Improvement of tensile strength of cold sprayed Fe deposits via in-process powder preheating

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**A B S T R A C T**

Cold sprayed Fe deposits were manufactured with non-preheated and preheated powders, respectively. The microstructure of the deposits was characterized by electron backscattered diffraction. The tensile property of the deposits was then evaluated. The results show that the preheated Fe powder deforms more extensively than the non-preheated powder during cold spraying due to their thermal softening. The ultimate tensile strength of the deposits made with preheated powder approximately doubles to $109.42 \pm 50.56$ MPa due to the enhanced intersplat bonding. However, the deposits in both cases exhibit poor ductility. The increased dislocation density and the nano-sized oxides on the preheated powder surface are considered to be the cause of low ductility of the deposits fabricated with preheated powder.

1. Introduction

Cold spray is a promising solid-state additive manufacturing process in recent years. However, cold sprayed deposits generally have unfavorable mechanical properties in their as-fabricated state due to the inherent microstructural defects, such as pores and incomplete intersplat bonding. This downside reduces its competitiveness and limits its applications in additive manufacturing structural materials.

Powder preheating is a strengthening strategy which uses a heating chamber to heat and thermally soften powder before they enter cold spray nozzle so as to facilitate their plastic deformation \cite{1}. Previous studies have reported that heating feedstock powder to a suitable temperature range before spraying can decrease critical velocity, increase deposition efficiency and lead to higher density and adhesive strength of the deposits \cite{1–3}. However, to date, an investigation on whether powder preheating can improve the mechanical properties of thick cold sprayed deposits is still lacking. Therefore, in this work, a targeted study was conducted on cold sprayed Fe deposits to clarify the role of powder preheating in the improvement of mechanical properties.

2. Material and methods

Water-atomized Fe powder with an average size of 27 μm was used as feedstock. Fig. S1 shows the surface morphology of the powder characterized by scanning electron microscope (SEM, Carl Zeiss ULTRA Plus, Germany). The deposition of Fe deposit was achieved by using a commercial cold spray system (Plasma Giken, Japan) with two different nozzles. Both nozzles had the same geometric dimensions, while the nozzle used for in-process powder preheating had a pre-chamber with a length of 100 mm and connected to the nozzle inlet. The long pre-chamber allows powder to stay in a high-temperature gas environment for a longer period and be heated to a higher temperature. Compressed nitrogen at 5.0 MPa and 850 °C was selected as propulsion gas. The standoff distance and nozzle traversal speed were 40 mm and 300 mm/s, respectively.

To investigate the microstructure of the deposits, the cross-sectional samples were prepared using standard metallographic procedures. The grain structure evolution and plastic deformation behavior were achieved by using a high-resolution field emission scanning electron microscopy (FE-SEM, ZEISS Sigma 300VP, Germany) equipped with an

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electron backscattered diffraction system (EBSD, Oxford Instruments, UK). The tensile property of the deposits was evaluated by a universal
tensile testing machine (INSTRON-3382, USA) at a displacement rate of
1 mm/min. Electrical discharge machining was applied to cut the tensile
specimens into dog-bone shape from the deposits. The specimen had a
gauge length of 25 mm, a gauge width of 3 mm and a thickness of 2 mm.
The ultimate tensile strength (UTS) and elongation (EL) of each sample
group were calculated based on the average value of two specimens. The
typical fracture morphology was observed by SEM.

3. Results and discussion

Fig. 1 shows the EBSD characterization results of the cold sprayed Fe
deposits fabricated using preheated and non-preheated powders. For the
deposits manufactured with non-preheated powder, the grains at
intersplat interfaces experienced significantly refinement due to dy-
namic recrystallization induced by severe plastic deformation, while
course grains remained in the splat interior. For the deposits fabricated
with thermally softened particles, similar grains feature of the impact-
induced ultrafine grains at the highly deformed intersplat interfaces can be observed. Moreover, increased subgrain boundaries appeared in the large-sized splat interior in particular, which implied the more extensive plastic deformation of these particles.

To further evaluate the degree of plastic deformation of the deposits in both cases, the Kernel average misorientation (KAM) and local misorientation distribution maps were achieved, as shown in Fig. 2. The high level of misorientation of the deposits fabricated with non-preheated powder was mainly concentrated near the interfaces between the micro-sized particles (see Fig. 2a and c), indicating the increased local dislocation density and dislocation pileup formation [4]. However, the strain-free regions remained in some of the splat interior due to the insufficient particle deformation. The KAM results are generally consistent with the grain structure feature in Fig. 1a and b. For the deposits manufactured with preheated powder, the high local misorientation appeared at both the intersplat interfaces and the splat interiors (see Fig. 2b and c), and the strain-free regions dramatically decreased. Such observation indicated the preheated powder experienced more severe global deformation and the increased density of the defects formed in the particle. Our previous modeling work also revealed that the effective plastic strain and the particle compressional ratio of a preheated Fe particle impacting onto a Fe substrate increased due to the enhanced thermal softening effect [1].

It is widely accepted that adiabatic shear instability is a critical condition for particle deposition in cold spray [5]. More specifically, particles can easily deform under low shear stress when thermal softening effect induced by adiabatic temperature rise dominates work-hardening effect resulted from increased strain. The thermal softening is considered to be triggered when the temperature of a deformed region exceeds a critical value (i.e., 0.4–0.5 melting temperature) [1]. For non-preheated powder in conventional cold spray, thermal softening mainly concentrates at the narrow interfacial region, and consequently metal jet forms at the rim of the particle. While for preheated powder, both particle preheating temperature and adiabatic heating contributed to the final particle temperature upon deposition. Therefore, the plastic flow induced by thermal softening effect could spread over a larger region, rather than only at interfacial areas [1]. In other words, preheated particles can experience more extensive plastic deformation than non-preheated particles, as evidenced by a higher level of local misorientation in Fig. 2c. The thermal softening effect can also be reflected from the increased deposition efficiency of the deposit prepared with preheated powder (92.57%), compared with that of the deposit manufactured with non-preheated powder (78.47%).

Fig. 3a and b show the representative engineering stress–strain curves, UTS and EL of the deposits. The deposit made with preheated powder had almost twice the UTS (109.42 ± 50.56 MPa) of the deposit made with non-preheated powder. However, both of the deposits exhibited poor ductility (~1%). Fig. 3c and d show the fracture surface morphology of the deposit fabricated with preheated powder. Nano-sized oxides can be seen on the surface of the preheated powder, which contrasts sharply to the clean surface of feedstock powder (see Fig. S1). It can be inferred that these nano-sized oxides were formed during the powder preheating.

For the deposits manufactured with non-preheated powder, the low strength and low ductility can be attributed to the incomplete intersplat bonding and microstructural defects. While for the deposit fabricated with preheated powder, the thermal softening effect can enhance metallurgical bonding between intersplat interfaces [6]. In addition, the metal jets of preheated particles can be enhanced due to thermal softening effect, resulting in stronger mechanical interlocking [6]. These factors jointly contributed to the enhanced particle bonding. It is worth noting that the oxides on the powder surface (see Fig. 3d) might cause oxide inclusion at intersplat interfaces, which was not conducive to the intimate bonding between particles and would weaken the promoted...
particle bonding induced by powder preheating. However, the enhanced interfacial bonding through thermal softening effect was probably more dominant, and therefore, the increased UTS was achieved on the whole. The low ductility of the deposits prepared with preheated powder can be mainly attributed to the increased dislocation density resulted from the enhanced plastic deformation. Furthermore, the oxides at the intersplat interfaces may hinder the movement of dislocations. Accordingly, the deposits fabricated with preheated powder exhibited increased tensile strength but low ductility. The current results clearly indicate that in-process powder preheating is an effective strengthening strategy to improve the tensile strength of cold sprayed deposits. However, the powder preheating temperature should be carefully controlled to prevent oxidation, particularly for oxidation sensitive materials.

4. Conclusions The effect of in-process powder preheating on the microstructure and tensile property of cold sprayed Fe deposits was investigated. The results suggest that preheated powder experience more severe plastic deformation upon impact than non-preheated powder. The UTS of the deposits fabricated with preheated powder dramatically increases from 64.58 ± 42.07 MPa to 109.42 ± 50.56 MPa because of the enhanced interfacial bonding. However, the deposits fabricated with preheated powder exhibit as low ductility as as-sprayed deposits due to the increased dislocation density and the formed nano-sized oxides at intersplat interfaces. In brief, in-process powder preheating significantly improves the tensile strength of cold sprayed Fe deposits. But the oxidation caused by powder preheating requires to be noticed because of its adverse effects on the tensile property of deposits.

CRediT authorship contribution statement

Declaration of Competing Interest
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data
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References