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Cold Spray Forming Inconel 718

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Abstract

In this investigation, Inconel 718, a material known to cause nozzle clogging upon cold spraying, was cold spray formed to 6 mm-thick using the Plasma Giken cold spray system PCS-1000. This was made possible due to the novel non-clogging nozzle material combined with a nozzle water cooling system. Coatings were as-spray formed using both nitrogen and helium as the propelling gasses. The resulting microstructures as well as the corresponding mechanical properties were studied. In addition, the effect of post-heat treatments was also investigated. It was found that for a given propelling gas used, the coating porosity level remained relatively similar (about 2.4% for nitrogen and 3.6% for helium) regardless of the coating treatment (as-sprayed or heat treated). Visual inspection from SEM micrographs showed a higher fraction of inter-particle metallurgical bonds for nitrogen gas sprayed coatings heat treated at 1250°C for 1 hour due to some sintering effect. This significantly affected its tensile properties with an average resulting ductility of 24.7%.

Introduction

Cold spray technology has tremendous potential for producing thick coatings with soft metals such as aluminium and copper (Ref 1-6). However, high critical velocity, residual stresses, and technical issues such as nozzle clogging have limited the number of materials that have been cold spray formed (Ref 7-10). For example, although the aerospace industry would consider cold spray forming nickel-based superalloys (i.e., Inconel 718) for many potential applications, this material has been challenging to spray mainly due to nozzle clogging. This problem has been recently solved in commercial cold spray equipment by using a non-clogging nozzle material combined with a nozzle water cooling system. In this work, Inconel 718 powder was cold sprayed using Plasma Giken PCS-1000 equipment to produce 6 mm-thick coatings on 8 cm-diameter aluminium tubes using both helium and nitrogen as the propelling gasses. The spray formed parts were sectioned to produce several samples for material characterisation through SEM and for mechanical tensile testing. The effect of post-sprayed heat treatment was also investigated.

Experimental

Feedstock Powder

Commercially available Inconel 718 powder (Amdry 1718) manufactured by Sulzer Metco (Troy, USA) was used. The powder size distribution was measured with a laser diffraction particle size analyser, the Beckman Coulter Model LS 13 320. Fig. 1(a) shows the particle size distribution of the Inconel 718 feedstock powder. Note that the powder revealed a narrow particle size distribution. The mean particle size was 37 µm. Fig. 1(b) shows the spherical morphology of the Inconel 718 feedstock powder, while Fig. 1(c) shows an as- polished cross-section. The latter micrograph indicates that the particles were non-porous.

Cold Spray Parameters

Coatings were produced with the PCS-1000 (Plasma Giken Co., Tokyo, Japan) cold spray equipment. The spray parameters using nitrogen gas include: propelling gas pressure and temperature of 5 MPa and 1000°C, respectively, a nozzle standoff distance of 25 mm, a substrate holder rotation speed of 600 rpm, and a gun traverse speed of 10 mm/s. The coating produced was approximately 6 mm-thick. Similar spray parameters were used using helium as the propelling gas; the only exception was that an inlet gas pressure of 2 MPa was used instead of 5 MPa. The Inconel 718 powder was cold sprayed onto 8 cm-diameter aluminium tubes.

Sample Extractions and Heat Treatment Conditions and Procedure

The as-sprayed Inconel 718 coatings were sectioned through wire-EDM in order to extract tensile test samples (perpendicular to the plane of spray direction). The dimensions of each tensile test are depicted in Fig. 2 above. Four tensile test specimens were kept as-sprayed while others were post-heat treated in a furnace under a 90% argon/10%

hydrogen atmosphere at 950°C/2 hours, 1010°C/2 hours, 1060°C/2 hours, and 1250°C/1 hour (3 samples for each heat treatment).

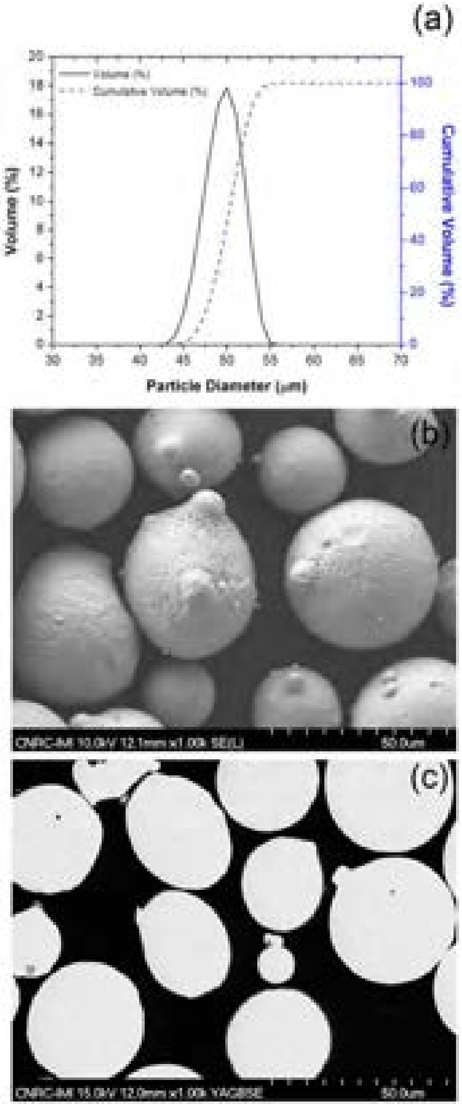

Figure 1. (a) Feedstock powder size distribution, (b) FEG-SEM micrograph depicting the morphology of the spherical Inconel 718 powder, and (c) As-polished powder cross-sections.

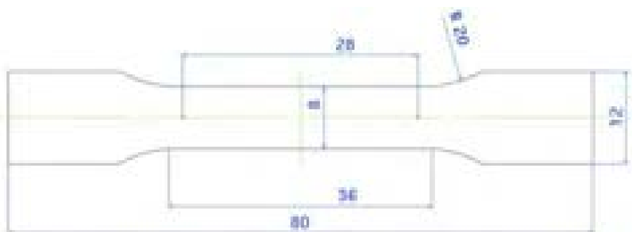

Figure 2. Dimensions of the tensile test specimens extracted via wire-EDM (all numbers are in mm).

The heat treatment procedure is outlined below. The samples were placed onto a rectangular metallic stage/holder which was pre-treated with boron nitride in order to prevent the samples from adhering onto the stage/holder. Next, the stage/holder was inserted into a glass tube which has two openings: one end with an opening diameter of 101.6 mm (from which the stage/holder was inserted) and the other end with a small pinhole opening diameter of 3.5 mm (for purging air from the glass tube). A specialised cover with a thermocouple and a plastic tube for argon gas flow injection attached to it was then placed and tightened onto the larger glass tube opening. Then, the argon gas at 6 SCFH (Standard Cubic Feet Hour) was released into the plastic tube for at least 20 minutes in order to purge the air from the glass tube and to completely surround the samples in an argon atmosphere (continuous argon gas flow). An air furnace with an opening of the same dimensions as the glass tube was set to 700°C. When this temperature was reached, the glass tube was partially inserted inside the air furnace and was left there while 10% hydrogen gas was injected into the argon gas flow. When the gas mixture was completed, the tube was fully inserted inside the air furnace where the temperature rose at a constant rate of 10°C per minute until the desired heat treatment temperature was reached for each set of coating heat treatment. After the heat treatment was completed, the hydrogen gas was turned off and the glass tube was partially removed from the air furnace in order to allow the remaining hydrogen gas to burn. Meanwhile, the tube was left to cool down to room temperature. This was performed while maintaining the continuous argon gas flow atmosphere inside the glass tube until the glass tube reached room temperature, after which the heat treated samples were removed.

Metallographic Preparation and Examination

Using standard metallographic preparation procedures, the as-sprayed and heat treated samples were sectioned with a coolant-assisted diamond wheel, cold vacuum-mounted in an epoxy resin then ground and polished. Microstructural observations were performed using a JEOL 840 scanning electron microscope (SEM) and a field emission gun scanning electron microscope, the Hitachi S-4700 (FEG-SEM). Powder morphology was analysed using FEG-SEM in secondary electron imaging (SEI) mode, while the as-polished coating cross-sections were analysed in backscattered electron imaging (BSI) mode. SEM BSI mode was also used to conduct coating porosity measurements. These were assessed on the as-polished samples with the use of image analysis software. A minimum of ten random images were taken and porosity was evaluated for each image.

Tensile Testing

Tensile tests were performed in accordance with the ASTM E8/E8M – 09 standard using an Instron 5582 universal testing machine (Burlington, Ontario, Canada) with a dynamic load up to 100 kN at a constant speed of 0.05 mm/sec. All tests used an extensometer with a gage length of 25 mm.

Results and Discussion

As-Sprayed and Heat Treated Cold Sprayed Inconel 718 Coating Characterisations

The porosity of the as-sprayed and heat treated coatings are presented in Table 1 (for both nitrogen and helium propelling gasses used). The error numbers presented in Table 1 represent the standard error.

Table 1. Coating Porosity of As-Sprayed and Heat Treated Samples

Coating Treatment	Porosity % (N_2 Inlet Gas)	Porosity % (He Inlet Gas)
As-Sprayed	2.7 ± 0.4	3.4 ± 0.4
950°C/2hrs	2.4 ± 0.2	3.3 ± 0.5
1010°C/2hrs	1.9 ± 0.2	3.7 ± 0.3
1060°C/2hrs	2.8 ± 0.2	3.7 ± 0.4
1250°C/1hr	2.0 ± 0.6	3.8 ± 0.3

Cross-sections of the as-sprayed and heat treated cold sprayed Inconel 718 coatings for nitrogen and helium propelling gasses are shown in Figs. 3 and 4, respectively. When comparing Figs. 3 and 4 from visual inspection, it appears that the as-cold sprayed and heat treated coatings using nitrogen as the inlet propelling gas were slightly denser than those produced using helium as the propelling gas. Quantitative analyses further support these observations (see Table 1). For coatings sprayed with a specific propelling gas (either with nitrogen or helium), the coating porosity level remained relatively similar regardless of the coating treatment (as-sprayed or heat treated). In all cases (Figs. 3 and 4), the pore distributions were relatively homogeneous across the coating cross-sections. However, closer observation revealed that, for the coating sprayed with nitrogen as the propelling gas and subsequently heat treated at 1250°C for 1 hour (Fig. 3(e)), its microstructure showed a distinct feature: the inter-particle boundaries were almost invisible compared to all other coatings investigated. This may perhaps be due to some sintering effect at this high temperature combined with the initial cold spray conditions.

Impacting particle velocities were measured for the cases of nitrogen sprayed (787 m/s) and helium sprayed (741 m/s) coatings. The impacting particle velocity for nitrogen sprayed coatings was faster than those sprayed with helium, possibly because: (1) although the inlet gas temperature was set to 1000°C in both cases, the inlet gas pressure was significantly lower when using helium as the propelling gas (2 MPa versus 5 MPa) and (2) the cold spray gun nozzle was not optimised. Thus, although the impacting particle velocity is usually higher when using helium as the propelling gas (Ref 11-12), in this specific situation, the particle velocity was faster when using nitrogen as the propelling gas. Consequently, higher particle plastic deformation is expected to occur with an increased fraction of adiabatic shear instability (ASI) occurrences between inter-particle boundaries. This is shown schematically in Fig. 5.

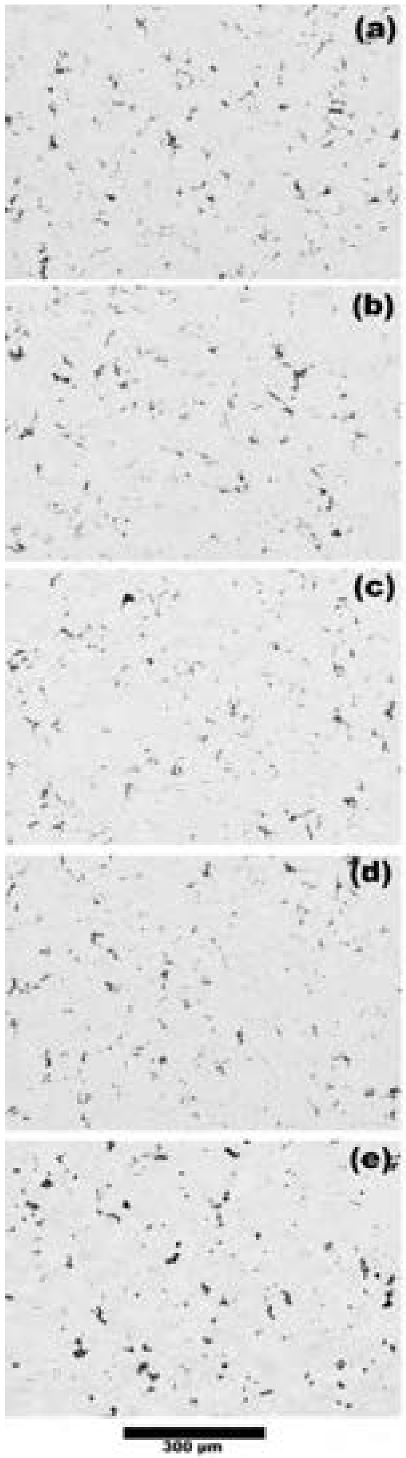

Figure 3. Cross-sections of cold sprayed Inconel 718 coatings using nitrogen as the propelling gas: (a) As-sprayed, (b) Heat treated at 950°C/2hrs, (c) Heat treated at 1010°C/2hrs, (d) Heat treated at 1060°C/2hrs, and (e) Heat treated at 1250°C/1hr.

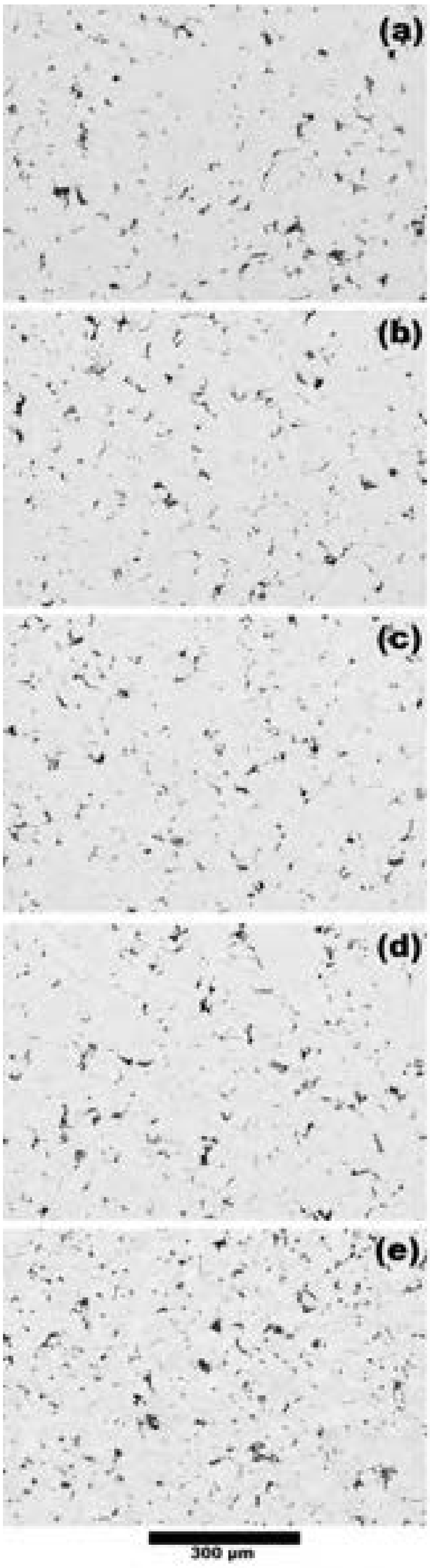

Figure 4. Cross-sections of cold sprayed Inconel 718 coatings using helium as the propelling gas: (a) As-sprayed, (b) Heat treated at 950°C/2hrs, (c) Heat treated at 1010°C/2hrs, (d) Heat treated at 1060°C/2hrs, and (e) Heat treated at 1250°C/1hr.

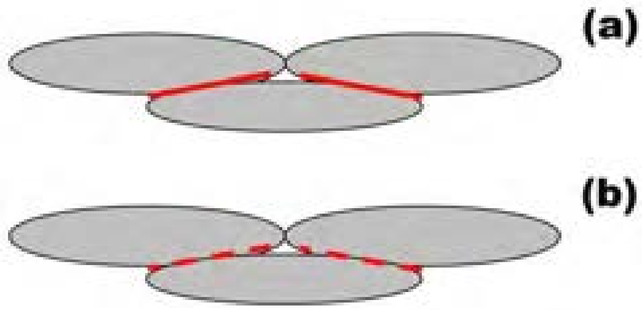

Figure 5. Schematic representation of particle deformation having higher impacting particle velocity and ASI occurrences (a) as opposed to lower impacting particle velocity and ASI occurrences (b).

Thus, the as-sprayed coatings using nitrogen as the propelling gas were expected to have an increased fraction of metallurgical bonding and consequently, a lower coating porosity level, as also shown from quantitative analyses through image analysis in Table 1. Following heat treatment at a high temperature (1250°C for 1 hour), sintering occurred. The stages of sintering are schematically depicted in Fig. 6. During the initial stage of sintering, some metallurgical bonding was already present from ASI during the cold spray process. Neck growth took place when moving from the initial to the intermediate stage, increasing the metallurgically bonded area. Finally, pore spherodisations occurred and in certain cases, these pores may be replaced by metallurgically bonded inter-particle boundaries (Ref 13). Due to the initially lower coating porosity level in the as-sprayed coatings produced using nitrogen gas (Fig. 3(a)) as opposed to the helium gas (Fig. 4(a)), fewer spherodised pores from the final stage of sintering would be expected for the nitrogen gas sprayed coatings. This was further confirmed from visual inspection of Fig. 3(e) and Fig. 4(e) at higher magnifications, shown in Figs. 7 and 8, respectively. The red arrows indicate the spherodised pores after sintering at a high temperature. Thus, this could explain the dissipation of the inter-particle boundaries seen in Fig. 3(e) and not in Fig. 4(e).

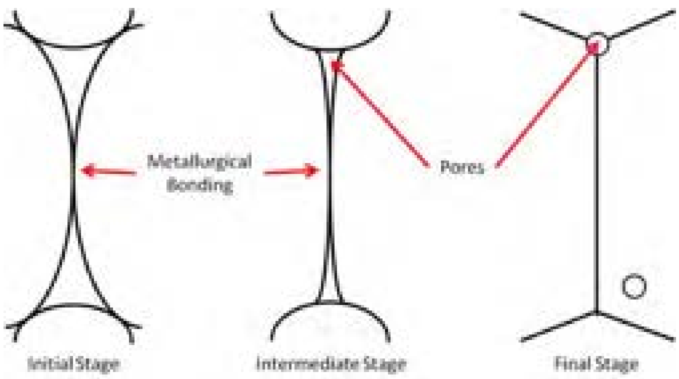

Figure 6. Schematic representation of the sintering stages.

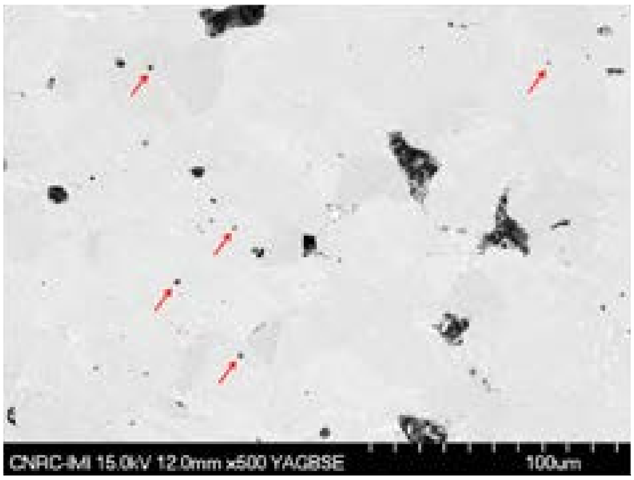

Figure 7. Cross-section of cold sprayed Inconel 718 coatings using nitrogen as the propelling gas heat treated at 1250°C for 1 hour.

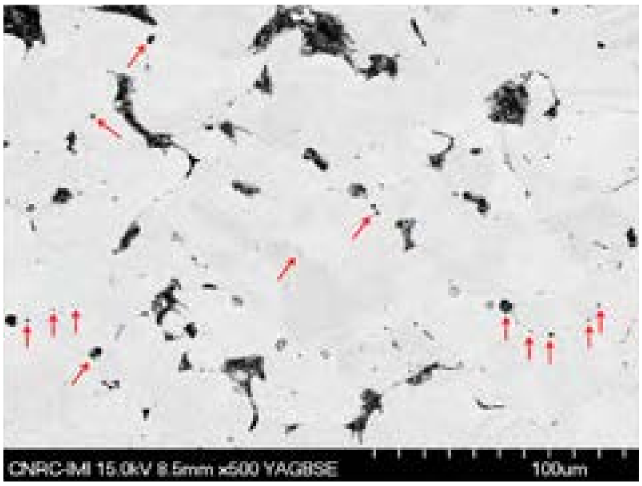

Figure 8. Cross-section of cold sprayed Inconel 718 coatings using helium as the propelling gas heat treated at 1250°C for 1 hour.

Tensile Strength and Ductility

The resulting observations from the previous Section may have strong influences on the resulting mechanical properties of these coatings. To verify this hypothesis, tensile tests were performed for all coatings presented in Figs. 3 and 4. Examples of the resulting engineering stress versus engineering strain curves are shown in Figs. 9 and 10, respectively.

From Figs. 9 and 10, it can be clearly seen that regardless of the propelling gas used to produce the Inconel 718 coatings, as the heat treatment temperature increased, the coating became more ductile (increasing engineering strain). This may be explained by the better inter-particle (metallurgical) bonding which resulted from the sintering at the high temperatures.

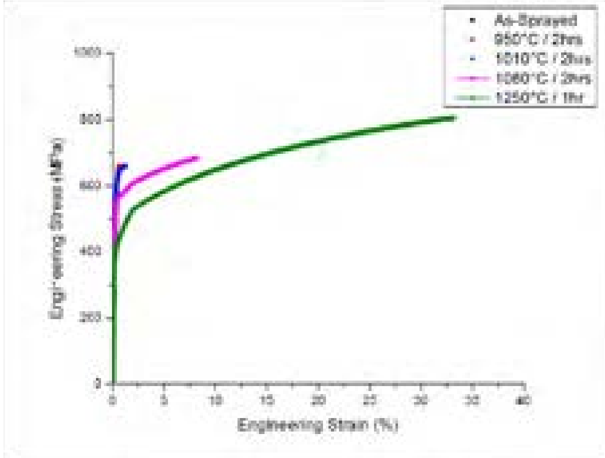

Figure 9. Tensile test results for as-cold sprayed and heat treated Inconel 718 coatings using nitrogen as the propelling gas.

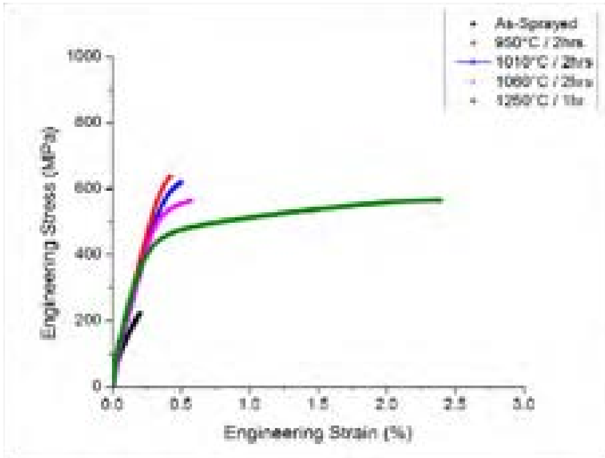

Figure 10. Tensile test results for as-cold sprayed and heat treated Inconel 718 coatings using helium as the propelling gas.

As expected, the Inconel 718 coating that was as-cold sprayed using nitrogen gas and was subsequently subjected to a heat treatment at 1250°C for 1 hour revealed the highest ductility by far, with an average engineering strain value of 24.7% from three repeats. This ductility has even exceeded certain specific bulk Inconel 718 sheets, strips, and plates that have been annealed and aged (24.7% versus 11-12%). On the other hand, the helium gas sprayed Inconel 718 coating that was subjected to the same heat treatment only showed an average engineering strain value of 2.2% from three repeats. One plausible explanation of the low ductility for the helium cold sprayed coatings would be due to the amount of plastic deformation (i.e., cold work) prior to the heat treatment. It is well known that the amount of cold work has a critical influence on the ductility of nickel and nickel alloys (i.e., Inconel 718) after annealing heat treatments, which are typically performed above 955°C for approximately an hour (Ref 13). When a low amount of cold work or plastic deformation is present prior to heat treatment (as is the case with the helium sprayed coating), full ductility usually cannot be restored by annealing because of excessive grain growth due to critical strain, even if the hardness is recovered to that of the pre-cold worked state (Ref 13). In addition, since the helium cold sprayed coatings would have a lower fraction of metallurgically bonded inter-particle boundaries after sintering than the nitrogen coatings, this would further reduce the likelihood of achieving higher ductility. Thus, a minimum amount of plastic deformation is required to ensure maximum ductility and softness after annealing heat treatments. Further investigation on this matter would be required. In addition, when comparing Figs. 9 and 10, for the nitrogen gas sprayed coatings, increasing heat treatment temperatures not only increased the engineering strain, but also increased the engineering stress. However, this was not observed for the helium gas sprayed coatings, in which increasing heat treatment temperatures increased the engineering strain but decreased engineering stress. These issues will be addressed in the near future along with a more detailed microstructural investigation of the coating cross-sections through chemical etching.

Conclusions

The microstructural and mechanical properties of as-sprayed and heat treated cold sprayed Inconel 718 coatings were investigated. The resulting coatings microstructures and porosity were analysed. In addition, tensile tests were performed and the results were analysed and discussed. It was concluded that:

(1) In general, coatings sprayed with a specific propelling gas (either with nitrogen or helium) revealed similar coating porosity levels regardless of the coating treatment (as-sprayed or heat treated).
(2) After heat treatment at 1250°C for 1 hour, the coating sprayed with nitrogen propelling gas showed a significant amount of inter-particle metallurgical bonds that was not seen in all other coatings investigated (i.e., coatings heat treated at lower temperatures or coatings sprayed with helium propelling gas). It was hypothesised that this could be due to some sintering effect at this high temperature combined with the initial cold spray conditions which in turn potentially resulted in high impacting particle velocities and consequently, a large fraction of adiabatic shear instability occurrences.
(3) Enhanced inter-particle metallurgical bonds significantly influenced the resulting mechanical properties of the coating such as ductility. From the tensile test results, the highest ductility was found to be at about 24.7% for the nitrogen gas sprayed Inconel 718 coating which was heat treated at 1250°C for 1 hour. The corresponding ultimate tensile stress was about 763.6 MPa, which is approximately 70% of that of the bulk strength.

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