzle. This has direct impact on the residence times. Consequently, if these are of interest, it is therefore advisable to target the gas phase velocity in the injection line to avoid such differences. Nonetheless, the residence times derived for the present data capture many aspects relatively well. Figure 6.24 summarises these, including the average values and the statistical span of residence times accordingly.

With respect to the qualitative trends in the figure, generally good agreement can be noted: While Titanium times decrease with increasing feed, Stellite times slightly increase. Caused by the semi-empirical boundary condition in combination with the collision mode, also the growth of the span calculated by the models is plausible. However, the absolute values of the ranges remain too small, which is explained above by the underestimated velocity range. The quantitative validity differs with material. Average Stellite residence

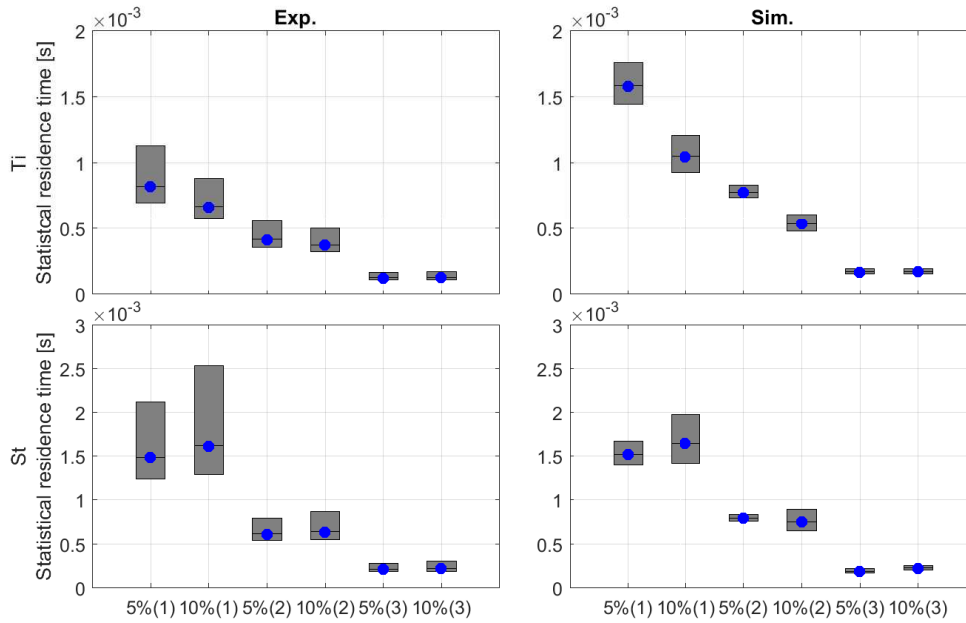


Figure 6.24: Validation of residence times of particles in the converging section of the nozzle, feeder wheel speed and injectors varied

times are represented well, with low errors for all setting, despite the underestimated range. This way, injector 1 leads to about $1.5ms$ in both experiments and simulation, injector 2 to half that with a tendency for over-estimation, while injector 3 causes negligible times that are similar for measurement and calculation. Titanium is represented less correctly. Because it is a result of the velocity level, errors in the speed sensitively reflect in the residence times. Apart from injector 3, all simulated Titanium results are too high, approximately by a factor of 2. Respective particles are more strongly affected by collisions and the overly high collision rate in the dense region of the model in combination with gas velocity differences leads to this observed mismatch.

Consequently, heating of heavier materials can be predicted more easily, although big particles may turn out too cold and small particles too hot because of the too narrow span of times. Lighter materials, in particular at low feed rates, tend to remain in the nozzle unrealistically long and hence acquire too high temperatures. The influence of pressure on residence times is implicitly captured with acceptable results by this type of model (app. p.250).

6.5.2 Nozzle clogging

In section 6.4, the dispersive motion is found to be slightly over-predicted in the injection region but noticeably under-predicted in the high velocity region down to some stand-off distance. Although this is even true for the most advanced model, it is the only version that can avoid unphysical clustering of particles and hence approaches the real distribution best. The other models disperse particles so weakly that particle-wall interactions rarely occur with relevant frequency and mostly only take place directly at the nozzle throat, which is dissimilar to the measured distributions. Wall interactions are the necessary condition for nozzle clogging risk, which has great importance for current nozzle analysis in CS. This risk is increased with more energetic wall collisions, i.e. with higher wall-normal velocity components upon interaction.

Therefore, the full model is evaluated in terms of its wall-normal velocity components analogously to the experimental clogging analysis in chapter 5.5. This comparison is shown in figure 6.25.

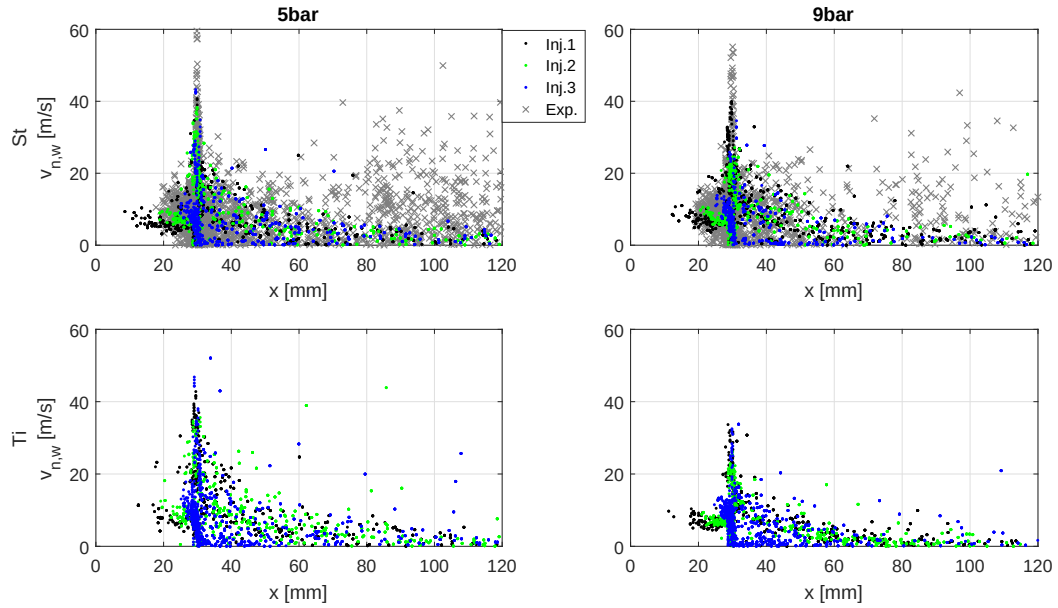


Figure 6.25: Wall-normal particle velocity within the observable section of the nozzle, comparison of simulations and experiments

As can be seen here, the normal velocity components are in good agreement for all injectors. Although a small fraction of particles interacts with the nozzle wall too far upstream when compared to measured data, the absolute values are very similar in the

converging section and the throat. At the restriction, the observed increase of wall-normal velocities, formed as a peak, is also adequately represented in length and magnitude at both pressure levels. In the simulations, downstream of this, the normal velocity component gradually decreases. In spite of a similar decrease of the majority of particles in the experiment, the simulations lack the increase of spreading and the resulting higher normal velocities of a minority of particles towards the end of the diverging section. In other words, due to the weak dispersion and low randomness of particle motion in the diverging section of the numerical model, the risk of high-velocity clogging is underestimated. At the same time, the contrary is true for the converging section, so that the domain of influence of the high velocity section is overestimated.

An approximate low speed clogging analysis is therefore possible with such a model, while the high speed mode is problematic in the current state. As this goes hand in hand with dispersion, the validity can be greatly amended by adapting the collision model in such a way, that it is relaxed in high volume fraction zone but more firm in the low density region. Only if this is guaranteed, the use of an erosion/accretion-model to mimic the actual clogging process makes sense. On a scientific level, a discrete element method (DEM), which calculates all particle collisions deterministically, may be helpful for reference data generation in future work. For industrial applications however, this is prohibitive and hence uninteresting due to the computational effort (besides the unresolved issue of stability of such models in CS nozzle flows). With an adapted stochastic model, estimation not only of low-speed but also of high-speed nozzle clogging is theoretically possible. In this regard, further understanding of the deposition mechanisms is required and therefore left as a recommendation for upcoming work.

6.6 Summary

This chapter introduced an advanced computational model for the flow channel of the quartz nozzle NZTR in the environment *Ansys FLUENT*. This model rebuilds the three-dimensional geometry for all injectors and solves the gas phase equations in a Eulerian-, and the particle movements in Lagrangian reference frames. It comprises, in its full version, the advanced methods of a two-way phase coupling, a full turbulence coupling, stochastic

collision modelling, and a tailored discrete phase boundary condition. The analysis in this chapter included the assessment of the velocity prediction capabilities, the dispersion trends and adequacy, as well as resulting consequences for further relevant two questions in CS nozzle flow analysis: particle residence times and nozzle clogging.

It was found that the full model is the most appropriate in order to mimic these aspects. The methods deliver valid results for the particle exit velocity and trends of the reaction to increasing loading that lie within the range of measurement certainty. The drag law was assessed by virtue of the overall and maximal acceleration of particles. Over the whole distance of the nozzle, it provides good results and follows the measurements closely. It predicts excessive peak acceleration values, but since the relative velocity adapts over the length of the nozzle, the final result is widely insensitive to this mismatch.

In contrast to the high validity of the velocity prediction, the dispersive motion of particles is less appropriate. Within the nozzle, the dispersion is highly sensitive to the injection type, geometry, and collision model. Turbulence plays a subordinate role and becomes important only in the jet for rather light particles. The dispersion plume at the injection needle can be amended by a user-defined release function of the DPM, but the collision model is too dispersive in combination with the latter when the volume fraction of particles is high. When it is low, the opposite is true, and particles disperse too little. The result is a too narrow particle spray spot, underestimated cross-motion of particles in the jet, and a relatively low dispersion rate with respect to measurements.

These findings, combined with each other, result in good trends of average residence times within the converging section, which is important for particle temperature prediction. The weakness is absolute values of light materials, which tend to remain too long in the warm part of the nozzle. The statistical range of residence times is approached by the most advanced model, but remains miscalculated with low values, such that small particles may be hotter and big particles colder than in reality when computed in this manner. Low-speed (throat) nozzle clogging can be assessed with the full model and gives valid particle-wall collision speeds, while the lack of dispersive motion in the diverging section prohibits the assessment of high-speed clogging. Adaptation of the collision model and the gas phase boundary conditions for correct gas velocity in the injector, as well as an improved version of the user-defined DPM boundary condition are likely to resolve

many of the found deficits. Particle-wall interactions play a major role in the dispersion and future studies are recommended to aim towards better understanding of such.

In summary, this chapter showed that the experiments introduced in this work could be used to validate an advanced numerical model in detail, using methods entirely new to the field of CS. The experiments showed valuable in adjusting boundary conditions and lead to an analysis, which was able to identify which aspects of computations, both fundamental and directly relevant for applications, are valid or should be improved in future work.

Chapter 7

Conclusions and future work

The present study reported the first nozzle-internal particle velocity measurement in the field of Cold Spray and demonstrated the use of such analysis for advanced numerical model validation. In this respect, the following points can be concluded.

7.1 Conclusions

Gap of knowledge

The background review introduced numerical and experimental methods of the fluid flow in Cold Spray, and pointed out that some areas are well-represented by both means, such as the nozzle design [43, 93, 97] and the analysis of main process parameters [27, 30, 109]. The review concluded that many numerical investigations are weakly supported by validation: predictions lie inconsistently over or under the measurements to strongly varying degrees [121]. This was attributed to a large number of different models, in particular to the phase-coupling and representation of particle loading. In face of this, models could be amended through further studies on the coupled fluid dynamics. Firstly, more detailed knowledge on the influence of particle loading, including varying materials and operating conditions, was required [83, 154, 167]. Secondly and most importantly, literature also implied that observations within the nozzle were the only way to dissolve the “black box” of particle motion upstream of the nozzle exit. This resulted in definition of four objectives, with the following outcome.

Objective 1: Extension of indirect investigations on gas-particle coupling

Consequently, the first objective set for this work was the extension of the current understanding of the gas-particle momentum exchange, of the influence of mass loading and the consequences for particle dispersion. By changing the particle loading, powder material (Stellite, Titanium, and Aluminium), as well as pressures (up to 30bar), while operating an application-tested nozzle (NZ1), the present work pursued this goal, using the common approach of particle measurements downstream of the nozzle exit. The particle velocity decrease due to particle loading was firstly compared for different materials and operation conditions, describing the flattening of the radial velocity footprints with increasing loading and with increasing material density (hence Stokes number). Moreover, the study characterised intensified particle dispersion and an average particle velocity reduction under augmented feed rate, in part as high as 20%. This relative drop in velocity can be influenced by both the relative volume fraction and the relative mass loading. For limits of high particle velocity parameters or Stokes numbers, the driving mechanism tends to be limited to the mass loading, while at a low velocity parameter or Stokes number, the volume fraction is an increasingly characteristic driver. Despite adding these novel insights on the mixture behaviour, the responsible mechanisms take place within the nozzle, further motivating the remaining objectives. Findings of this chapter were published in [173, 174].

Objective 2: Transparent nozzle and direct experiment design

A transparent quartz nozzle was therefore designed, along with a measurement rig to combine Cold Spray and Particle Tracking Velocimetry systems. The conceptual approach emphasised the importance of a hard, highly clear material for the nozzle (quartz), which leads to a pressure limitation of 9-10bar. The nozzle expansion ratio and length were designed for this level and its composition into four pieces, two contour- and two imaging blocks, facilitated imaging and leak-tight assembly. Stellite was set as the main feedstock material, and compared to Titanium where possible, at pressure levels up to 9bar, feeder wheel speeds between 1 and 20% and injector designs for injections between 80 and 9mm

upstream of the nozzle throat.

Objective 3: Direct analysis of nozzle-internal particle motion

Volume-related loss and dispersion mechanisms could be connected to particle-particle interactions and primarily take place in the converging section of the nozzle. Mass-related loss and dispersion mechanisms could be connected to the overall forces throughout the nozzle-internal particle flight, making the injection and the diverging section likewise important. Respective phase-interactions were hence directly observed by nozzle-internal particle velocimetry and the following main aspects were concluded:

- A homogeneous particle stream entering the nozzle showed an unexpected increase in average particle velocity with loading in the low speed section, which increased with pressure, while the particle velocity in the jet dropped, which decreased with pressure. A more randomised motion due to enhanced inter-particle collisions was deemed the possible explanation.
- Average peak accelerations in the throat were found to be as high as $8.7 \cdot 10^5 m/s^2$, which continues regressively downstream of the throat and jet.
- The dispersion of particles at the injection is approximately linear with distance and at well-set main-line to feeder-line flow ratio, the particle velocity is independent of distance from the nozzle centre.
- Normal probability density functions of velocity and trajectory angle found that the downstream shift of the injector location reduces the dynamic range of velocities. Increasing pressure aligns particle trajectories with the x-axis. Titanium particles tend to have a velocity distribution that agrees less with a log-normal profile, that corresponds to drag-driven motion, than Stellite. Lower Stokes number range causes a stronger deviation from this theoretical case, due to more drastic path changes upon collisions in the longitudinal directions.
- The internal particle motion could be used to gather new insights into the phenomenon of nozzle clogging. Two types of clogging were distinguished: low velocity

(throat-) and high velocity (exit-) clogging. Both types have a similar order of maximal wall-normal velocity components, suggesting a different particle deformation pattern for high velocity clogging. Average particles are likely to collide with walls throughout the nozzle in all cases and a beneficial injection location could be derived.

- Resulting from the internal motion, the dispersion in the jet shows different trends with changing materials: Stellite particles mostly disperse outwards, Titanium particles have a more randomised motion and consequently higher probability to move back towards the centreline at more strongly inclined trajectories.

Objective 4: Demonstration of novel computational model validation

The generalisation of such experimental findings is by nature difficult. Therefore, the final objective of this work aims to benefit the scientific community in the most direct way, hence by demonstrating computational model validation and identifying the adequacy of respective modelling aspects. An advanced model was introduced for the flow channel of the quartz nozzle in the environment *Ansys FLUENT*. The full model had the following features, of which each added advanced level of complexity was investigated individually:

- Exact three-dimensional fluid domain rebuild.
- Eulerian-Lagrangian framework (Discrete Phase Model for particle phase).
- RANS for the gas phase with k - ε -turbulence model and non-equilibrium wall functions.
- Discrete log-normal Rosin-Rammler particle size distribution.
- *Advanced*: Two-way momentum and energy coupling.
- *Advanced*: Full turbulence coupling including gas modulation.
- *Advanced*: Stochastic collision model.
- *Advanced*: User-defined particle release boundary condition.

This analysis included the assessment of the velocity prediction capabilities, the dispersion trends, as well as resulting consequences for two relevant contemporary challenges in Cold Spray operations - particle residence times and nozzle clogging. Following points could be concluded:

- The full model was the most appropriate for all aspects. The method delivered valid results for the particle exit velocity for Stellite and slightly underestimated results for Titanium due to small errors in drag force. Trends of the reaction to increasing loading lie within the range of measurement certainty.
- The drag law was assessed by virtue of the overall and maximal acceleration of particles. Over the whole distance of the nozzle, it provides good results and follows the measurements closely. It predicts excessive peak acceleration values, which is equalised well over the length of the nozzle. Light materials are more sensitive to this.
- The dispersive motion of particles is less appropriate, as the dispersion is highly sensitive to the injection type, geometry, and mostly collision model. Turbulence plays a subordinate role and becomes important only in the jet for rather light particles. The distribution can be amended greatly by a user-defined release function in combination with the collision model, but stays overly dispersive when the volume fraction of particles is high, and the opposite for low volume fractions. The result is a too narrow particle spray spot and underestimated cross-motion of particles in the jet.
- Good trends of average residence times within the converging section can be simulated, which is important for particle temperature prediction. On average, the light material remains within the warm part of the nozzle up to 1.8 times too long when calculated, while the heavy material behaves realistically. The statistical range of residence times is only approached by the most advanced model, but their values remain low, such that small particles may be hotter and big particles colder than in reality when computed in this manner.
- Low-speed nozzle clogging can be assessed with the full model and gives valid

particle-wall collision speeds. These wall collisions are important for correct dispersion but respective mismatches in the high-speed section imply invalid reflection laws. Consequently, the assessment of high-speed clogging is not yet possible, due to a lack of dispersive motion in the diverging section.

Relevance for industry

Although this study primarily offers benefits for research, as future studies can advance the presented aspects to learn about the CS process, the benefit reaches beyond the academic sphere. As CS research is tightly interconnected with industrial applications and their challenges due to the vast innovation opportunities. The emerging new difficulties remain to be overcome, and CFD is a key tool in this process.

CS industry relies strongly on the discussed commercial computational tools. Very unique powder and substrate materials, or difficult-to-reach locations require specialised nozzle designs and therefore high-quality CFD models. For industrial applications, the idea to calculate the statistical distributions of powder particles upon impact, to which this study significantly contributes, offers a substantial benefit. It can lead to a method to derive the efficiency of deposition prior to testing, which would open up opportunities to operate in pre-calculated optima. This can reduce the need to re-iterate tests with new material combinations and help deriving required powder properties for a certain optimal operation of the production line.

For industrial applications, the presented methods should be extended to high pressure regions (see recommendations), but initially, similar CFD models could directly be employed for higher pressure regimes, expecting similar accuracy for the particle velocity, as this precision does not strongly change with pressure. In contrast, the dispersion of particles will be increasingly underestimated, as the calculated spray dispersion excessively reduces with pressure. It may be useful for industrial applications to extrapolate the errors to account for the additional mismatch, until future studies extended the present findings to high pressure range. The benefit over alternatively using simplified calculations that cannot assess the dispersion is still significant.

Detrimental nozzle wear reduction is a challenge that is constantly faced in industry, and this work provides new information about the topics of nozzle clogging. Industrial

developments of CS nozzles may therefore make use of the beneficial injection location, which minimised the clogging risk, or apply the presented holistic model to their nozzle design processes. This may lead to development of novel injector or nozzle geometries based on suggestions from this work. The same innovation process is likely to lead to the definition of new shapes that influence the particle beam, an ability which would be a very distinguishing market advantage for companies. CFD models that may enable these aspects for a larger fraction of industry and research, would represent the benefits defined in section 2.5. In this context, this work contributes in the following way.

- Higher accuracy and reliability estimations of particle velocity and temperature:
The particle velocity estimations under increasing loading conditions can be calculated with the presented model. For thermal aspects, the particle residence times in the converging section are crucial, and the presented model can cover this aspect. Future work (details below) may further refine the accuracy for light materials.
- Statistically valid impact properties enabling derivation of deposition efficiency:
The model covers calculation of the statistical range of velocities, which is only slightly underestimated. This can lead to correct estimation of the deposition efficiency in some cases, although this is not yet generally possible due to underestimated particle dispersion.
- Possibility to calculate particle dispersion, impact angle and spray spot:
Calculating the dispersion of particles is significantly improved over simplified methods, and the spray spot can be approximated with some underestimation. However, the dispersion calculation has still some deficits, such that future studies are yet required to improve dispersion through particle collisions, in order to reliably predict the impact angle.
- Development of nozzle designs compensating for loading and dispersion:
As the model offers above improvements, shapes can be tested for reduced dispersion and favourable designs chosen according to computed dispersion trends. Nozzles that are specifically designed for a single powder can include the loading effects and hence pin-point the resulting impact velocity range.

- Prediction and prevention of nozzle clogging with respect to powder injection:

This study offers a strategy to reduce nozzle clogging based on the particle dynamics. Prevention of the phenomenon is questionable, but the reduction of the risk is possible also computationally for the low-speed (throat) clogging mechanisms.

7.2 Recommendations for future work

Based on these conclusive findings, the following aspects are recommended for future work on Cold Spray flow analysis and optimisation.

Nozzle design

The measurement approach introduced in this work can be amended on some levels. With respect to nozzle design, one of the main limitations to date is the nozzle design pressure. Due to the interplay of manufacturability, imaging and illumination quality, assembly and handling, the quartz glass solution was limited to a pressure of 9-10bar. Although many Cold Spray applications employ this pressure level (in particular for hard substrates), the majority of depositions is only possible at significantly higher pressures. A success of future studies that may emerge from this basic work would be analogous measurements at higher pressure levels. With new design and sealing ideas, pressures of up to 20bar are deemed possible, however with the corresponding financial expenditure. Likewise, the imaging quality and hence measurement error can be reduced.

Feed rate effect

Although this study found new aspects on the effects induced by feed rate, their characteristics change with material and most likely with size distribution. It is important to investigate a larger variety of materials individually in this regard. This does not necessarily mean nozzle-internal measurements, but rather a stronger focus of velocity and dispersion changes in the jet, which are induced by varying the particle loading. Observations of impinging particles, e.g. by high speed imaging of the actual impact are certainly another possible example of progressive experimental analysis to gather information about impact angles and velocities, potentially tying them to deformation patterns.

Nozzle-internal measurements

One important focus of potential future measurements inside Cold Spray nozzles should be the simultaneous recording of particle size and velocity. Overcoming the challenges in combining techniques like Particle Shadowgraphy with the transparent nozzle may lead to such a possibility. Herein, the accuracy and the operation at sufficiently high feed rates are expected to be most difficult to integrate. Another point may be the resolution of the field, limited by the magnification of such methods. Apart from the particle in-flight size measurement, a temporal resolution of the particle trajectory, even restricted to specific sections of the field, would be very beneficial. Evaluation of individual particle paths, incident and emerging angle from wall-collisions and diversion angles and curves from particle-interactions are examples of the additionally obtainable information. A third point would be the extension of the data set by more detailed resolution of the operational settings. Since this is the very first study of this sort, it is inevitable that the data is highly specific and that findings are limited to parameters within the investigated bounds. Extension of such is consequently key to more detailed understanding.

Construction of highly advanced models

This study suggested several opportunities to advance the modelling in Cold Spray applications. Some of these are not affordable computationally, such as the resolution of turbulent fluctuations or the employment of Reynolds Stress Models. Others are directly connected to the findings in the study and may easily lead to improvements in future work. The main points in this respect are the adaptation of the drag coefficient and particle collision model, the correction of the particle release function, the adjustment of the gas flow boundary in the injector and the improvement of the wall-particle collisions. The combination of these aspects in future work may lead to models to have statistically correct representation of particle velocity span, dispersive spray spot, risk or even time-frame of nozzle clogging, and finally particle heating time ranges. Following this path, in modern computations in Cold Spray, the deposition efficiency and coating properties could be predicted due to the knowledge of the origin and variance of all particle impact properties. During the process of such investigations, new ideas and means may arise that allow for the manipulation of these properties in unforeseen ways.

Experiment

Converging section

- Velocity range, average speed, orientation range increase
- Statistics indicate particle-particle collisions
- Stronger for light material
- Peak acceleration as high as $8.7 \times 10^5 \text{ m/s}^2$

→ Next: nozzle for high pressure, faster imaging

Injector

- Injection plume linear with distance
- Velocity uniform in cross-section
- Needle position shift reduces velocity range

Clogging

- Low velocity and ...
- Similar maximal wall-normal ...
- Up to 93% of particles ...
- Collision probability high ...
- Collision speed high ...

→ Next: develop favourable ...

Injector

- Needle-internal particle properties initiate later dynamics
- Injector geometry and internal dispersion are essential
- Clustering of particles at centreline due to biased paths

→ Next: Improvement of particle-wall collisions and adjustment of injection statistics

Clogging

- Wall-normal velocity level is good in converging section and throat ...
- Number of collisions over- ...
- Injection downstream shift: collision probability ...

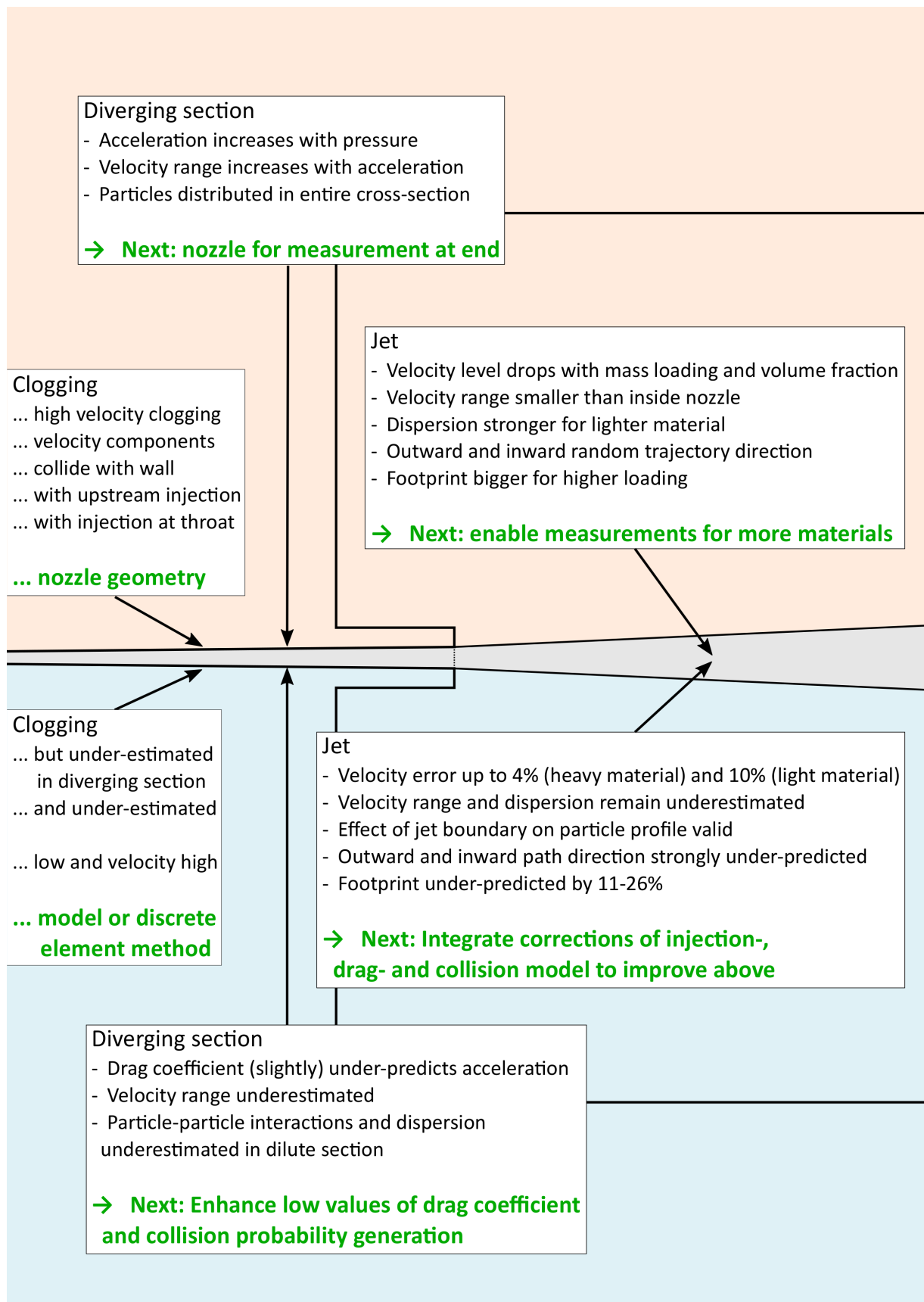
→ Next: Correction of particle-wall collision ...

Converging section & throat

- Simple model under-predicts velocity and dispersion
- Particle-particle collisions over-predicted in dense part
- Particles dampen turbulence level
- Particle residence times over-estimated
- Drag coefficient over-predicts peak acceleration by 10-60%

→ Next: Dampen peak value for drag-coefficient and collision probability generation

Simulation



List of Publications

The following is a list of the authors publications and presentations associated with this work.

Reviewed journal publications

- Meyer, M., Yin, S., & Lupoi, R. (2017). Particle in-flight velocity and dispersion measurements at increasing particle feed rates in Cold Spray. *Journal of Thermal Spray Technology*, 16(12), 60-70.
- Meyer, M., Yin, S., McDonnell, K. A., Stier, O., & Lupoi, R. (2016). Feed rate effect on particulate acceleration in Cold Spray under low stagnation pressure conditions. *Surface and Coatings Technology*, 304, 237-245.
- Meyer, M., & Lupoi, R. (2015). An analysis of the particulate flow in cold spray nozzles. *Mechanical Sciences*, 6(2), 127-136. (Contribution invited by committee of 31st International Manufacturing Conference).
- Yin, S., Meyer, M., Li, W.-Y., Liao, H., & Lupoi, R. (2016). Gas Flow, Particle Acceleration, and Heat Transfer in Cold Spray: A review. *Journal of Thermal Spray Technology*.
- Stenson, C., McDonnell, K. A., Yin, S., Aldwell, B., Meyer, M., Dowling, D. P., & Lupoi, R. (2016). Cold spray deposition to prevent fouling of polymer surfaces. *Surface Engineering*, 844(September), 111.
- Stenson, C., Meyer, M., Twomey, B., & Lupoi, R. (2015). Particle speed analysis in CoBlast. *Procedia CIRP*, 37, 7782.

Conference presentations

- Meyer, M.(2017). Nozzle-internal particle velocity measurements and loading effect on

particle acceleration inside a Cold Spray nozzle. Presentations of the Young Professionals Award Competition at the International Thermal Spray Conference 2017, Düsseldorf, Germany.

- Meyer, M., Caruso, F., & Lupoi, R. (2017). Nozzle-internal particle velocity measurements and loading effect on particle acceleration inside a Cold Spray nozzle. In Lectures and Posters of the International Thermal Spray Conference 2017, Volume 336 (pp. 214-220). Düsseldorf, Germany.

- Meyer, M., Yin, S., & Lupoi, R. (2016). Particle in-flight velocity and dispersion measurements at increasing particle feed rates in Cold Spray. In Lectures and Posters of the International Thermal Spray Conference 2016, Volume 324 (pp. 37-42). Shanghai/P.R. China.

- Meyer, M. (2016). Supersonic Spray Advanced Modelling - SSAM. Posters of the 19th Sir Bernard Crossland Symposium. Belfast, Northern Ireland: Queen's University Belfast.

- Meyer, M., & Lupoi, R. (2014). Computational Prediction of Cold Spray Nozzle Performance for the Deposition of Titanium. In Proceedings of the 31st International Manufacturing Conference Cork Institute of Technology (pp. 107-112). Cork, Ireland: Cork Institute of Technology. (Contribution awarded for second best presented manuscript).

- Aldwell, B., Trimble, D., Meyer, M., Robbe, C., & Lupoi, R. (2015). A novel strategy for the deposition of diamond based materials with Cold Spray, with particle speed analysis. In International Thermal Spray Conference 2015. Long Beach, California, USA: ASM International.

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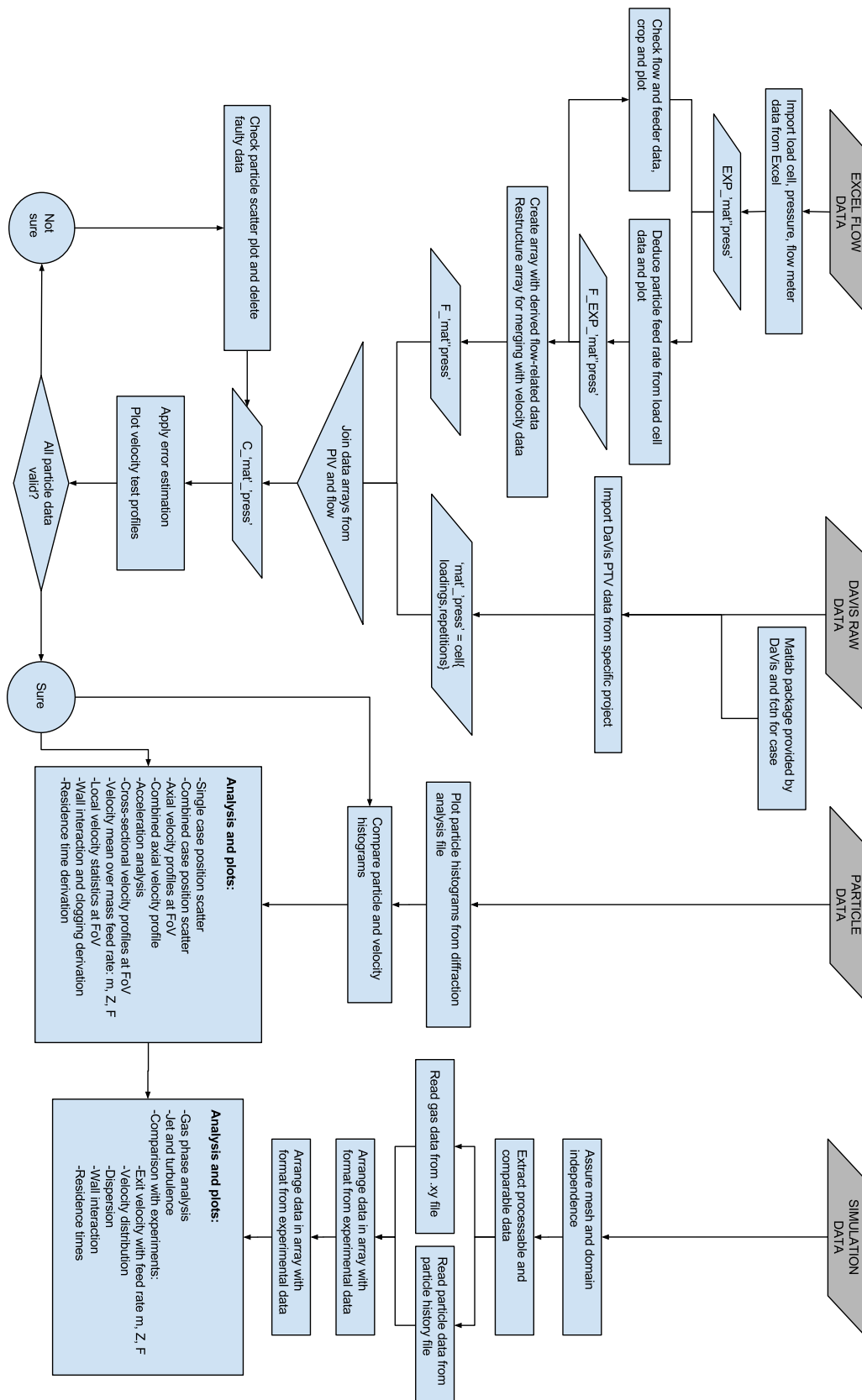
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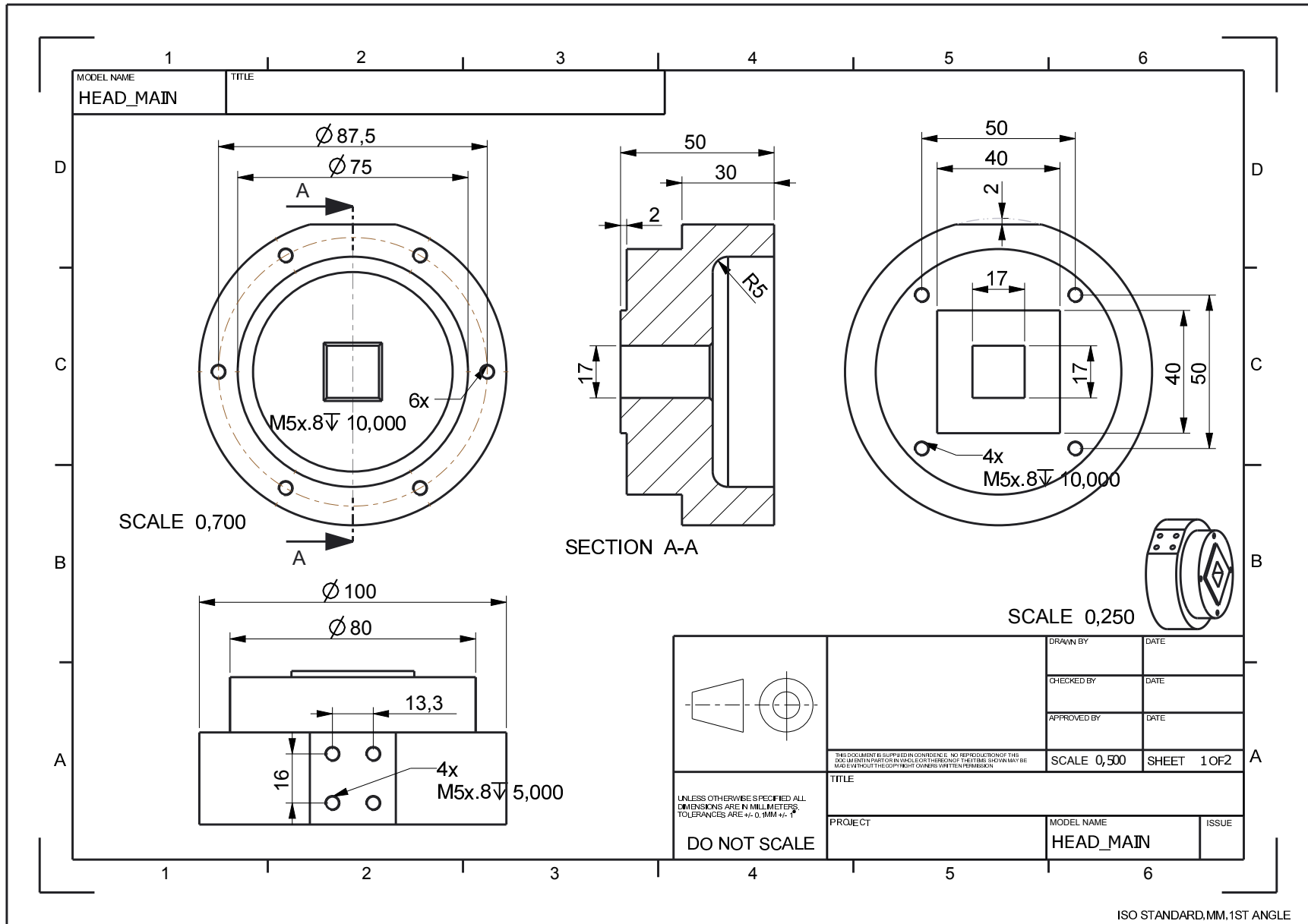
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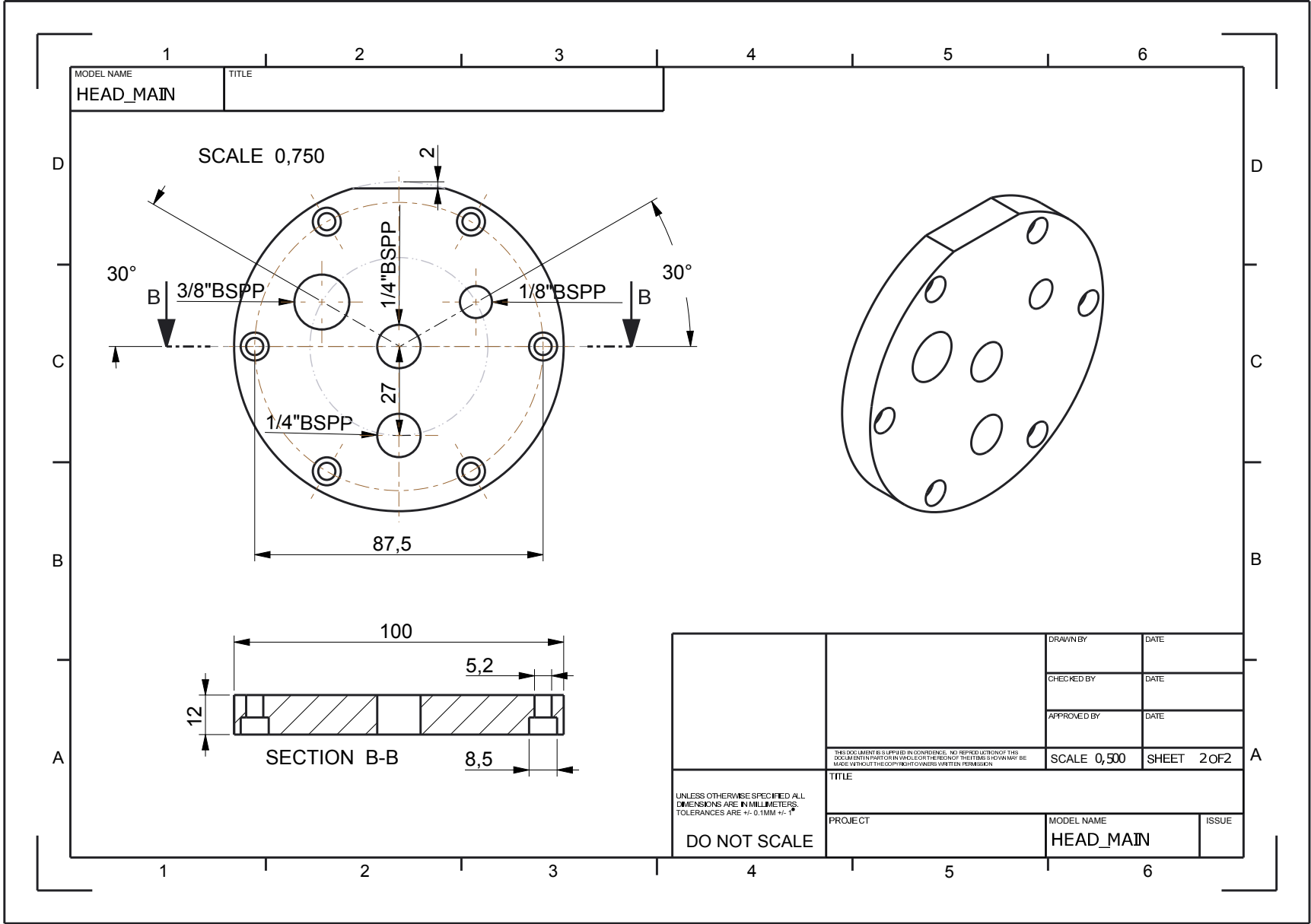
Appendix

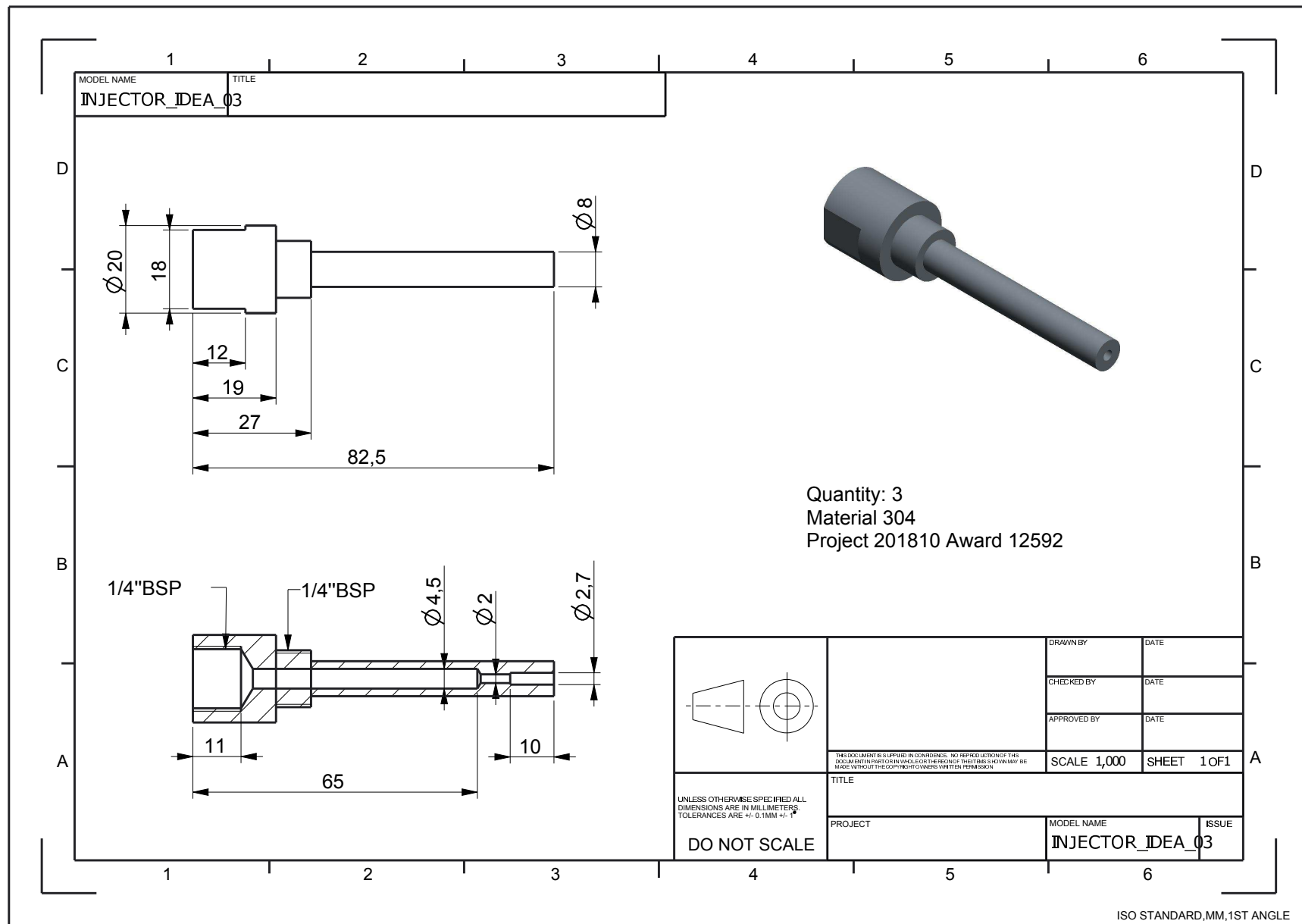
A1 - Supporting material

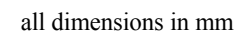
Data analysis workflow





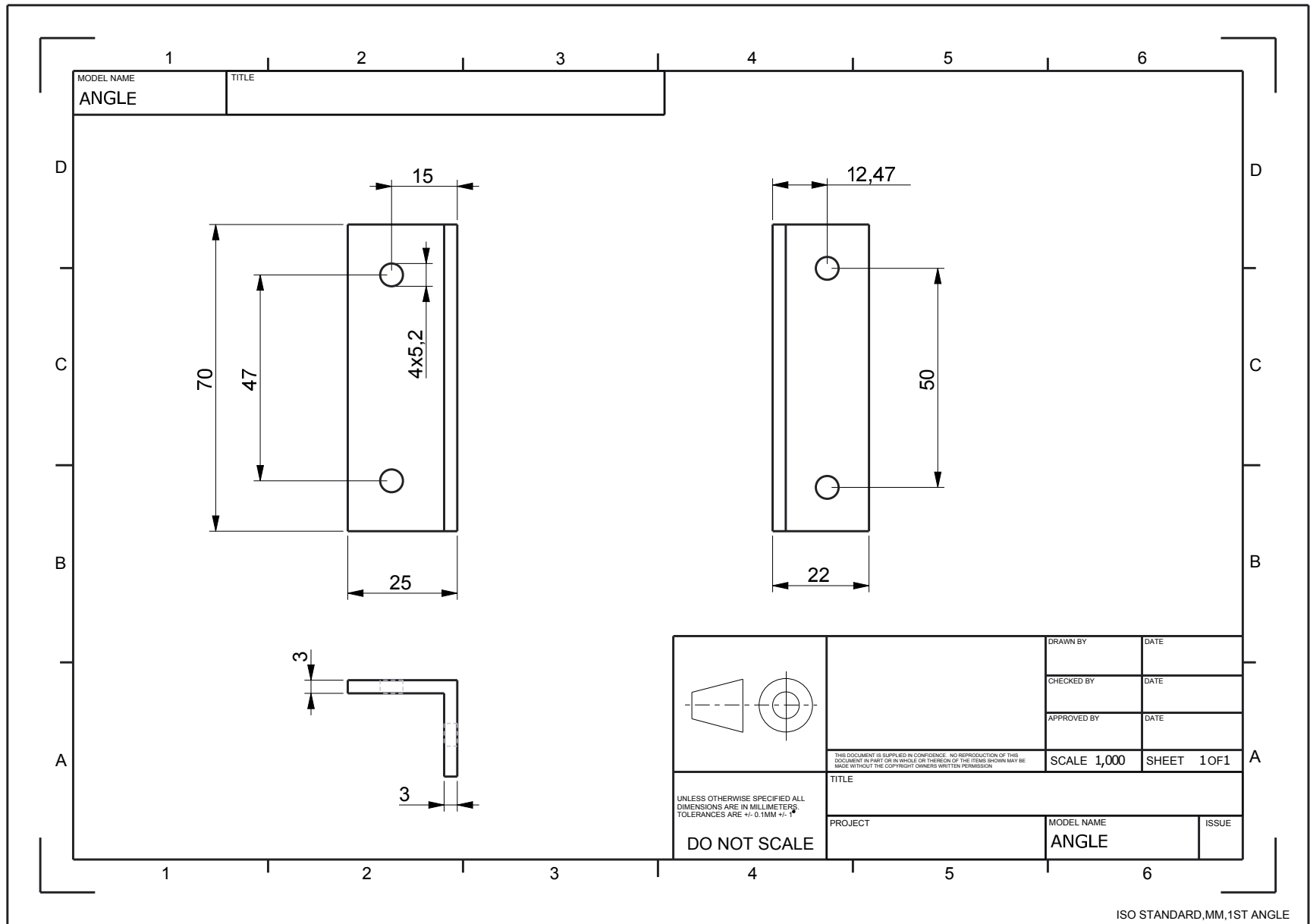


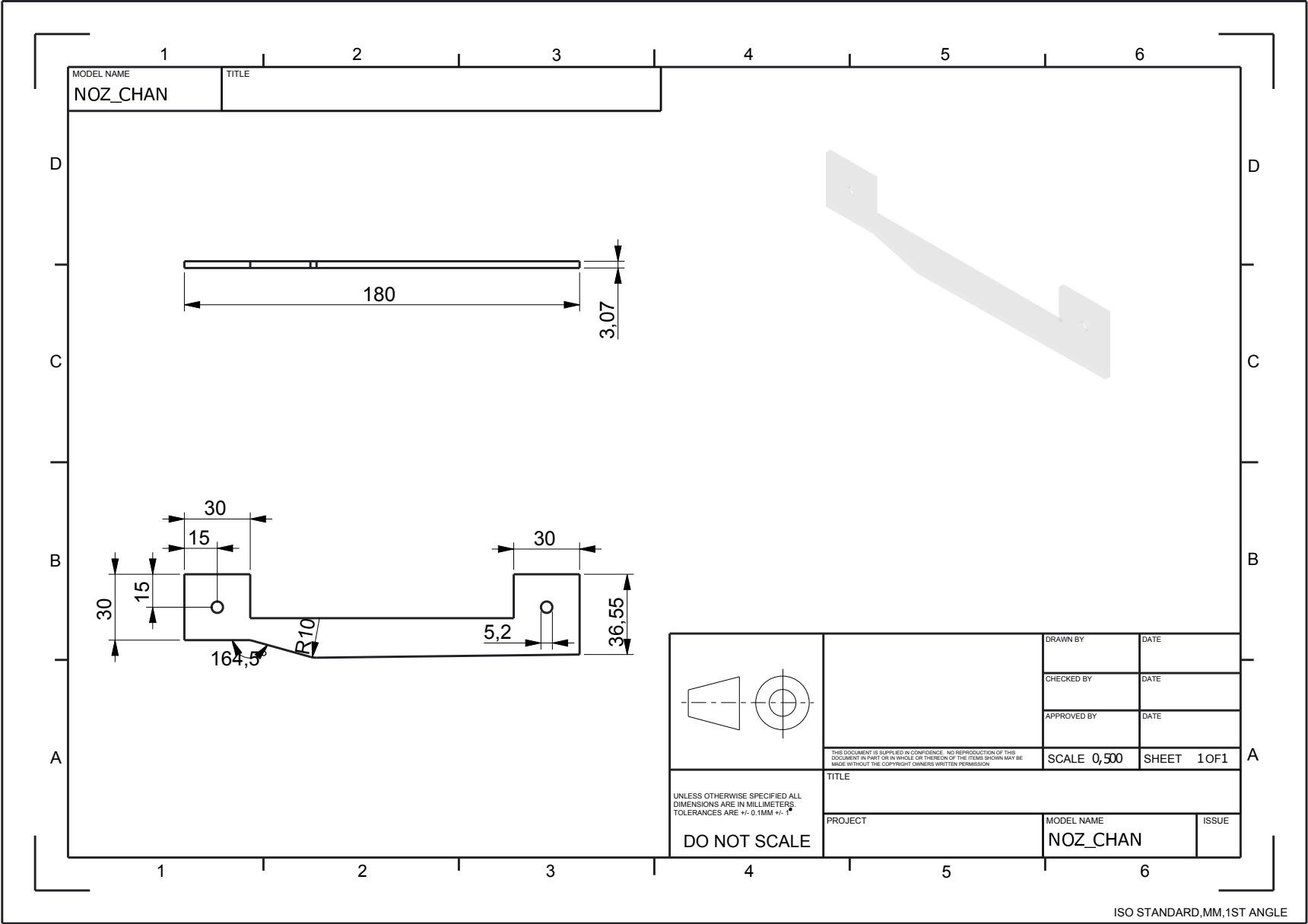


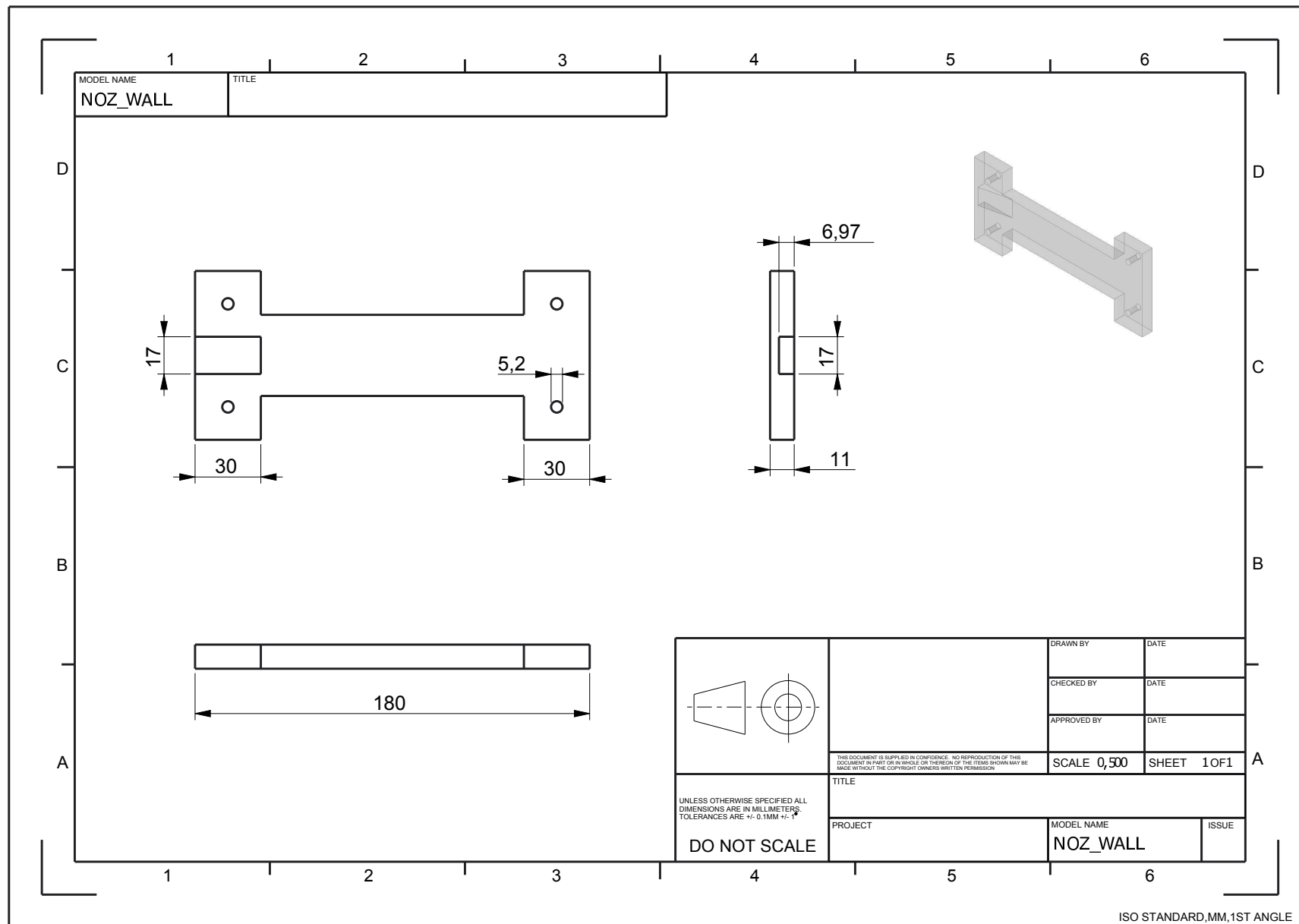


Dr. Rocco Lupoi

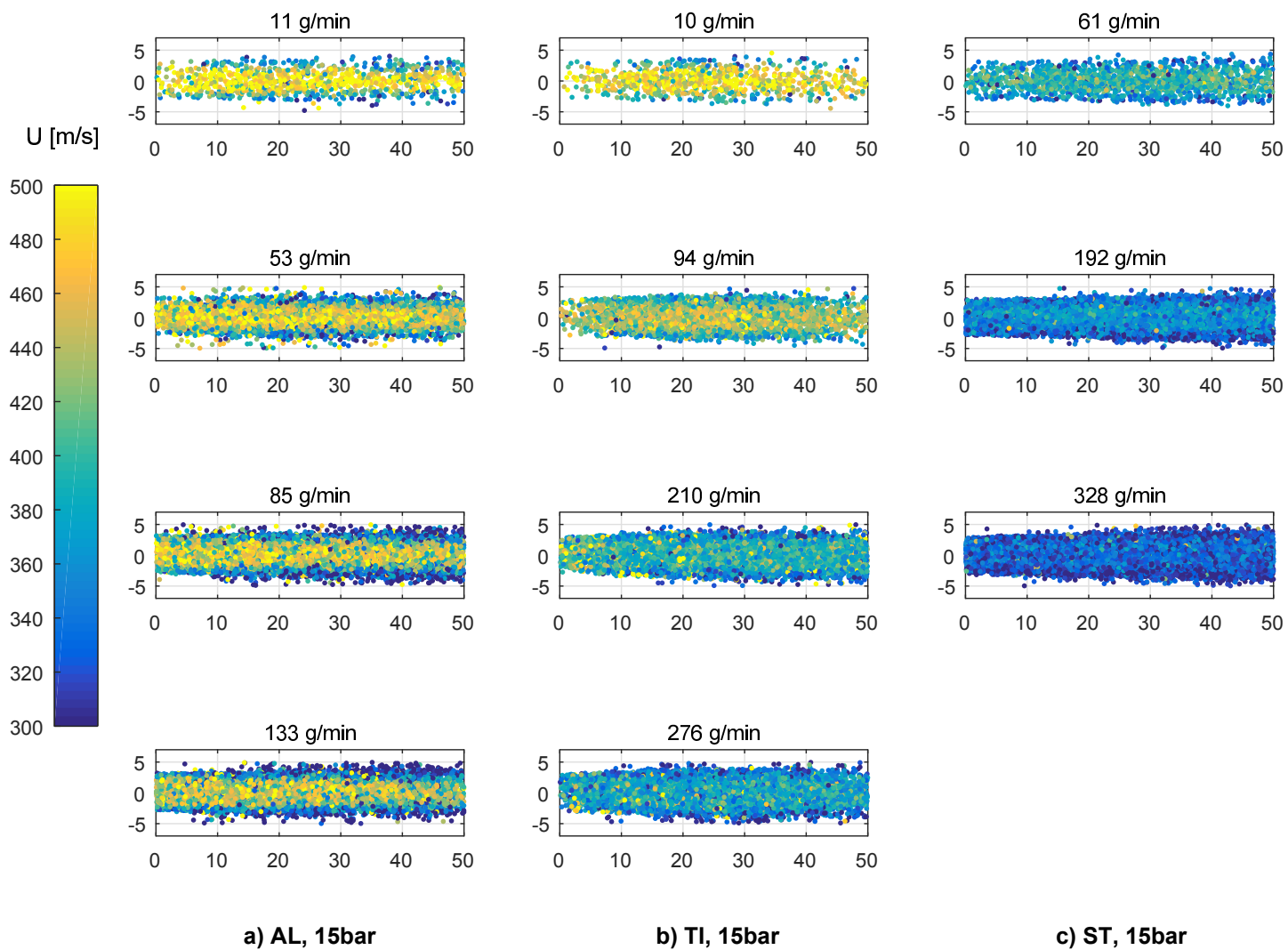
General Tolerance ±0.1mm	General Surface Finish minimum 0.8 Ra	Not to scale
Title: NZ-1	Material: WC-15Co	



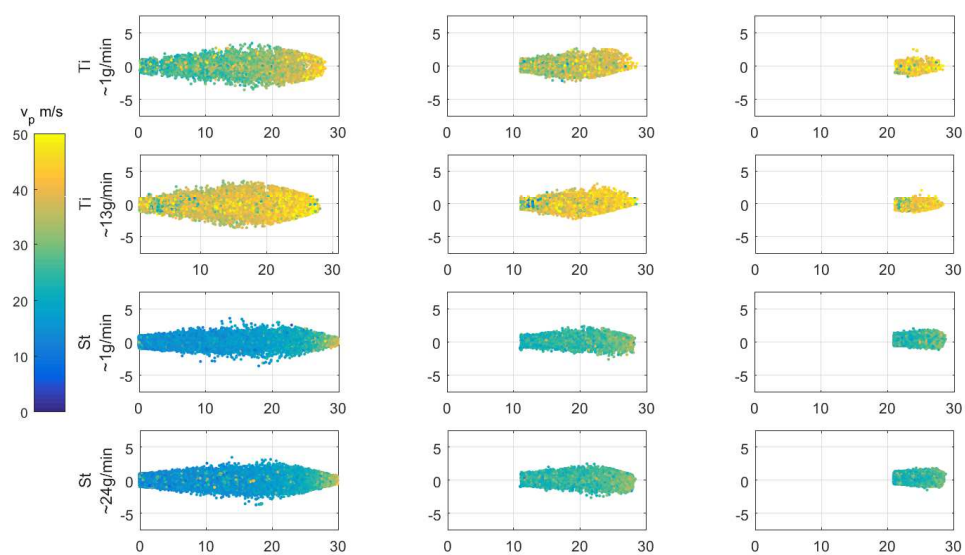




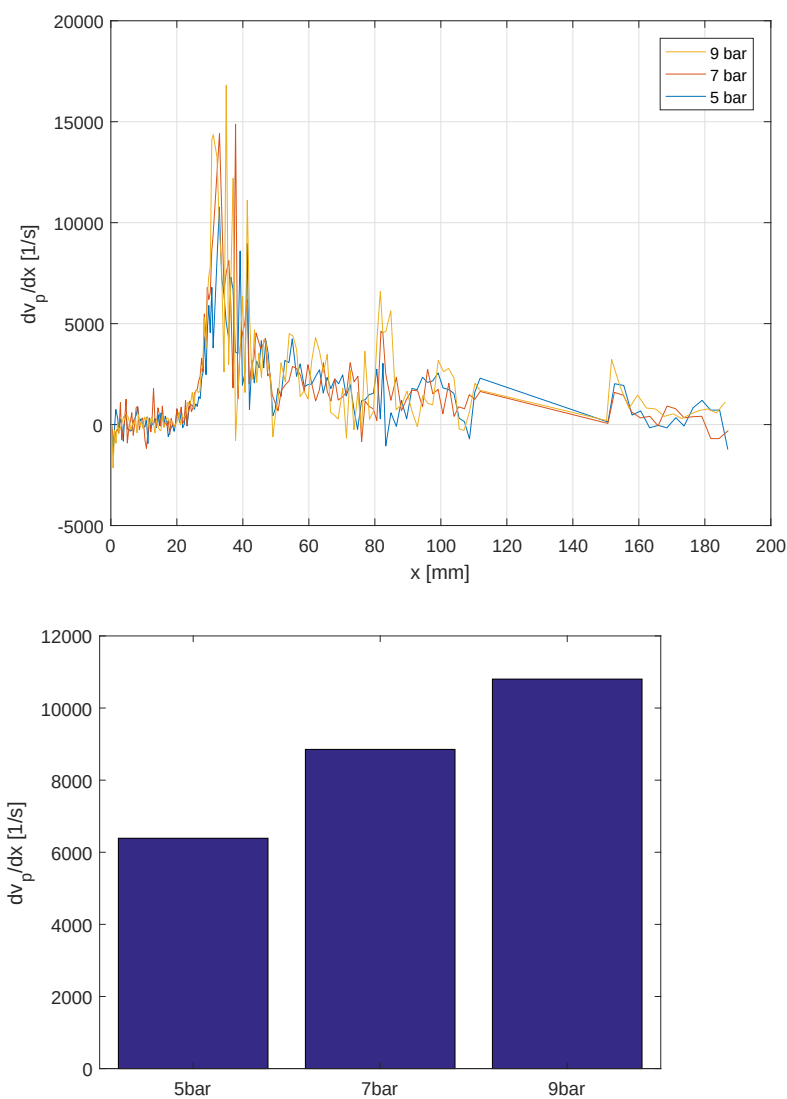
Particle velocity comparison in jet of Al, Ti, St at 15bar with NZ1



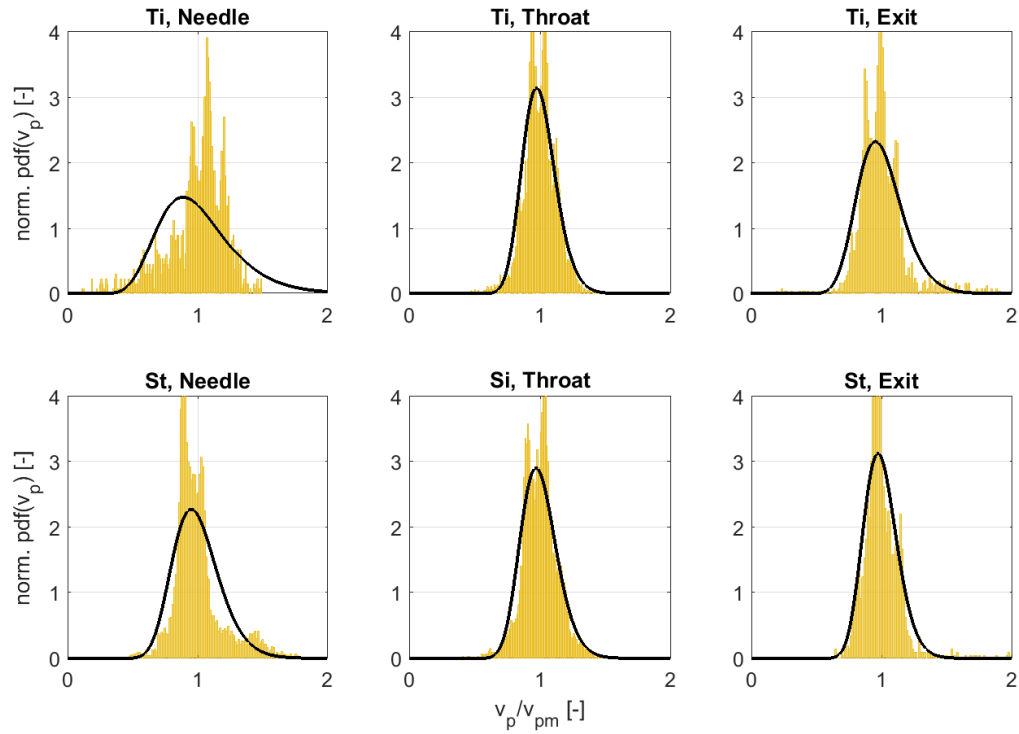
Feed rate comparison of injection plume of Stellite and Titanium



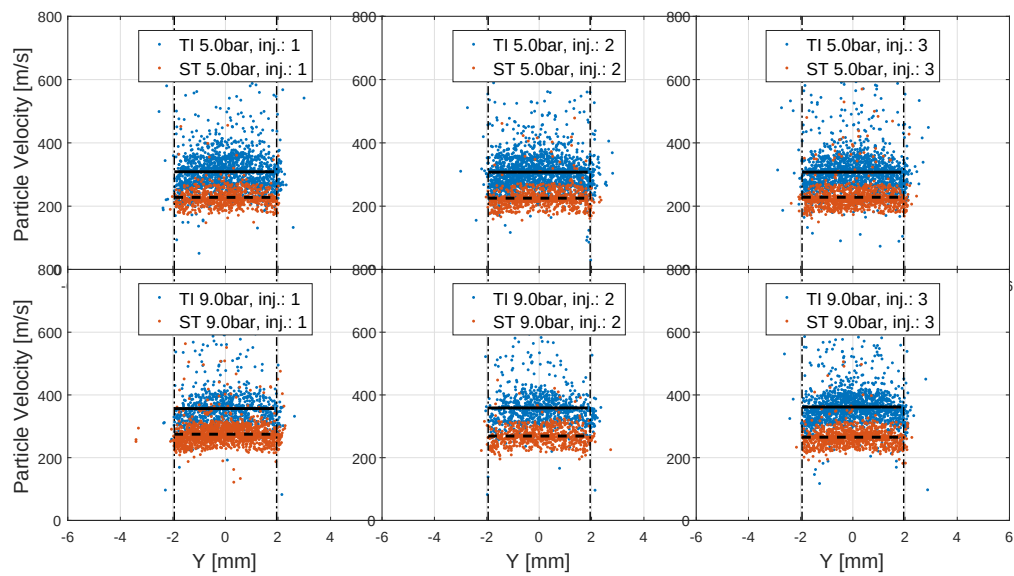
Substantial derivative of particle velocity data along the nozzle and peak value comparison in throat



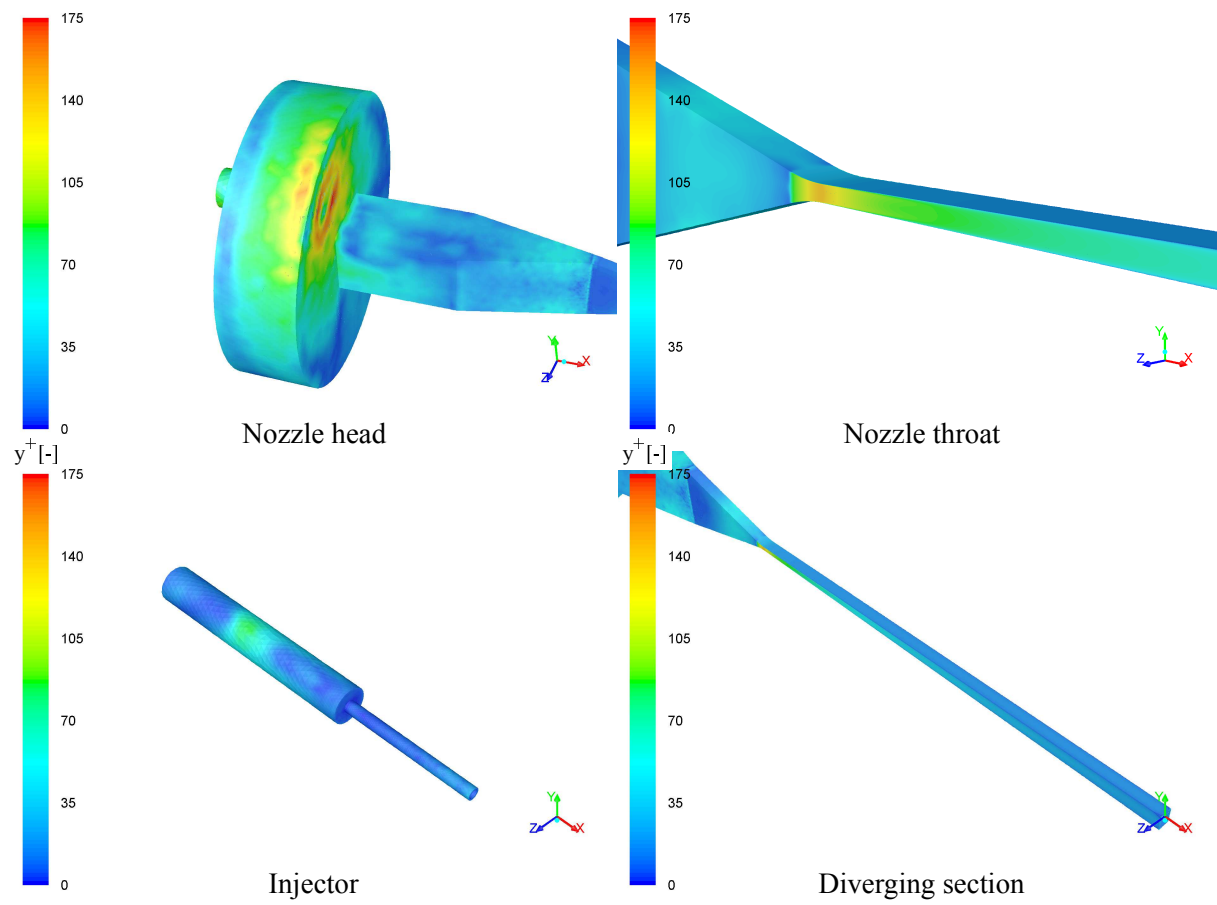
Comparison of pdf against log-normal profile: Strong differing in throat and needle, Titanium stronger than Stellite



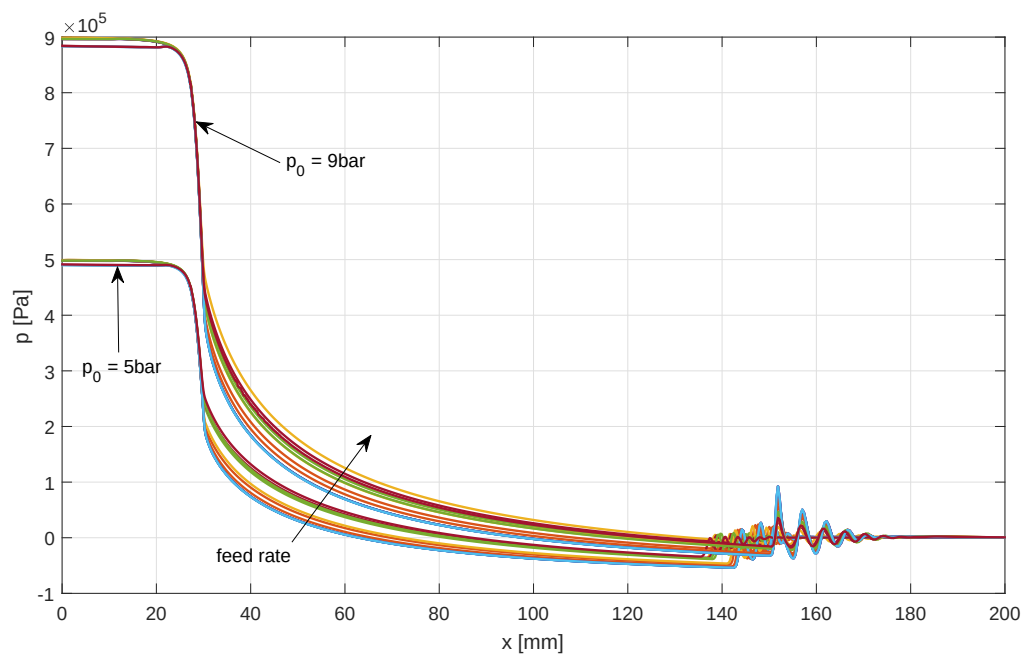
Particle exit velocity comparison: Flat profiles and strong dispersion



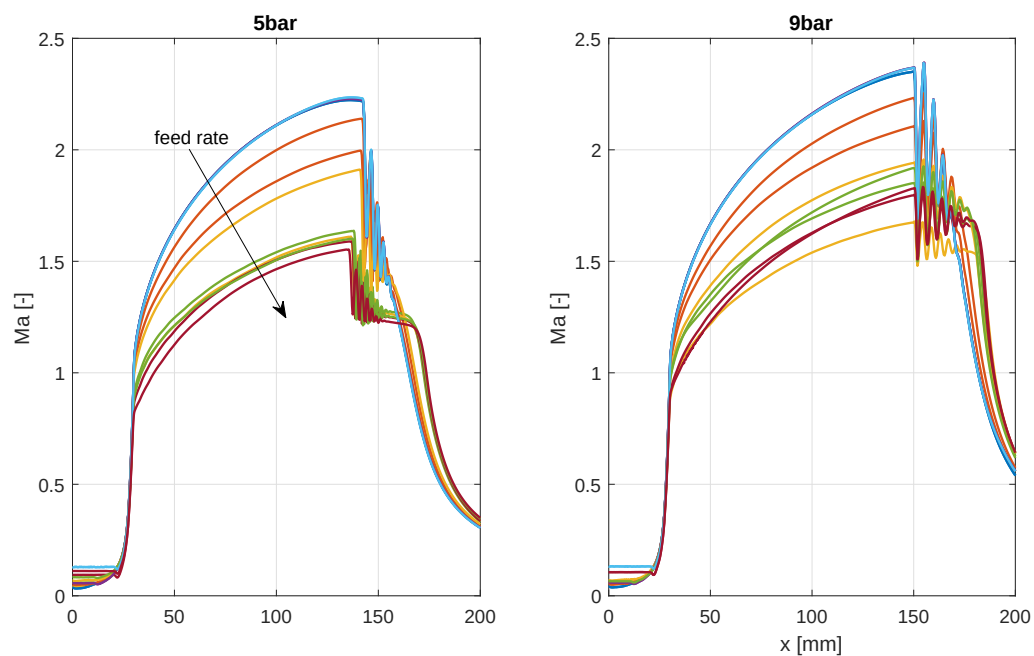
Mesh check: y^+ at nozzle walls



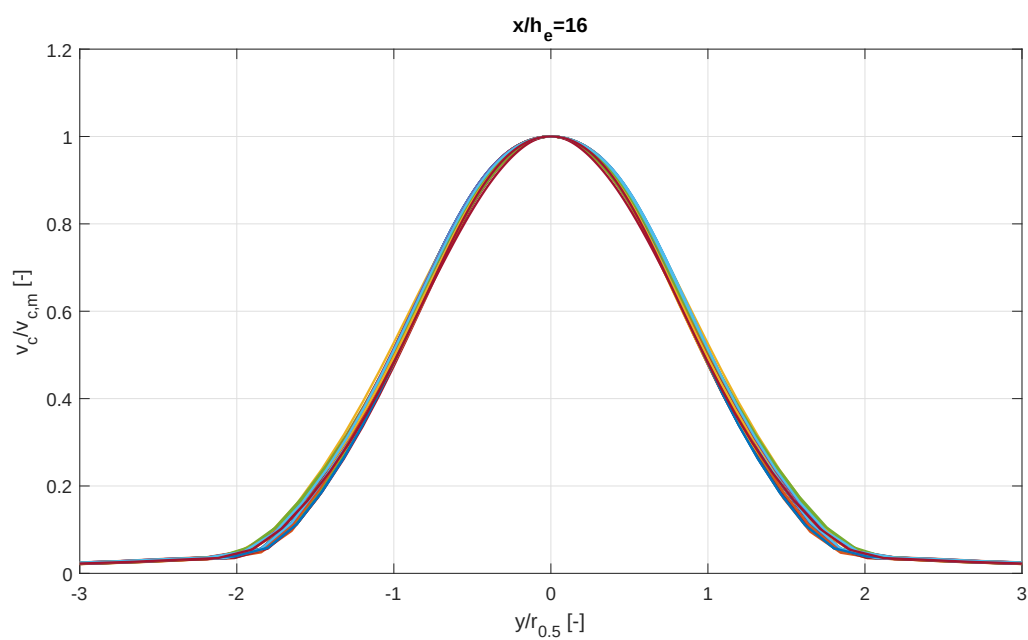
Axial gas pressure distribution



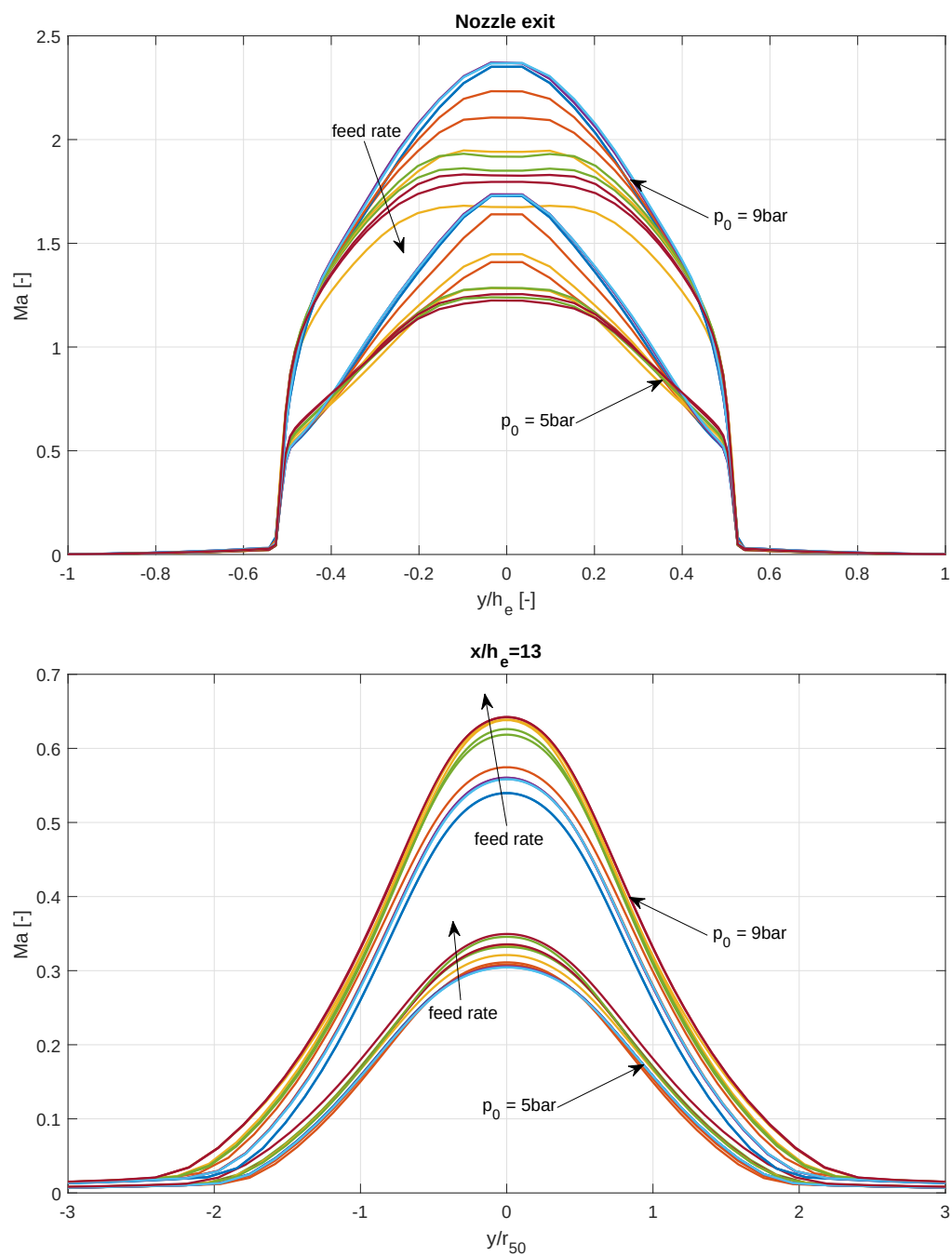
Axial gas Mach number distribution

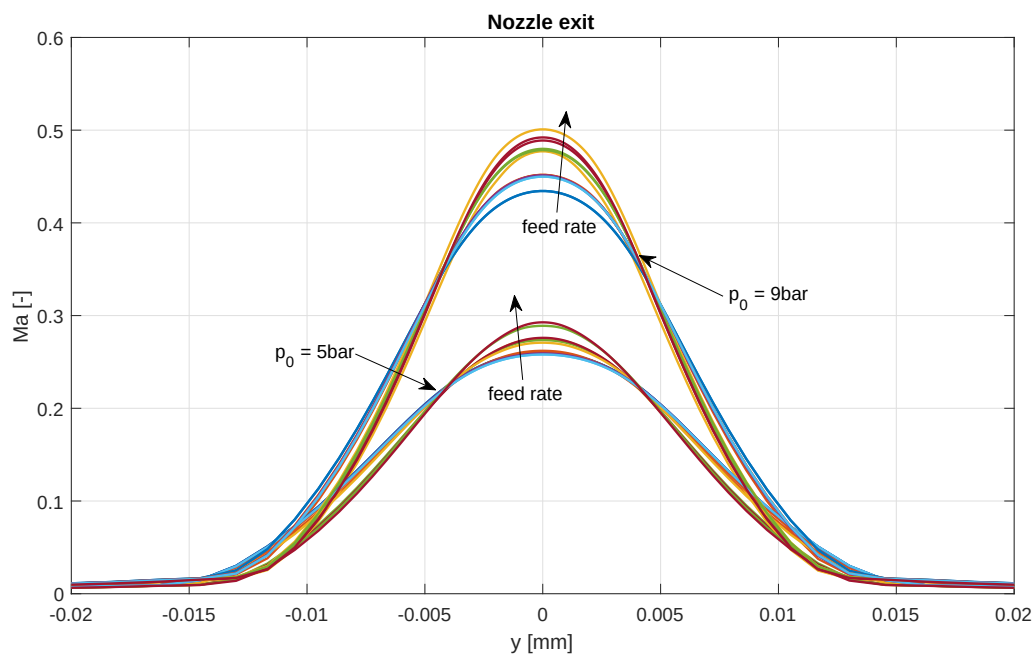


Cross-sectional normalised velocity profile in self-similar jet

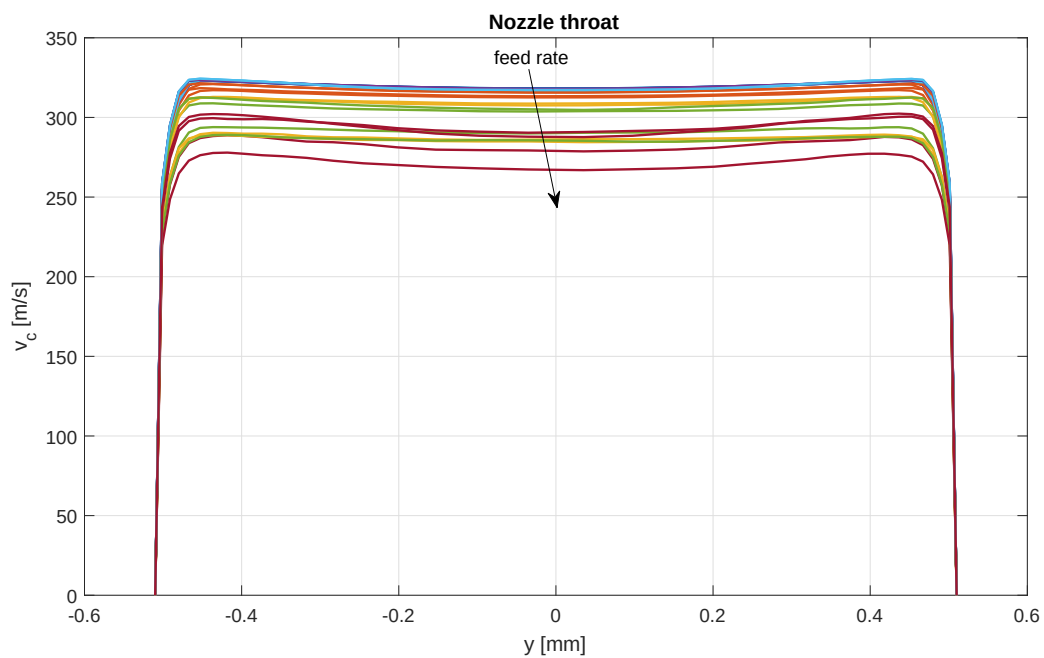


Cross-sectional Mach number distribution

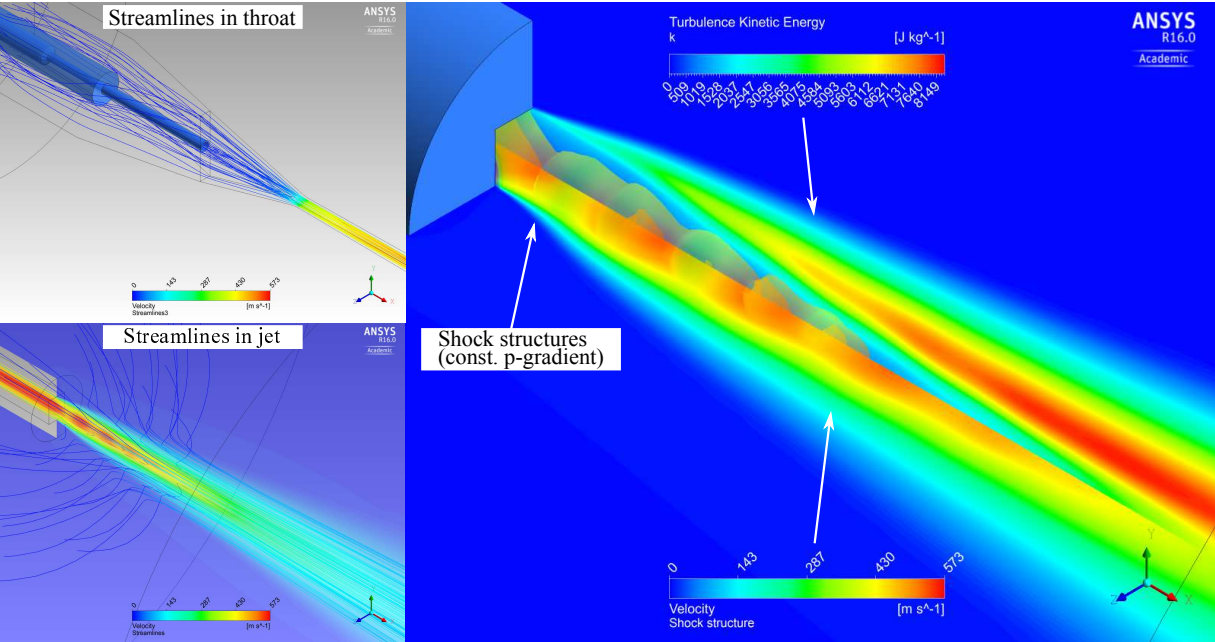




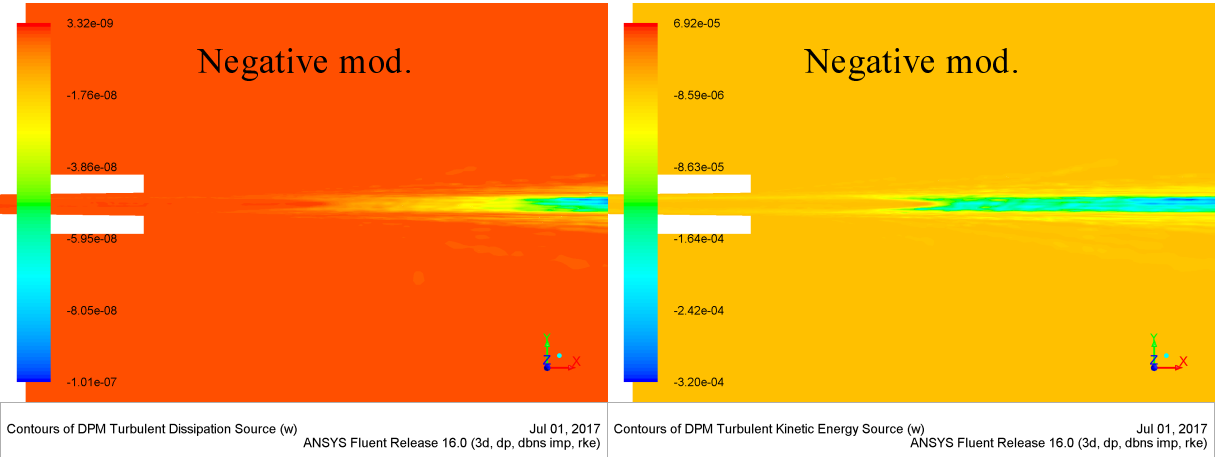
Cross-sectional gas velocity profiles in the throat



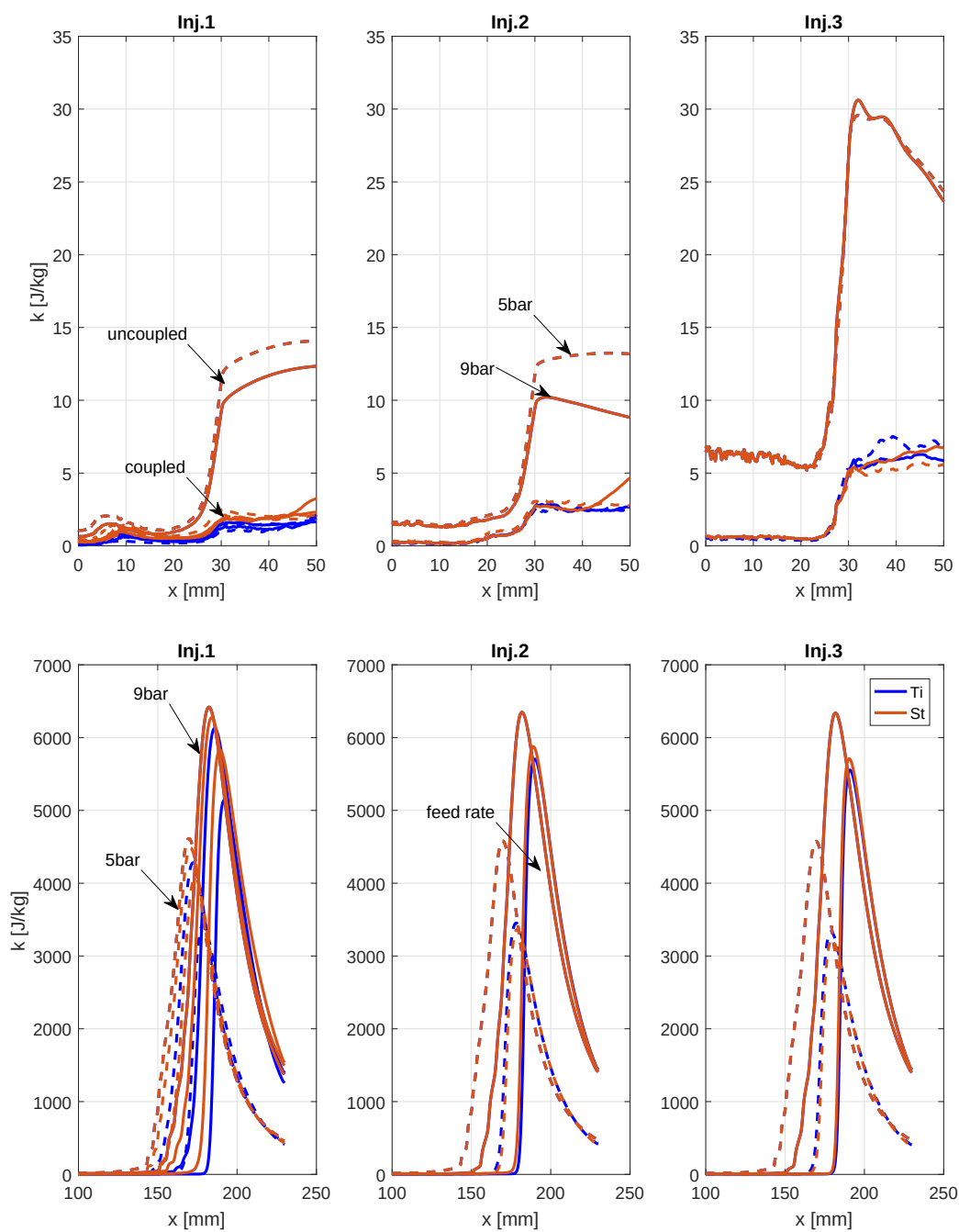
Jet shock structures, velocity and turbulent kinetic energy



Negative source term of turbulent modulation in the jet



Turbulent modulation in throat an jet



User-defined function for particle release boundary condition

```

/*****
  UDF that initializes particles on a surface injection with random
  y- and z-velocity components in range +/- (rng/10) m/s (see comment),
  and magnitude mag m/s
*****/

#include "udf.h"
#include <stdlib.h>

DEFINE_DPM_INJECTION_INIT(init_particles,I)
{
  Particle *p;
  int n, rng, rngx; /*y, rz; /* ry and rz are random number */
  double mean, rx, ry, rz;

  Message("Initializing Injection: %s\n",I->name);

  loop(p,I->p_init)
  {
    mean = 25; /* in m/s */
    rng = 50; /* must be int for rand, so rng/10 is in
              m/s, so factor 1e-1 needed rng = 5 means +-0.5m/s */
    rngx = 100;

    n = rand() % (rng*2+1); /* random range is still [-rng,rng], so
                           shift needed using modulo */
    ry = (n-rng)*1e-1; /* factor to make small ranges
                       possible */

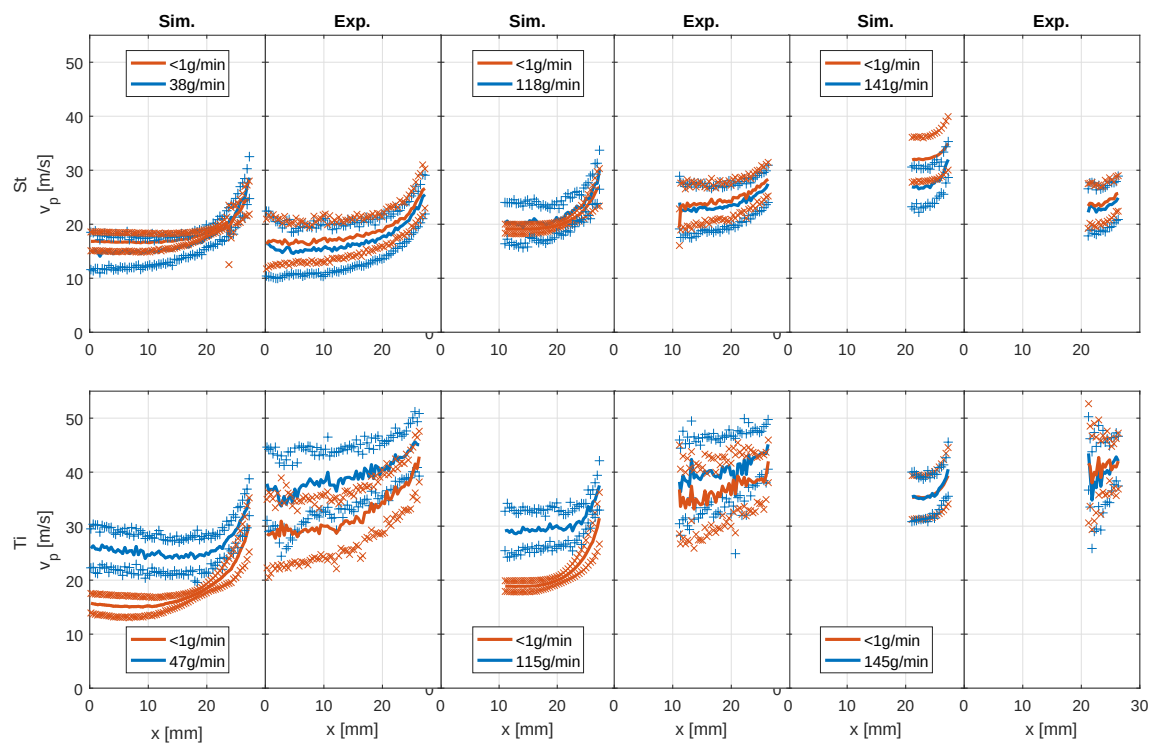
    n = rand() % (rng*2+1);
    rz = (n-rng)*1e-1;

    n = rand() % (rngx*2+1);
    rx = mean + (n-rngx)*1e-1;

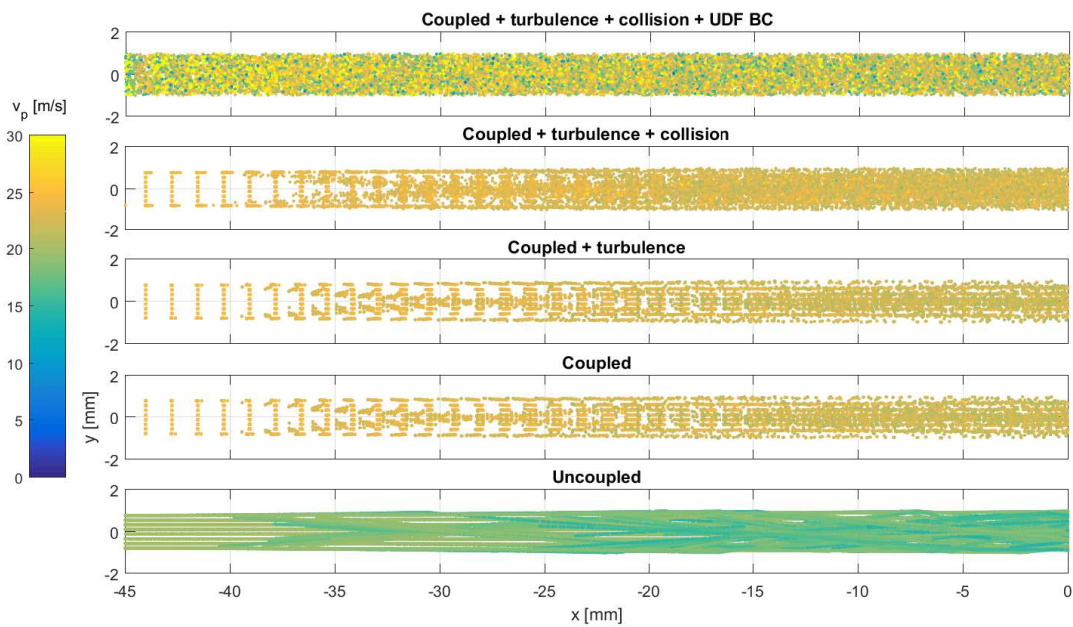
    P_VEL(p)[0] = rx;
  }
}
```

```
    P_VEL(p)[1] = ry;  
    P_VEL(p)[2] = rz;  
  }  
}
```

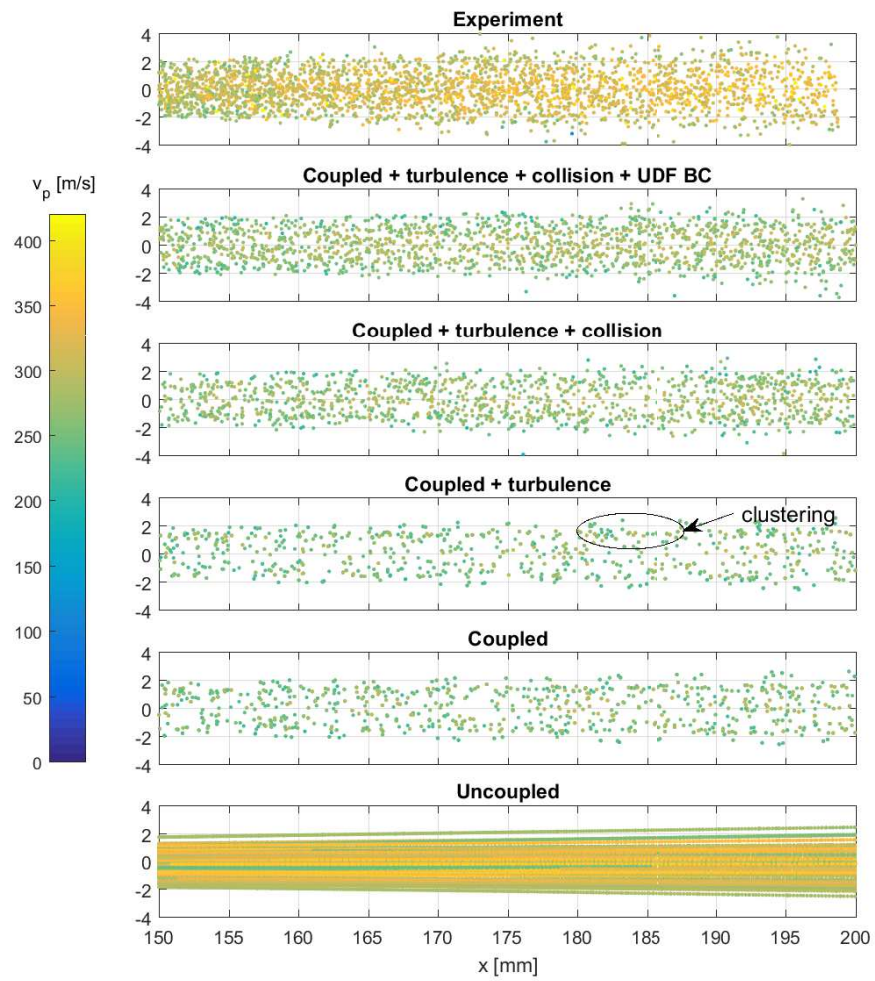
Validation of axial particle velocity distribution at injection location



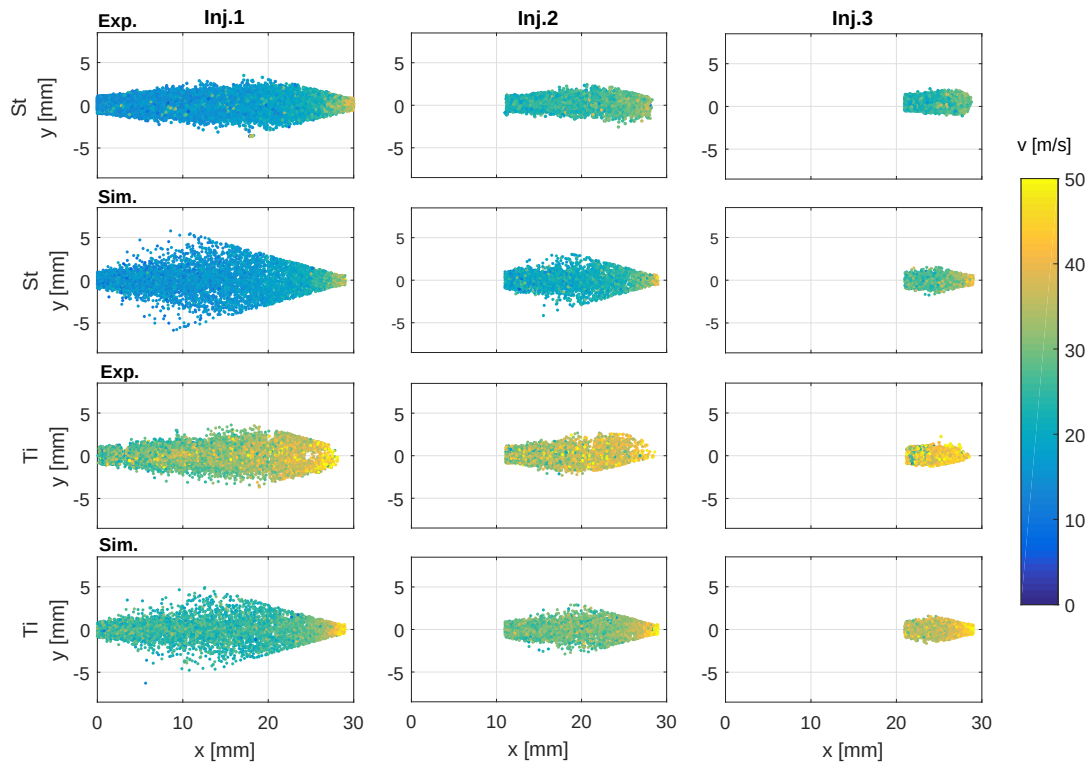
Needle-internal particle phase development: comparison of models



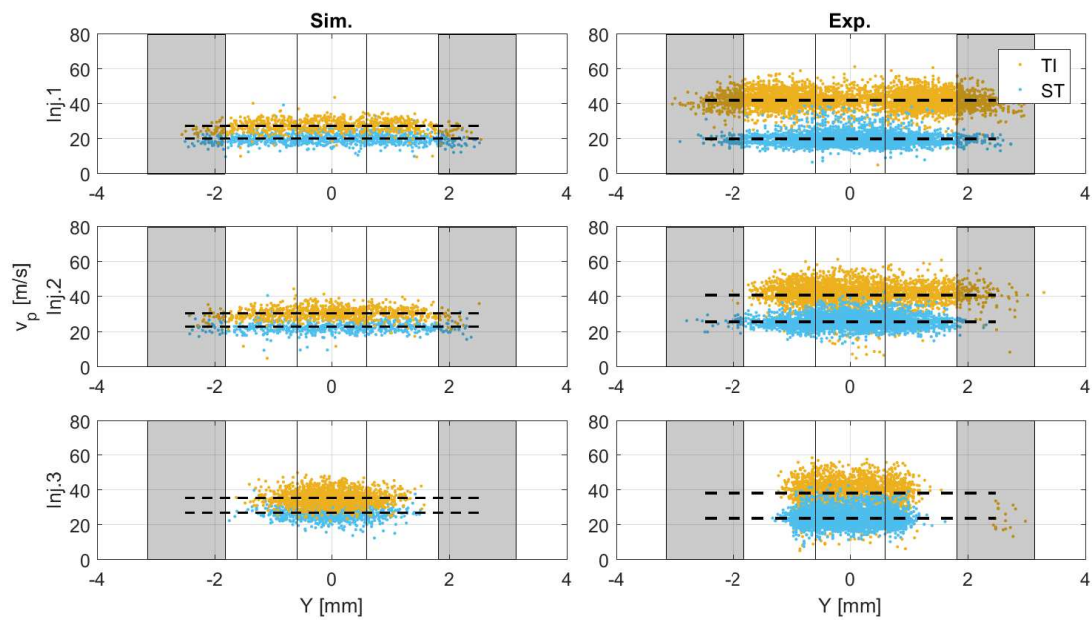
Jet particle phase development: comparison of models



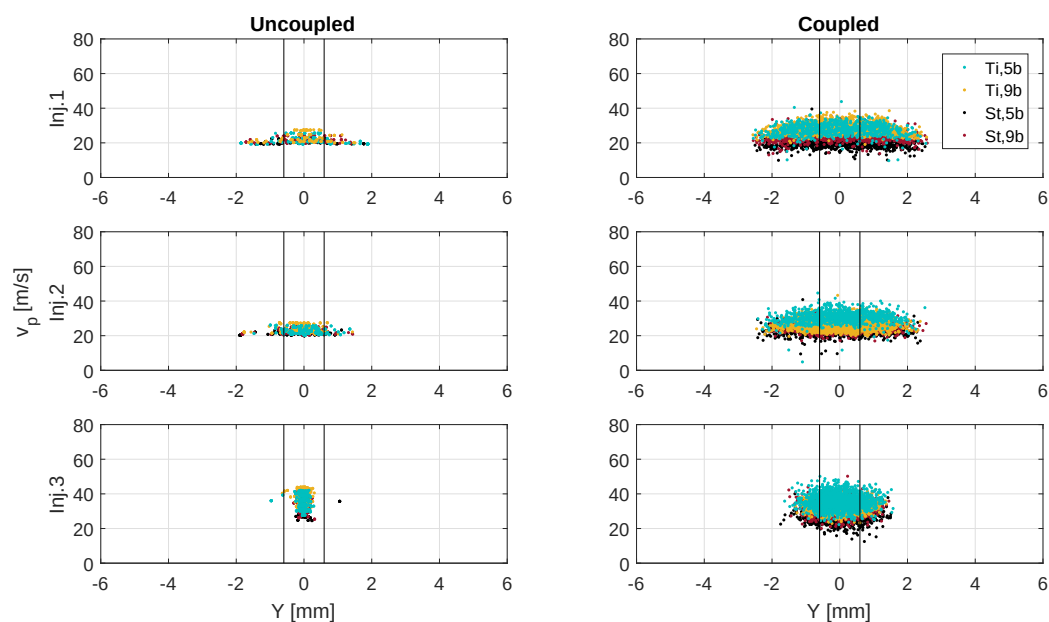
Validation of injections



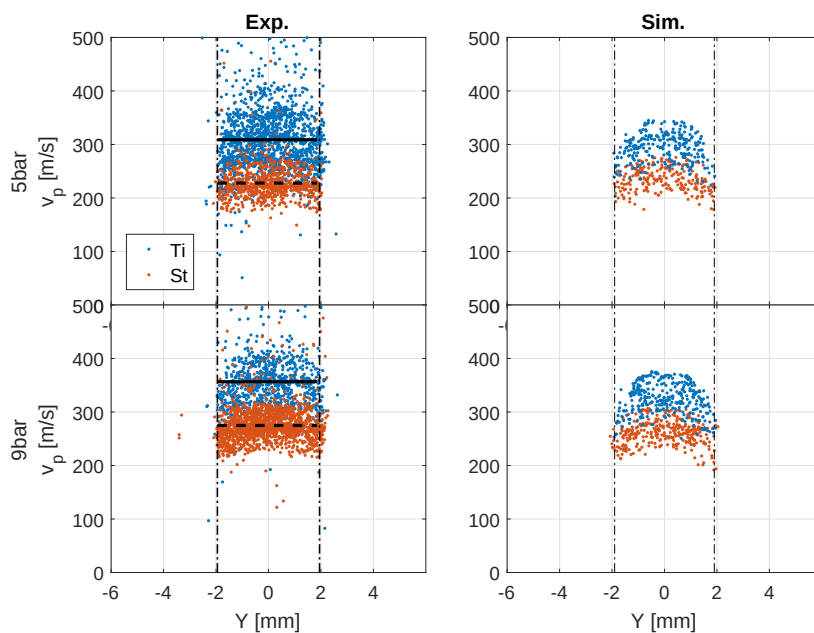
Validation of nozzle-internal cross-sectional particle velocity profile



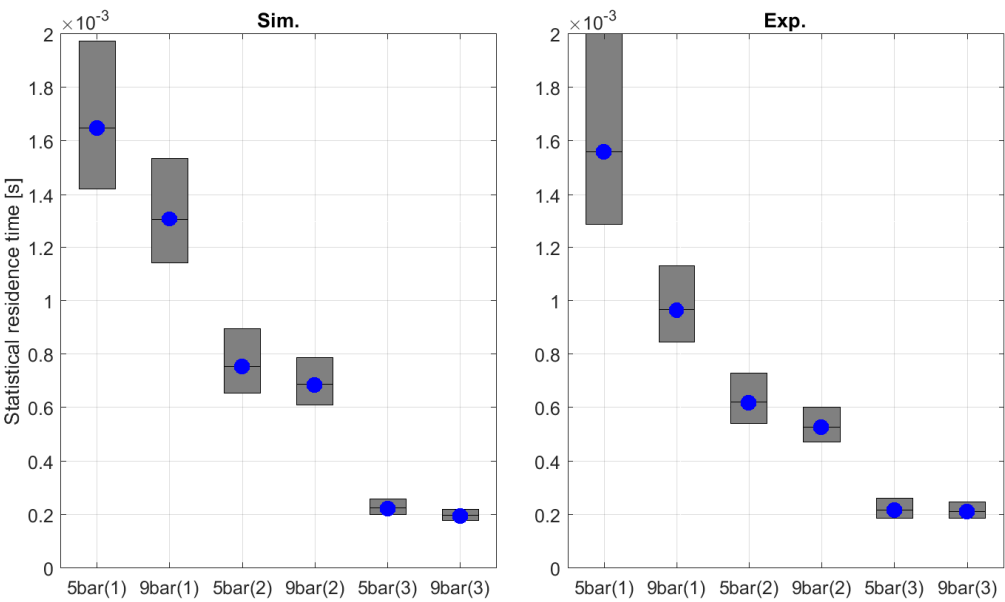
Comparison of models: dispersion at nozzle throat



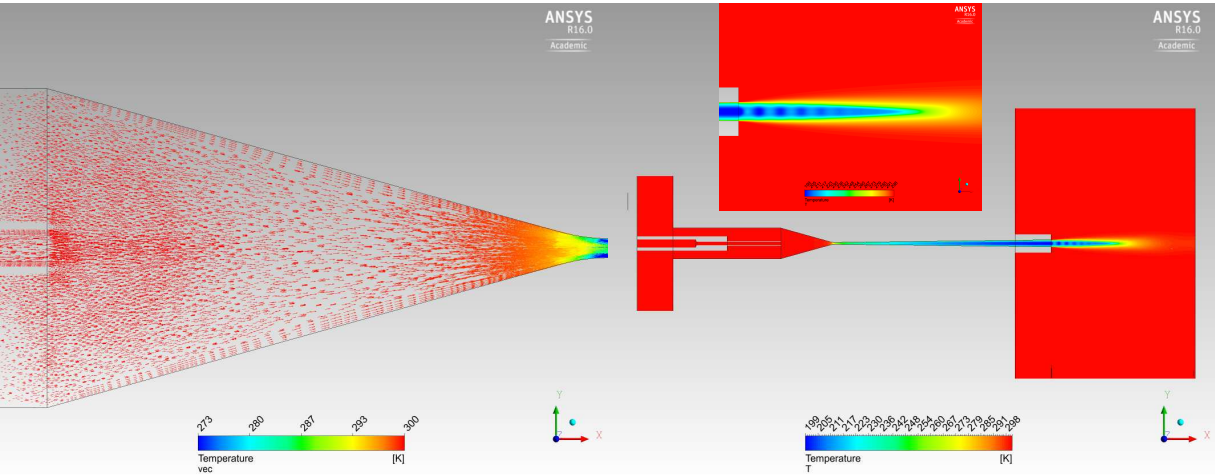
Validation of velocity and dispersion at nozzle exit



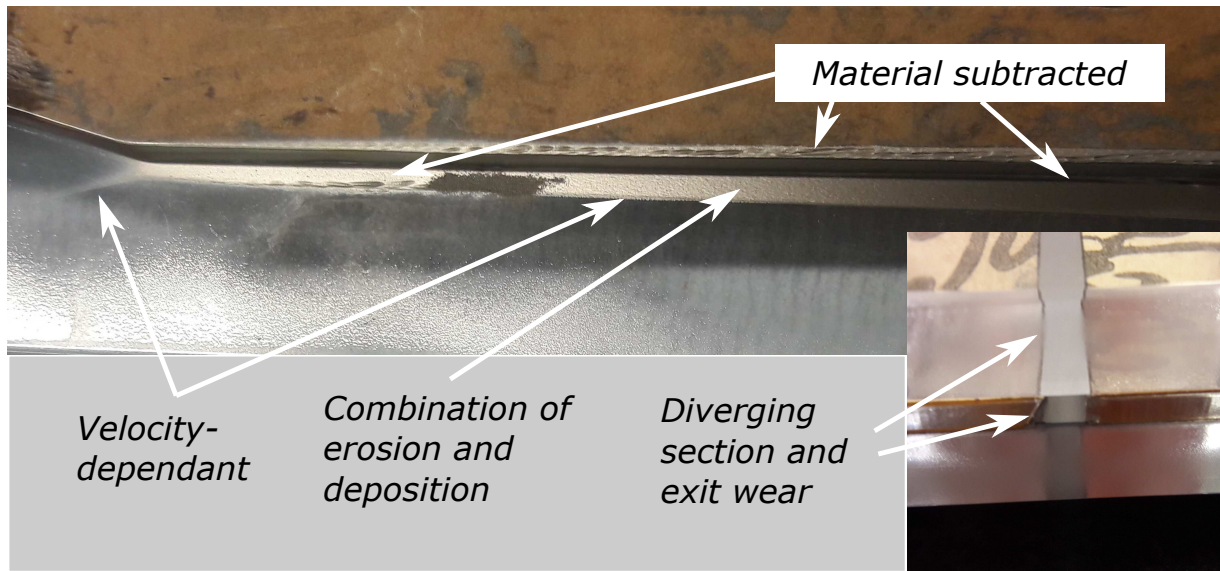
Validation of statistical particle residence times with changingn pressure, injectors and feed rate



Gas temperature distribution



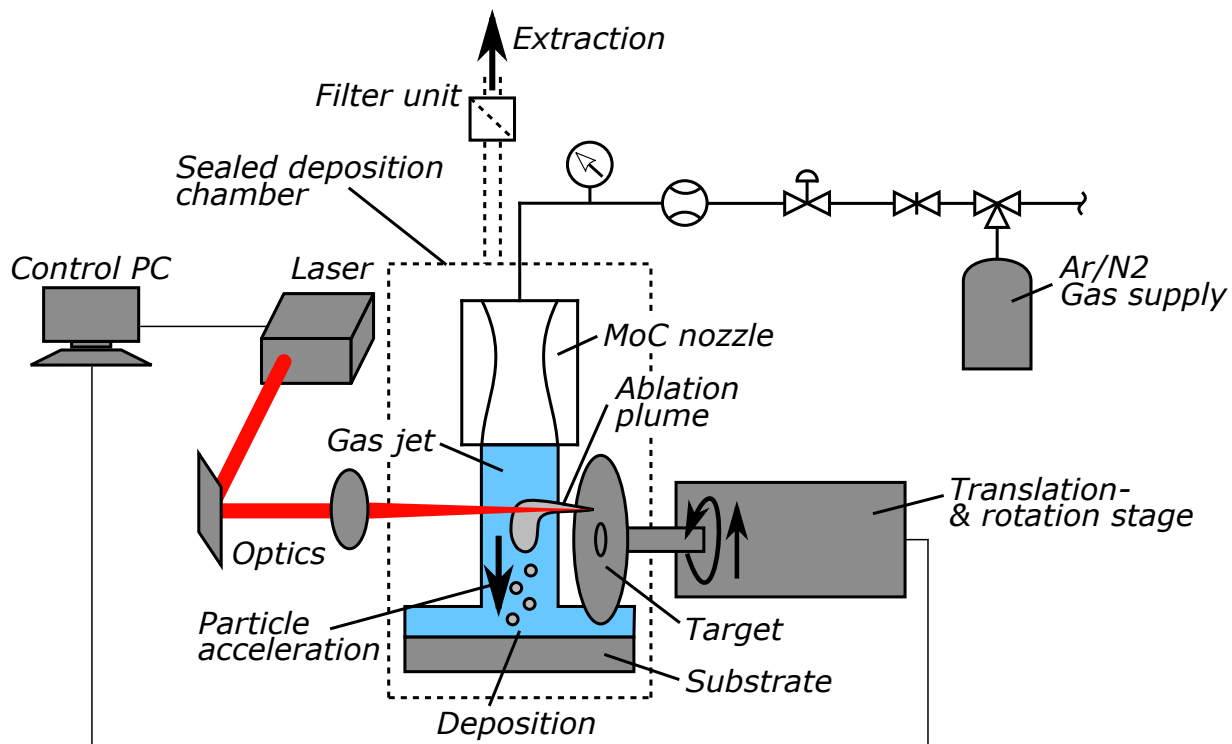
Nozzle erosion and particle dust smear deposition



A2 - Side-study: Minimum length nozzle for Pulsed Laser Deposition

A side-study was conducted within a cooperation project with Prof James Lunney and Dr Taj Khan from the School of Physics in Trinity College Dublin. Due to several similarities of fluid dynamic analysis and design methods, the goal of the side-project collaboration is the design and manufacture of a nozzle, adequate to provide a gas jet required for coating formation in Pulsed Laser Deposition (PLD).

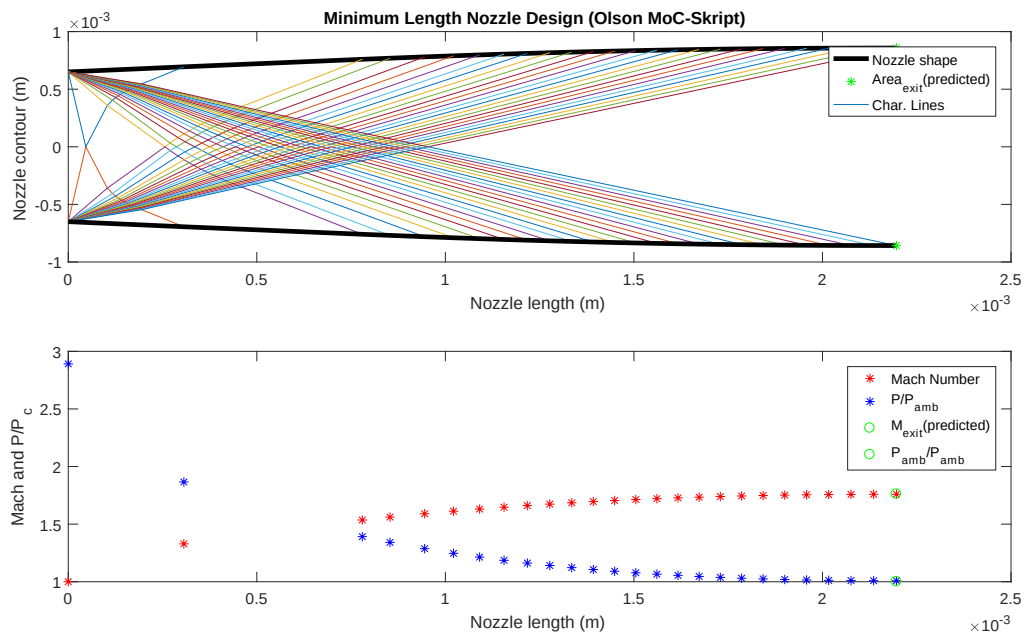
Pulsed Laser Deposition (PLD)



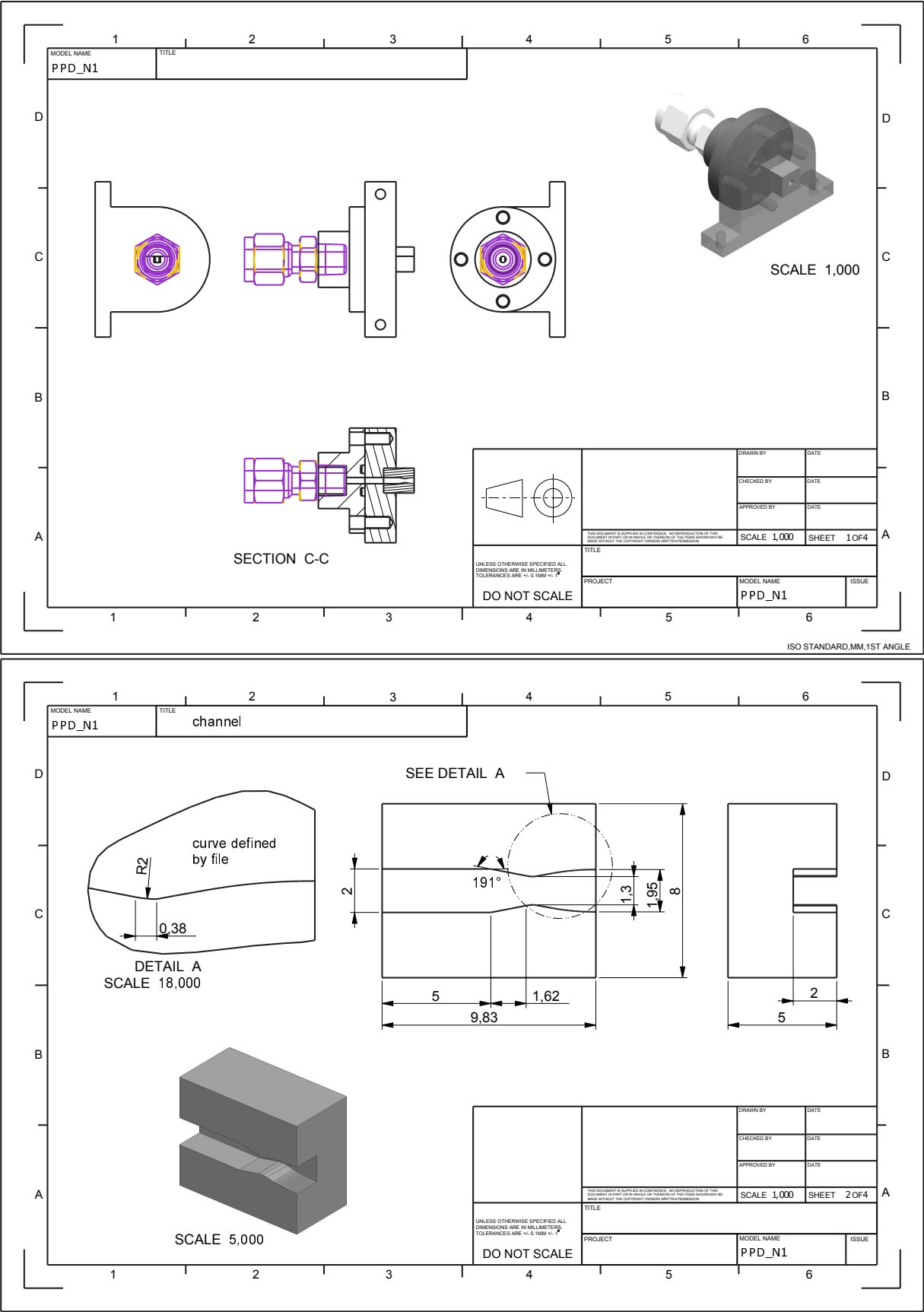
PLD is a related deposition technology, in which nano-sized particles are produced by evaporating material from a target by a high-energy laser pulse. In standard PLD, the particles are accelerated strongly during evaporation in an expanding plasmatic plume, because the environment is set under vacuum. The material is then deposited onto a substrate by impinging on it. In PLD, unlike CS, is a high-temperature technique for ultra-thin film deposition. One main problem is the operation in the vacuum chamber which is required for particle acceleration. In a new study, a merger of PLD and CS is attempted - a supersonic pulsed laser deposition. Instead of generating a pressure difference by vacuum, the acceleration of particles is realised by a supersonic jet. This way, it is possible to operate at ambient pressure and increase handling time and reduce cost. Another advantage is the possibility to redirect the particle plume and therefore make use of space more efficiently.

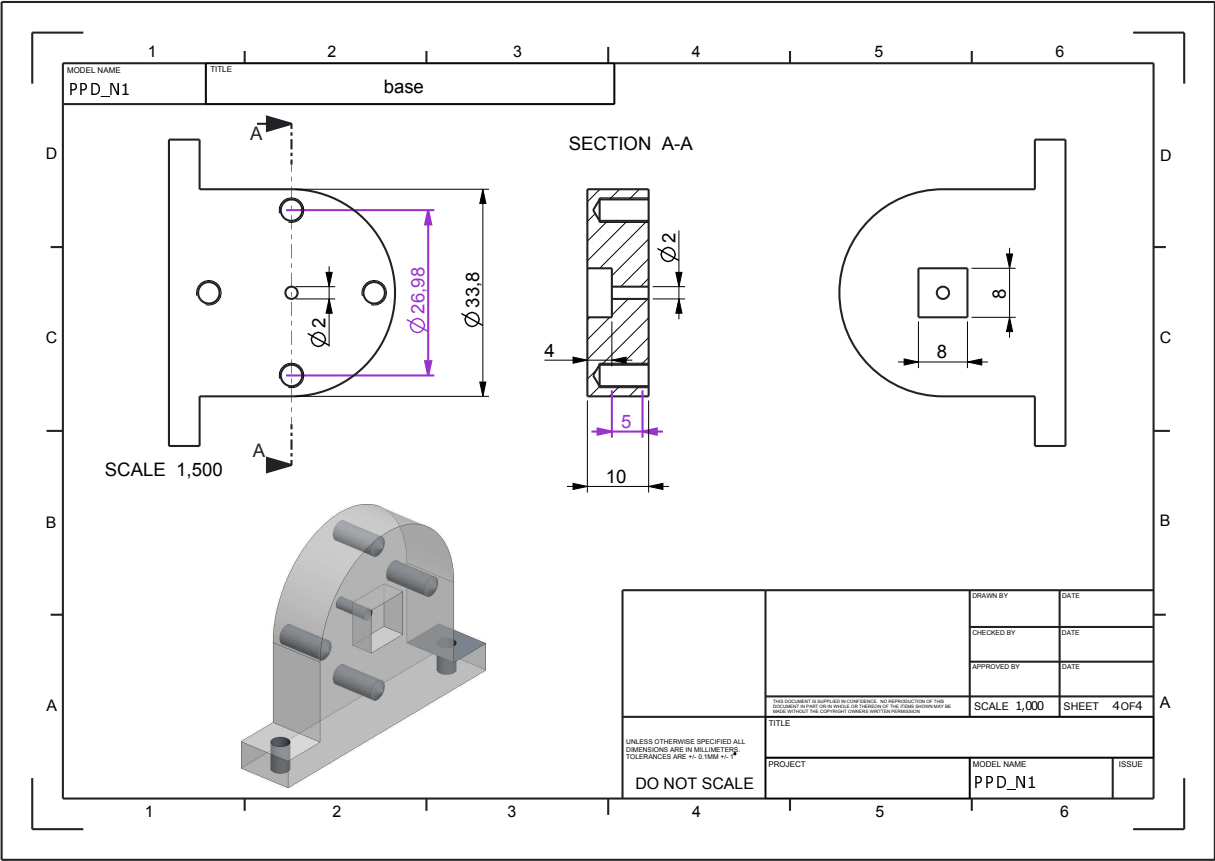
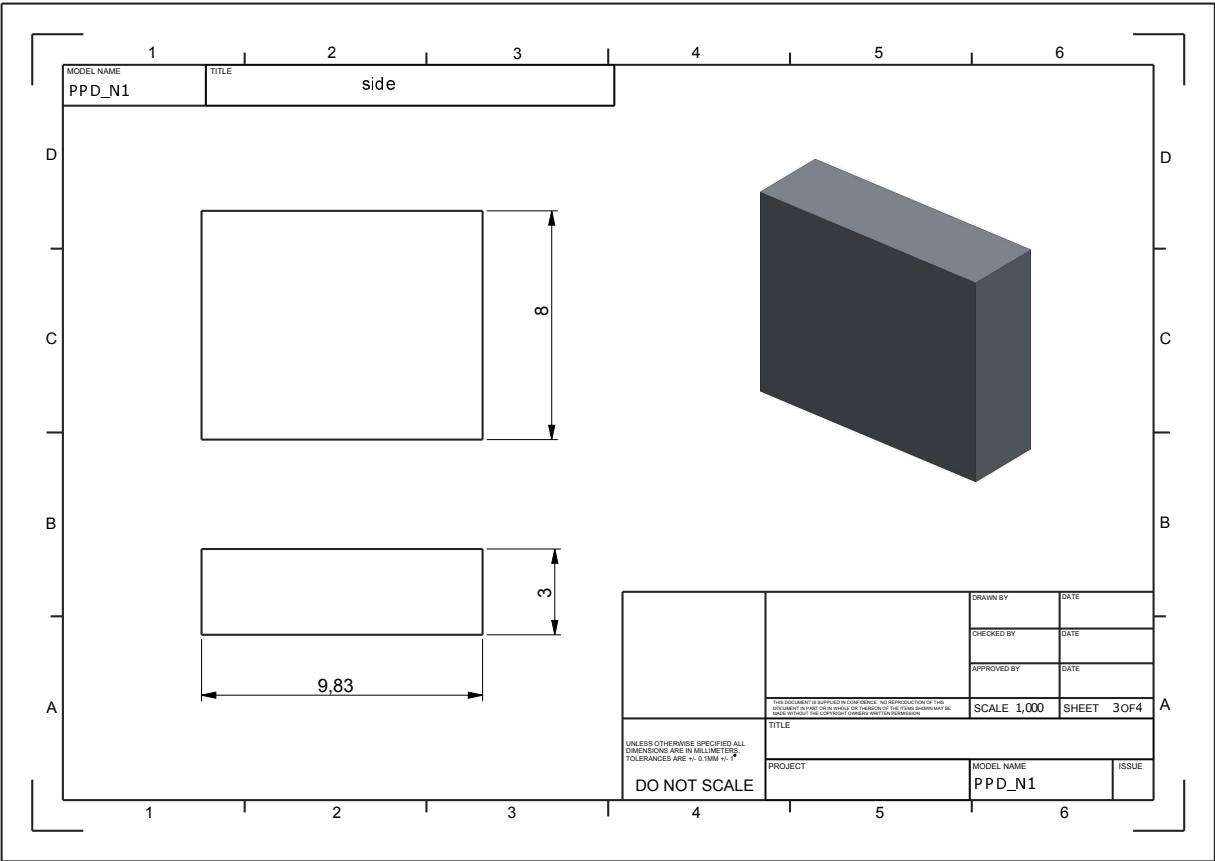
The gas jet is provided by a nozzle, and should be well-adapted and shock-free, since the plume is required to be captured by a homogeneous gas phase. The nozzle length is not a critical parameter, such that a nozzle with uniform outflow can be designed. The method of characteristics was used to calculate the contour of a minimum-length nozzle with such outflow. A quasi-2D nozzle was chosen as a solution due to simple manufacturing. It was sized according to the problem and specified for ambient pressure and a gas velocity of 400m/s with Argon gas. The following figures show the result for a RANS calculation to adjust and verify the flow field of the pre-designed contour. The nozzle was manufactured according to the subsequent drawings and is currently used for testing. First results of this study are expected to be reported shortly in a reviewed journal.

Method of Characteristics for PLD-nozzle



PLD-nozzle drawings





Velocity and temperature distribution of PLD-nozzle under operation

