

# Manufacture of Reflective Aluminium Surfaces Using Cold Spray

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## Abstract

This research demonstrates the use of cold spray (CS) as an additive manufacturing process to manufacture reflective aluminium coatings. Nitrogen was used as a carrier gas at various gas heating temperatures. Following deposition, the coatings were finished using a number of machining and/or polishing processes to surface roughness values of 20-150 nm. The samples were characterised with respect to total reflectivity within the wavelength range of 400-1800 nm, porosity, surface roughness, and density. The reflectivity of the coatings approached that of bulk material, and 99% dense coatings were obtained. Increasing the gas heating temperature did not decrease the porosity with the lowest gas heating temperature found to deliver the best reflectivity. This work demonstrates that CS can be used to coat thin layers of aluminium onto various materials, which can be subsequently polished to create composite reflectors. This provides a novel reflector with the reflectivity of aluminium, and the structural and thermal properties of the substrate material, allowing for greater flexibility in the manufacture of reflectors.

## Introduction

CS is a surface coating technique where small particles, typically metals, in the range of 10-50  $\mu\text{m}$  are accelerated to speeds of 300-1200 m/s using a De Laval nozzle before impacting upon a substrate [1,2]. A schematic of the cold spray process is shown in Figure 1. The kinetic energy of the particle upon impact causes substantial plastic deformation of both the particle and substrate allowing bonding to occur through mechanical interlocking and adiabatic shear instability [2,3]. CS allows coatings to be manufactured which show little oxide

inclusion, exhibit low porosity and are free from melting and hence significant adverse changes in microstructure. This allows the manufacture of coatings with properties similar to those of the feedstock powder. CS has been successfully applied to the deposition of a wide variety of materials for different applications including aluminium, copper, Ti and its alloys, WC-Co, Super-alloys and others onto various substrates [4-14].

Metals have many uses in structural, thermal, and optical engineering applications; in particular, metals can be used to manufacture reflective bodies or coatings. Metals are often used in optical applications for their durability, hardness and high reflectivity values over a wide range of wavelengths. Aluminium based alloys have high reflectivity values (>90%) in the visible and near-infrared wavelength range [15] which makes aluminium highly suitable for the cost-effective manufacture of reflectors. Aluminium is also plentiful in supply and straightforward to produce with traditional manufacturing techniques. Silver can also be employed as a reflective coating and has been shown to have the highest reflectivity of any known material over an extended wavelength range [16]; however, the drawback of silver is its softness, cost, and tendency to tarnish quickly through the formation of silver sulphide which markedly reduces reflectance [17]. Gold coatings are also employed in a number of applications such as cryogenics [18], thermal protection of orbiting satellites [19], biomedical applications [20,21] and optical reflectors; however, gold coatings come at a higher cost and lower reflectivity in the UV range when compared to aluminium [15]. Copper is also a highly reflective material in the visible and infrared range but forms a thick oxide layer reducing reflectivity even at room temperature [22]. This issue is exacerbated at elevated temperatures typically present in some

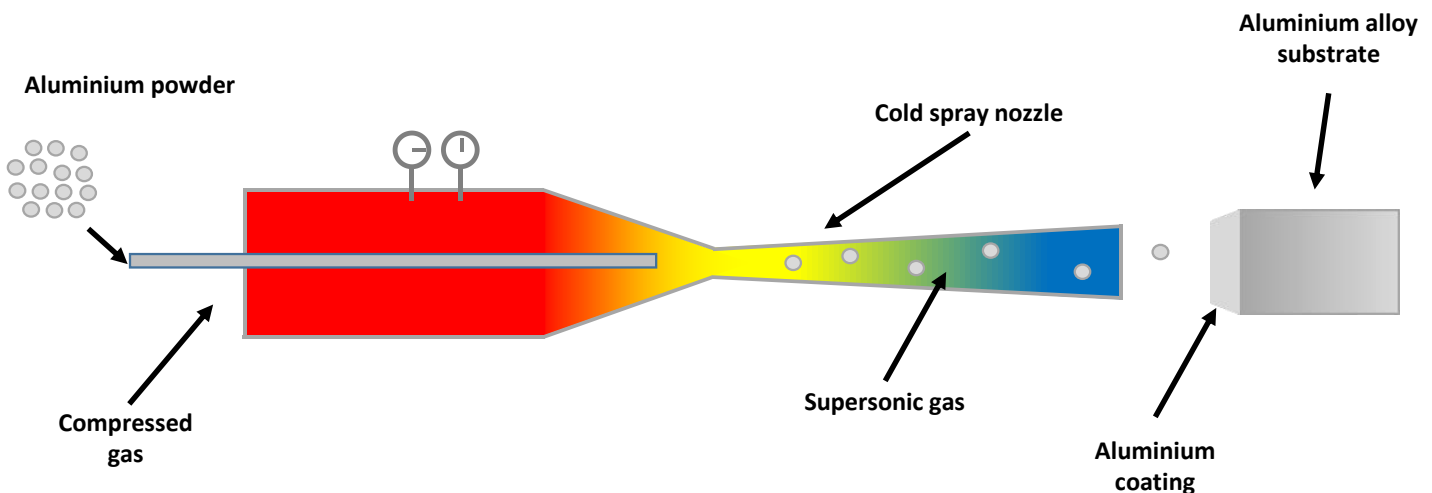


Figure 1: Cold spray schematic

of the applications mentioned. Thus aluminium is one of the most cost-effective and materials for manufacturing reflective surfaces and is the material which will be investigated.

There are a number of methods by which metallic coatings can be manufactured such as chemical vapour deposition [23], laser cladding [24], hot dipping [25], electroplating [26], sputtering [27], and thermal spray [28]. However, reflective coatings are typically manufactured with sputtering, electroplating or chemical vapour deposition. Recently, Zhu et al. investigated the optical properties of plasma sprayed Al, Cu and Ag coatings [29]. The results show coatings with excellent bond strength and high total reflectivity despite the lack of surface finishing. This work showed that optical properties of a sprayed coating are affected by the porosity and roughness of the coating, with a lower porosity and roughness being desirable. Oxidation can also play a significant role in the reflectivity of the coating, which is one key benefit of CS over plasma spray for this application, as CS is an oxide free process due to the use of inert gases within the gas stream.

As with many additive processes, finishing will be achieved with removal processes such as machining, polishing and superfinishing. However, there has been little research into the effect of removal processes on cold spray coatings with exception of the work of Aldwell et al. whom studied the machinability of cold spray coatings [30]. In addition, there has been lack of research carried out into the properties of 'sprayed' metal coatings besides the work of Zhu. Thus this paper aims to determine if it is possible to manufacture bespoke reflective surfaces using cold spray, specifically commercially pure Al, and investigate the most effective methodology to manufacture a high-quality reflector both regarding the CS parameters and post-spray finishing process.

## Experimental setup

### Coating fabrication procedure

The reflective coatings were manufactured using the in-house CS systems at Trinity College Dublin. The CS system comprises a de Laval nozzle, gas heater, CNC system, powder feeder, computer monitoring and control system, and a nitrogen supply. Nitrogen was used as the process gas for all the experiments undertaken. A schematic of the CS system is

highlighted in Figure 1. The gas is heated before injection into the nozzle head using a high-temperature gas heater capable of temperatures of up to 1000°C. Four gas heating temperatures ranging from 400°C to 550°C in 50°C intervals are investigated. The compressed gas at 30 Barg is introduced into the nozzle, and the powder is fed into the nozzle through the feedstock inlet. Before spraying the system was allowed to stabilise for 1 minute allow the gas heater to reach equilibrium. The gas and entrained powder enters the converging-diverging de Laval nozzle which accelerates the powders to supersonic speeds. High purity aluminium (H-30, >99.7% Al, Valimet Inc., California) was used in this investigation with a size distribution of 10-100 µm (Figure 3). The size distribution of the powders was measured using a laser diffraction particle analyser (Horiba LA 920).

Nozzle clogging can occur when spraying aluminium at elevated temperatures [31] and thus a nozzle cooling jacket was employed during this investigation. The nozzle cooling jacket is similar to the device used by Wang et al. [32]. Clogging began at 250°C without nozzle cooling, but spraying was possible up to 600°C without clogging while the nozzle cooling jacket was employed. The coatings were sprayed onto 30mm diameter x 25mm height billets of Aluminium 5 series alloy substrate as shown in Figure 2. Before spraying the surface of the billet was cleaned with isopropyl alcohol to remove residue or coolant remaining from machining. All samples were sprayed at a traverse speed of 100 mm/s, and the number of passes on each sample was varied to obtain a coating thickness of at approximately 1.5 mm. After spraying, the samples were machined or polished to between 0.4 mm and 0.5 mm coating thickness to remove the rough, undulating surface from the coating.



Figure 2: Substrate, cold sprayed sample, polished sample and buffered sample (left to right)

### Characterization and property tests

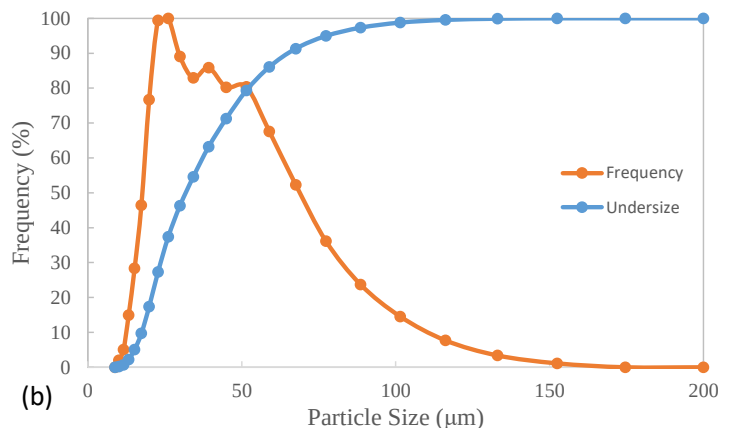
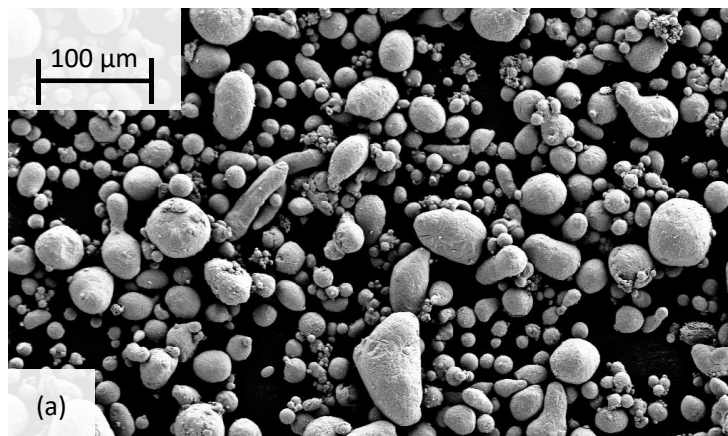


Figure 3: SEM of feedstock powder (a), particle size distribution (b)

Porosity was measured using ImageJ on polished cross-sections of the samples using an SEM (Carl Zeiss ULTRA, Germany). The visible surface porosity of the aluminium samples was measured using five randomly selected SEM surfaces images at a fixed magnification. ImageJ was used to process the images by employing the threshold feature to find the porosity value. The arithmetic average of the surface roughness ( $S_a$ ) was measured using a white light interferometer (WLI, Filmetrics, Profilm 3D) using a magnification of 50x. Five  $S_a$  measurements were taken for sample to increase accuracy. Porosity was also inferred from five density measurements for each sample carried out using an Archimedes balance. Samples were sprayed and subsequently removed from the billets using a lathe after being faced-off. The density values obtained using the Archimedes balance method allowed the porosity to be determined through the bulk density of aluminium. Reflectivity measurements were carried out using Perkin Elmer Lambda 900 between 400 and 1800 nm wavelength range in steps of 0.5 nm.

### Polishing procedure

The polishing compounds and grit papers sourced from MetPrep (UK) and the buffering wheels used and P175 Yellow compound were sourced from Metal Finishing Supplies Ltd (UK). To maximise the reflectivity of the final coating a number of polishing procedures were tested.

1. Samples were polished using P180 grit, P1200 Grit, 6  $\mu\text{m}$  diamond suspension, 1  $\mu\text{m}$  diamond suspension, and 0.06  $\mu\text{m}$  colloidal silica suspension for a total time of 1-2 minutes, 3 minutes, 3 minutes and 10 minutes respectively. These samples will be referred to as the polished samples herein.
2. Samples were polished using P180 grit, P1200 Grit, 6  $\mu\text{m}$  diamond suspension, 1  $\mu\text{m}$  suspension and 0.06  $\mu\text{m}$  colloidal silica for a total time of 1-2minutes, 3 minutes, 3 minutes and 10 minutes respectively, then polished with the buffering wheel before again polishing with 0.06  $\mu\text{m}$  colloidal silica suspension for 30 seconds. P175 Yellow compound was generously applied to the buffering wheel, and the sample was buffered until the polish was used which took approximately 5 minutes. These samples will be referred to as the buffered samples herein.
3. A single coating sample was milled from 1.5 mm to 0.5 mm thick before polishing with P1200 grit, 6  $\mu\text{m}$  diamond suspension, 1  $\mu\text{m}$  suspension, and 0.06  $\mu\text{m}$  colloidal silica suspension for a total time of 30 seconds minutes, 3 minutes and 10 minutes respectively. This sample will be referred to as the machined samples herein.

The polishing process was developed based on the recommended procedure and materials from MetPrep. It was found that this procedure did not produce coatings without visible porosity (3-7%), and therefore alternative finishing techniques were investigated. Firstly, a buffering process was added to the procedure as this was found to provide an excellent surface finish even with porous cold spray coatings. Secondly, a milling process was used to remove the top porous layer of

cold spray which was also found to densify the remaining layer of coating.

## Results and discussion

### Effect of gas heating temperature on coating quality

The primary characteristic of concern in this investigation is the reflectivity of the final surface produced by the CS parameters and the post-spray processes employed. The first section of the results will focus on the effect of the CS parameters (gas heating temperature) on the surface quality. The total reflectivity data of the polished samples is shown in Figure 4.

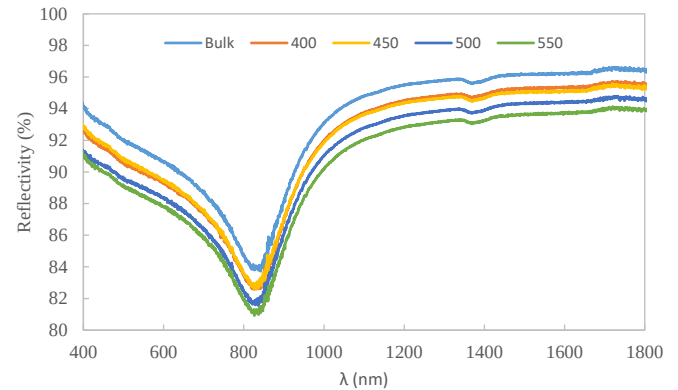


Figure 4: Total reflectivity data for various gas heating temperatures and bulk aluminium with polished sample preparation

It is clear that the bulk aluminium sample has the highest reflectivity of the range of wavelengths tested. The bulk aluminium will not have any significant surface porosity and/or defects that will present in the CS coatings and thus should have the highest reflectivity and provide a benchmark for the CS samples. There is a clear trend between the CS parameters tested and the final reflectivity values obtained: the surface reflectance decreases with an increase in gas heating temperature. The 550°C sample had the lowest average reflectance while the 400°C sample had the highest average reflectance.

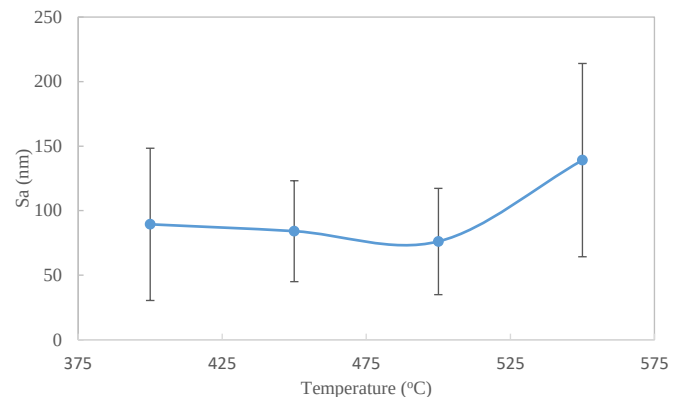


Figure 5: Gas heating temperature vs surface roughness for the polished samples with 95% confidence interval error bars

Figure 5 also shows that gas heating temperature affects the final surface quality with a higher gas heating temperature resulting in a higher coating surface roughness ( $S_a$ ) value. In

the majority of cases, a higher gas heating will result in improved CS coatings [33]; however, it is hypothesised that the increase in deposition efficiency that arises from the higher gas heating temperatures may cause a reduction in the ‘tamping’ effect on the coating. Tamping [34] has been shown to reduce the porosity of a CS coating and is typically employed by including a small percentage of hard materials (5-15%), such as alumina, within the feedstock powder. However, at low deposition efficiencies, the impacting powder which fails to deposit will cause densification of the coating through the tamping effect. In addition, the powder type used has a wide distribution of powder sizes. Larger particles are accelerated to lower speeds and thus may not exceed the critical velocity of the material and fail to deposit at low gas heating temperatures. At higher temperatures, the larger powders may exceed the critical velocity and deposit but form a poor coating due to their lower velocity when compared to smaller particles. Thus, the data from Figure 5 suggests that for this powder, lower gas heating temperatures lead to higher density coatings.

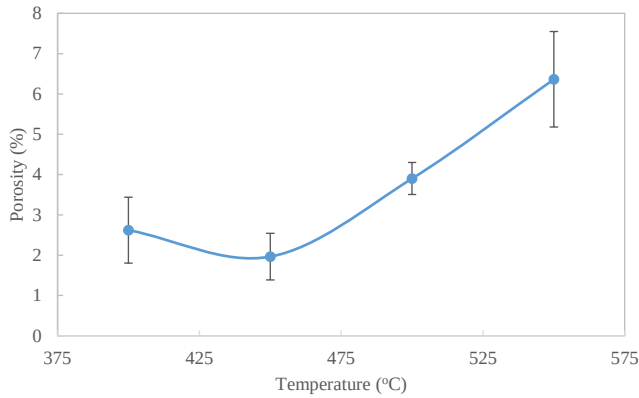


Figure 6: Gas heating temperature vs surface porosity measurements using ImageJ for the polished samples with 95% confidence interval error bars

Figure 6 shows effect of gas heating temperature on surface porosity, with porosity increasing with gas heating temperature. This reinforces the data presented in Figure 4 and Figure 5 which suggests that higher gas heating temperatures did not lead to increased coating quality as would have been expected. Again, this data suggests that lowest gas heating temperatures lead to improved density (due to the tamping effect), although there is a notable level of uncertainty at the lowest temperatures tested.

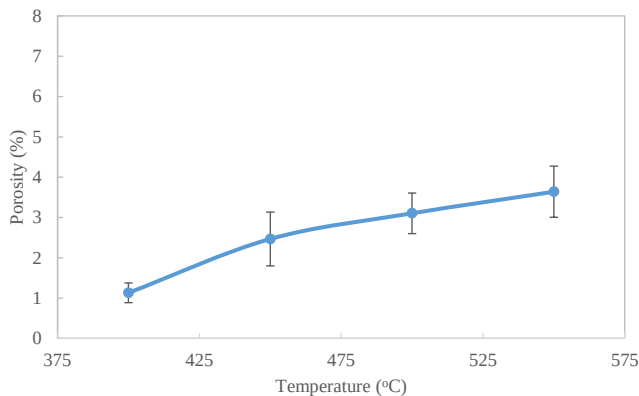


Figure 7: Gas heating temperature vs bulk porosity measurement with 95% confidence interval error bars

Figure 7 shows the bulk porosity of the coatings determined using the density of the samples which was measured with the Archimedes balance. This data confirms the trend that an increase in gas heating temperature leads to a decrease in density of the coating. This data would also indicate that a spray temperature of 400°C would provide the best reflectance values as shown in Figure 4.

Table 1: Summary of polished samples data

| Sample | Average Reflectance (%) | Sa (nm)        | Surface Porosity (%) | Bulk Porosity (%) |
|--------|-------------------------|----------------|----------------------|-------------------|
| Bulk   | 93.07                   | 19.49 ± 7.85   | 0.04 ± 0.03          | -                 |
| 400°C  | 91.97                   | 89.40 ± 59.03  | 2.62 ± 0.82          | 1.13 ± 0.24       |
| 450°C  | 91.92                   | 84.10 ± 39.13  | 1.96 ± 0.58          | 2.47 ± 0.66       |
| 500°C  | 91.00                   | 76.12 ± 41.22  | 3.90 ± 0.40          | 3.10 ± 0.50       |
| 550°C  | 90.35                   | 139.10 ± 74.31 | 6.36 ± 1.19          | 3.64 ± 0.63       |

### Effect of polishing procedure on surface finish

Figure 8 shows the total reflectivity for the buffered samples for various gas heating temperatures. The overall trend of gas heating improving the surface reflectance is visible in this data with the 400°C sample and the 550°C showing highest and lowest reflectance respectively.

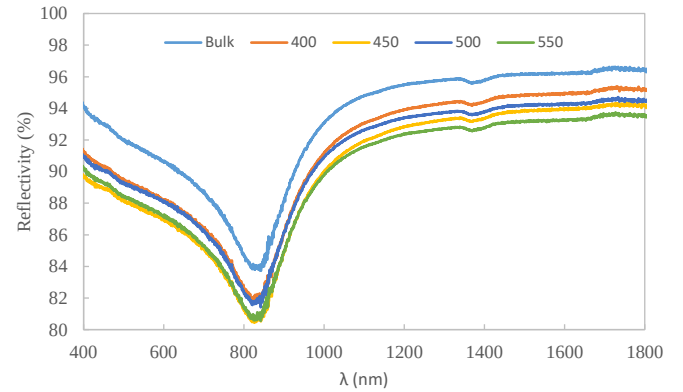


Figure 8: Total reflectivity data for various gas heating temperatures and bulk aluminium with buffered sample preparation

However, it is clear from Figure 8 that the buffered samples have a lower reflectance than the polished samples suggesting that the buffering process did not improve the surface finish despite the improved visual appearance. The effect of gas heating on the coating quality has also notably become less significant for the buffered samples which suggests that the buffering process smoothed the surface of the coating.



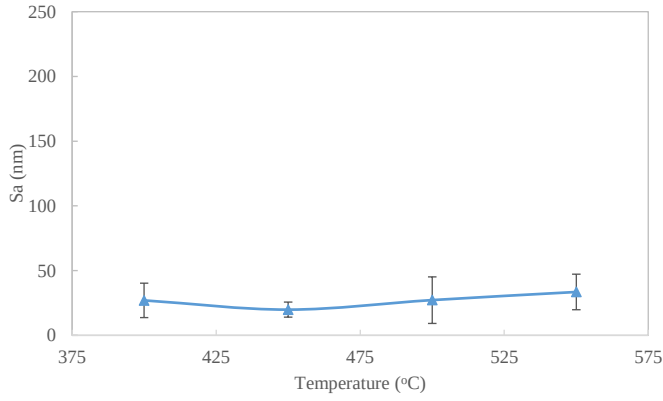


Figure 9: Gas heating temperature vs surface roughness ( $S_a$ ) for the buffered samples with 95% confidence interval error bars

The surface roughness of the buffered samples is significantly lower than the polished samples when comparing the data in Figure 9 to Figure 5. The buffered samples have a surface roughness ranging from approximately 20-50 nm whereas for the polished samples the  $S_a$  values range from 60-160 nm. This is in contrast to the reflectance data presented in Figure 3 and Figure 8 where the buffering process was shown to have a lower average reflectance than the polished samples. The buffering process decreases the variability in the measurements of the surface roughness with an average standard deviation of 6.5 nm compared to 27 nm for the polished samples.

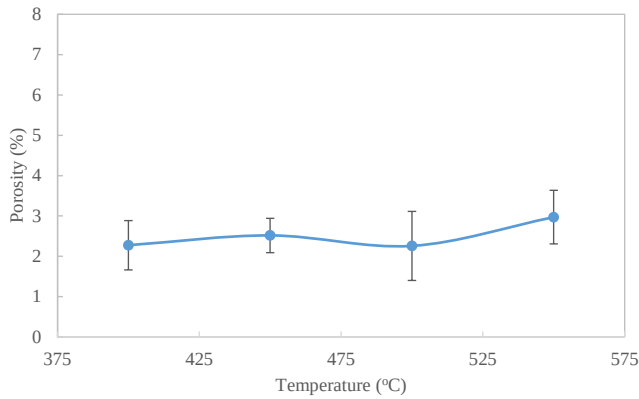


Figure 10: Gas heating temperature vs surface porosity measurements using ImageJ for the buffered samples with 95% confidence interval error bars

The porosity values data presented in Figure 10 for the buffered samples are lower than that seen in Figure 6. The maximum porosity values decreased from 6% to 3%. Thus, it is evident that the surface porosity is being “filled” by the buffering process which would be expected to improve the quality of the coating. A decrease in surface porosity and roughness should increase reflectivity as there are fewer pores to absorb the incoming photons. The  $S_a$  and porosity data do not corroborate the decreases in reflectance of the buffered samples compared to the polished samples. There is no apparent explanation for this discrepancy, and therefore, further investigation is required.

Table 2: Summary of buffered samples data

| Sample | Average Reflectance (%) | $S_a$ (nm)    | Surface Porosity (%) | Bulk Porosity (%) |
|--------|-------------------------|---------------|----------------------|-------------------|
| Bulk   | 93.07                   | 19.49 ± 7.85  | 0.04 ± 0.03          | -                 |
| 400°C  | 91.26                   | 26.98 ± 13.23 | 2.2754 ± 0.61        | 1.13 ± 0.24       |
| 450°C  | 90.10                   | 19.84 ± 4.88  | 2.5174 ± 0.43        | 2.47 ± 0.66       |
| 500°C  | 90.85                   | 27.22 ± 18.00 | 2.2572 ± 0.85        | 3.10 ± 0.50       |
| 550°C  | 89.87                   | 33.48 ± 13.67 | 2.9702 ± 0.66        | 3.64 ± 0.63       |

Effect of machining on coating quality

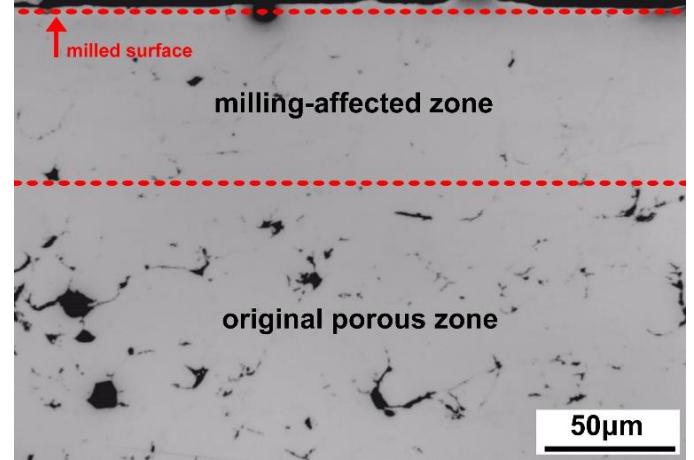


Figure 11: Cross section of a milled sample showing densified top layer

Figure 11 shows an image of a cross-section of an aluminium sample which was machined for this investigation; the image shows that the top layer has been densified by the machining process while the lower layers are porous. The densification of the top layer of cold spray coatings has been described previously in the literature [30]. This technique could potentially reduce surface porosity levels by smearing the surface layer which could then be polished to achieve higher reflectivity levels. The reflectivity data for the machined, the bulk aluminium, the polished and the buffered sample are shown in Figure 12.

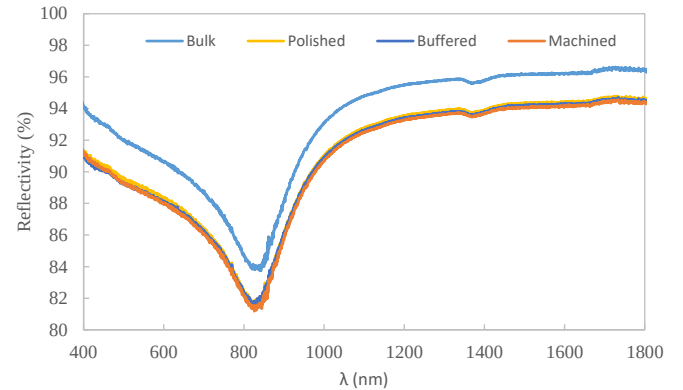


Figure 12: Total reflectivity data for various sample preparation techniques sprayed at 500°C

The reflectivity data of the various sample preparations show that there is no notable difference between any of the samples

and that the samples had reflectivity values below that of the bulk sample. This would suggest that the most crucial factor tested is the temperature of the gas heating. The effect of machining on the reflectance is negligible and the buffering process has a negligible or slightly adverse impact based on the data presented table 1 and table 2.

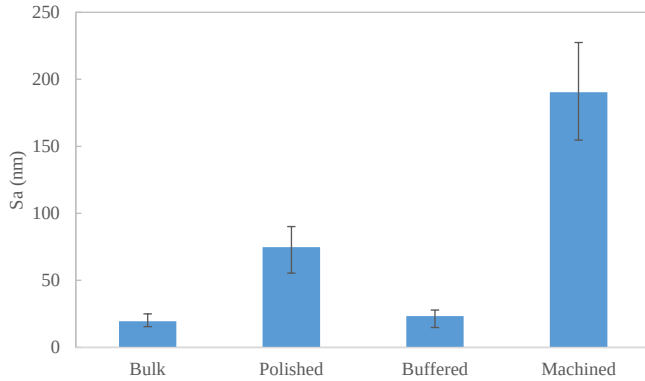


Figure 13: Surface roughness of various sample preparation techniques sprayed at 500°C with 95% confidence interval error bars

The surface roughness data for the various preparation techniques are compared to the bulk aluminium sample in Figure 13. The polished sample showed a higher Sa value than the bulk whereas the buffered sample showed a similar Sa value to the bulk aluminium sample. This data is again contradictory to the reflectance data which showed that the buffering adversely affected the coating quality. The machining process did not have a positive effect on the roughness of the substrate and in fact resulted in a higher roughness than the polished sample. The increase in roughness could potentially be due to changes in material properties such as hardness which was not explored in this study. This area of the study warrants further investigation.

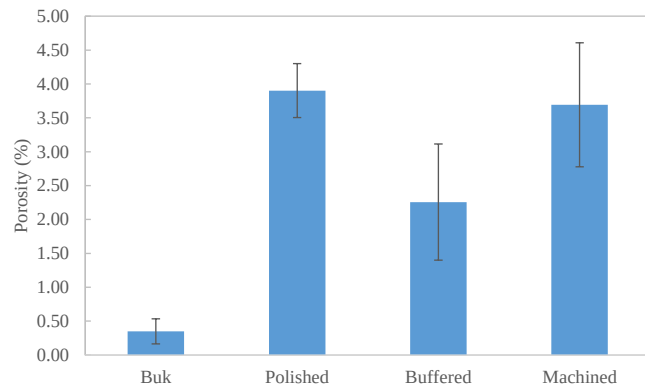


Figure 14: Surface porosity measurement for various sample preparations sprayed at 500°C with 95% confidence interval error bars

The porosity data in Figure 14 for the various samples show that the bulk material had a significantly lower porosity than the other techniques investigated. This is to be expected as the CS process will not produce coatings which are 100% dense. The porosity of the polished sample is significantly higher than the

bulk sample (4% vs 0.3%) and the buffered sample shows a significant improvement over the polished sample (2.5%). Data for the machined coating shows a negligible effect on porosity; however, it is difficult to tell definitively due to the error associated with the measurement of porosity.

Table 3: Summary of data for various finishing techniques at 500°C gas heating temperature

| Sample   | Average Reflectance (%) | Sa (nm)         | Surface Porosity (%) |
|----------|-------------------------|-----------------|----------------------|
| Bulk     | 93.07                   | 19.49 ± 7.85    | 0.04 ± 0.03          |
| Polished | 91.00                   | 76.12 ± 41.22   | 3.90 ± 0.40          |
| Buffered | 90.85                   | 27.22 ± 18.00   | 2.26 ± 0.85          |
| Machined | 90.72                   | 151.02 ± 109.15 | 3.70 ± 0.91          |

## Conclusions

This research demonstrates that CS can be used to manufacture reflective coating which approaches the reflectance of bulk material (92% vs 93% average reflectance respectively). A higher porosity of the CS coatings was predictably shown to decrease the reflectance of the CS coatings. The porosity of the coatings was shown to increase with gas heating temperatures; this is thought to be a result of the size distribution of the feedstock powder. The polishing procedure had a negligible impact on the final coating quality despite the buffering process decreasing surface porosity and roughness. The decrease in surface roughness and porosity did not translate to an increase in coating reflectance. Finally, machining of the surface before polishing did not appear to have an impact on the reflectance of the coating despite previous tests showing a densified top layer after the machining process.

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