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2019

Sun, W., Bhowmik, A., Tan, A. W.-Y., Li, R., Xue, F., Marinescu, I., & Liu, E. (2019). Improving microstructural and mechanical characteristics of cold-sprayed Inconel 718 deposits via local induction heat treatment. *Journal of Alloys and Compounds*, 797, 1268–1279.
doi:10.1016/j.jallcom.2019.05.099

<https://hdl.handle.net/10356/144721>

<https://doi.org/10.1016/j.jallcom.2019.05.099>

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Improving microstructural and mechanical characteristics of cold-sprayed Inconel 718 deposits via local induction heat treatment

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Abstract

In order to improve the quality of cold sprayed Inconel 718 coatings, post deposition heat treatment is an essential procedure. However, traditional furnace heat treatment methods present disadvantages such as low efficiency and oxidation issues. In this study, we presented a novel strategy for post heat treatment of cold sprayed Inconel 718 coatings, by using eddy current to generate heat as well as electron migration. The as-sprayed and heat-treated Inconel 718 coatings were analyzed by using optical microscopy, scanning electron microscopy, X-ray diffraction and

transmission electron microscopy. The mechanical properties of the coatings were characterized by using three-point bending test. The results indicated that the as-sprayed Inconel 718 coating presented a high density of dislocations and $\{111\}$ $\langle 11-2 \rangle$ -type twin bands due to a high degree of plastic strain. The results also showed that the eddy current field could promote atomic motion and assist recovery of the severe plastically deformed splats, thus resulting in significant reduction in dislocation density within the cold sprayed IN718 coatings. Besides, induction heat treatment could increase the flexural strength and ductility of the cold sprayed IN718 coatings due to that eddy current promoted mass transport between the splats. Therefore, induction heat treatment is a potential method for locally heat treating the cold spray repair areas.

Keywords

Inconel 718; High pressure cold spray; Local induction heat treatment; eddy current field; Microstructure evolution; Mechanical properties

1. Introduction

Cold gas dynamic spray or simply ‘cold spray’ is an emerging technology for repair or additive manufacturing processes [1]. The basic principle of cold spray process is that the particles are accelerated by a high pressure preheated gas (e.g. nitrogen or helium) to a supersonic speed (e.g. 500-1000 m/s), and then the particles impact the substrate and adhere to the substrate surface. Subsequently, a thick and dense coating is built via layer-by-layer deposition with low or no oxidation [2]. On account of such low temperature input, the powder particles can retain solid steady during cold spray process and the substrate will not suffer from high temperature distortion and thermal stresses [3-5]. Inconel alloy 718 (hereinafter referred to as ‘IN718’) is a high-strength and corrosion-resistant nickel-based superalloy well suited for service in extreme environments subjected to high pressure and temperature. IN718 finds applications throughout various industries, including aerospace, marine & offshore, oil & gas and power generation [6-8]. Prolonged exposure to harsh working conditions often leads to severe degradation of these components due to thermo-mechanical fatigue, surface erosion, hot corrosion and oxidation. Instead of replacing an entire damaged part, it is both economically and resourcefully beneficial to locally repair the component [9].

Nevertheless, it has always been a challenge to achieve good quality of IN718 coating by using cold spray technique due to high critical velocities required and post deposition heat treatment methods are required [10-11, 43]. Wong et al. [10] reported that the post-annealing treatment could improve the ductility and tensile strength of cold sprayed IN718 coating. Levasseur et al. [11] studied the post-spraying sintering effects on microstructure evolution and mechanical properties, which showed that the pressureless sintering could improve the flexural strength and ductility of IN718 coating. Ma et al. [43] studied the effects of heat treatment (990 °C for 4 h) on cold sprayed

IN718 coatings and the results showed that both the adhesive and tensile strengths improved after heat treatment. However, the traditional furnace heat treatment is not possible to be used for locally heat treating the cold spray repair areas. Thus, a more effective and suitable heat treatment method is required to be better applied for cold spray repair applications. One of the solutions can be local induction heat treatment via eddy current. Shen et al. [13] reported that eddy current could promote electromigration, thus could be used to achieve the rapid formation of FeCrAl or β -FeAl coating layers within only 10 min. Current field effects were also studied in spark plasma sintering process [14-18], which showed that inter-diffusion between the particles could be accelerated by Joule heating and electromigration.

To evaluate the effects of eddy current field on the coating microstructure evolution and mechanical properties, IN718 coatings were post treated via both furnace heat treating and local induction heat treating. The microstructures and mechanical properties of the coatings were systematically investigated by using scanning electron microscopy, transmission electron microscopy, X-ray diffraction and three-point bending testing.

2. Materials and Methods

2.1 Materials

A commercial IN718 powder (25-45 μm in particle size) was used for cold spray deposition, which was produced via plasma atomization process (AP&C, Canada). The chemical compositions are Ni 53.0, Cr 18.7, Fe 18.51, Nb 4.92, Mo 3.00, Ti 0.97, Al 0.48, Mg 0.17, Cu 0.11, C 0.05, Si 0.03, Co 0.02, O 0.017, N 0.015 in weight percent, as provided by the manufacturer. The particle size distribution was measured by a laser assisted equipment ColdSprayMeter (CSM, Tecnar Automation Inc., Canada). Annealed cold rolled IN718 plates (50 mm $\times$ 50 mm $\times$ 3.2 mm in size,

provided by Top Alloys Pte Ltd, Singapore) were used as the substrates in this study. Prior to cold spray deposition, the substrate surfaces were ground by P320 SiC grit paper, and then polished by 9 μm and 3 μm diamond suspensions on polishing discs, respectively. The substrate surfaces were finally ultrasonically cleaned in an ethanol bath before the cold spray deposition.

2.2 Methods

2.2.1 Cold spray process

A high-pressure cold spray system (Impact Innovations GmbH, Germany) was used to do the deposition. N_2 was used as the propelling gas at 1000 $^\circ\text{C}$ and 4.5 MPa. The standoff distance between the spraying nozzle exit and the substrate surface was 30 mm and the spray gun was vertical to the substrate surface. The nozzle scanning speed was 500 mm/s with the feed rate of IN718 powder being around 46 g/min [12, 37-38]. The number of deposition layers over a substrate was 10, which led to a final coating thickness of about 1.3 mm with a thickness increment of about 0.13 mm per layer.

The cold spray parameters were selected by using the commercial software package from Kinetic Spray Solutions (KSS, Germany) - in such a way to obtain a velocity ratio ($\eta = v_p/v_{crit}$, v_p is particle velocity, v_{crit} is particle critical velocity) within the window of deposition [20]. The calculated particle velocities were cross-checked by velocity measurements using a cold spray velocimeter.

2.2.2 Heat treatment process

The as-sprayed IN718 samples were placed underneath a copper coil inside an induction heat treatment (IHT) system with a high vacuum environment (GH Induction Atmospheres, USA). The distance between the copper coil and the IN718 coating surface was around 3 mm. Alternating

current (AC) was passed through the copper coil to produce an alternating magnetic field in and around the coil; by means of this, eddy currents were induced in the IN718 coatings. The frequency of the current was 107 KHz and the current density was around $1.22 \times 10^5 \text{ A/m}^2$. The skin depth could be derived from a practical formula as follows [21]:

$$\delta = \sqrt{\frac{2\rho}{(2\pi f)(\mu_0\mu_r)}} \approx 503 \sqrt{\frac{\rho}{\mu_r f}} \quad (1)$$

where δ is the skin depth in meters, μ_r is the relative permeability of the medium ($\mu_r = 1.0011$ for IN718), ρ is the resistivity of the medium in $\Omega \cdot \text{m}$ ($\rho = 1.25 \times 10^{-6} \Omega \cdot \text{m}$ for IN718), and f is the frequency of the current in Hz.

From Eq.1, the calculated skin depth was around 1.7 mm. Surface temperature of the IN718 samples was around 900 °C, as measured by laser thermometer and calibrated by thermocouples. The duration time was 10 mins and cooled down with argon protection. For comparison, traditional furnace heat treatment (FHT) was carried out via a vacuum tube furnace (Lenton, UK) with the vacuum level of 4×10^{-5} Torr. The temperature and duration of FHT is the same with IHT.

2.2.3 Characterization

Scanning electron microscopy (SEM, JEOL-JSM-5600LV, Japan) was used to observe the as-received IN718 powder and the coating surface morphologies. Optical microscopy (Zeiss Axioskop 2, USA) was used to analyze the cross-sectional microstructures of the IN718 coatings. ImageJ software was used to calculate the coating porosity levels, which was guided by ASTM E2109-01. Electron backscatter diffraction (EBSD, JEOL-7600F with Oxford EBSD detector) was taken by using a 200 nm step size at 15 keV to analyze the grain shapes and grain sizes. X-ray diffraction (XRD) was performed to analyze the crystallographic phases, crystallite size and

microstrain by using PANalytical Empyrean equipment which used Cu K α as the X-ray source. The scan speed was 1°/min with a scan step of 0.02° and the scan range was from 30° to 90°. Transmission electron microscopy (TEM, JOEL JEM-2010 with Oxford EDS) was used to analyze the coating microstructures and chemical compositions in high magnification. The TEM lamella were prepared by focused ion beam (FIB, ZEISS Crossbeam 540, Germany) milling.

In order to investigate the IN718 coating flexural strength, MTS 810 Material Testing System was used to carry out the three-point bending test. The samples used for bending test were 50 mm $\times$ 10 mm $\times$ 4.2 mm and the loading rate was 0.5 mm/s until failure occurred [37]. Three samples were repeated for each condition. The sample fracture surfaces were further analyzed by SEM to understand the fracture mechanisms.

3. Results and discussions

3.1 Microstructure characterization

The micrograph of the IN718 powder obtained from SEM is shown in Fig. 1(a). The powder particles are spherical in shape and the particle surfaces are smooth without satellite particles attached, indicating superior flowability of the particles during cold spray process. The IN718 particle size distribution is displayed in Fig. 1(b), which shows that the particle size is 20 μ m to 45 μ m and the average particle size is around 32 μ m. Fig. 1(c) represents the Inverse Pole Figure (IPF) micrograph of the IN718 particles obtained from EBSD. It is clear that the individual particles contain a number of randomly oriented grains. The grain size distribution in the as-received powder is shown in Fig. 1(d), which indicates that most of the grains are 1~4 μ m. However, there are also some large grains (10~25 μ m) within the particles, which is probably attributed to the quenching effect during plasma atomization process of powder fabrication [19].

Fig. 1 (a) SEM micrograph of IN718 feedstock powder, (b) IN718 powder size distribution, (c) inverse pole figure map of as-received IN718 powder, (d) grain size distribution in as-received

IN718 powder, (e) section of IN718 lamella cut out from a single IN718 particle and (f) TEM bright-field image of IN718 particle showing a large strain-free crystalline region with a low dislocation density.

Fig. 1(e) shows TEM sample preparation from an IN718 single particle using a focused ion beam (FIB) milling to cut out the lamella. Fig. 1(f) shows a bright-field TEM image of the as-received IN718 particle. It can be seen that the sample has a large strain-free crystalline region with a low dislocation density and the inset selection area diffraction (SAD) pattern shows that it comprises of γ phase. No other phases are found in the as-received IN718 particle especially the strengthening intermetallic phases due to the quenching effect during the powder fabrication process [19].

The particle velocity distribution is demonstrated in Fig. 2(a), which shows that most of the particle velocities fall within the range from 600 to 800 m/s and the average particle velocity is around 713 m/s. Particles travelling at or above critical velocity will result in deposition and coating formation. The critical velocity and deposition window in this study is calculated using the KSS software and the results are presented in Fig. 2(b). If the particle velocity is below the ‘critical velocity’, the particle plastic deformation is too low to cause bonding. However, if the particle velocity is above the ‘erosion velocity’, hydrodynamic penetration leads to strong erosion. If the particle velocity is between these two characteristic velocities, optimum conditions for deposition are reached. In this study, the particle velocities are found to fall within the window of deposition which implies a high deposition efficiency. For particles with the sizes of 25 μm , 32 μm and 46 μm , the particle temperatures are 640 °C, 657 °C and 616 °C, respectively.

Fig. 2 (a) IN718 powder particle velocity distribution measured by CSM and (b) window of deposition for the IN718 particles and calculated particle velocities, particle temperatures and η values for different particle sizes (i.e. 25 μm , 32 μm and 46 μm).

A representative microstructure of the as-sprayed coating cross-section from this study is shown in Fig. 3(a). The as-sprayed IN718 coating porosity level is around 2.5% and the irregular pores are relatively homogeneously distributed across the coating. After FHT, the irregular micropores change into rounded pores as shown in Fig. 3(b) and the coating porosity level does not change obviously. These results are in line with those achieved by Wong et al [10], who found that the cold-sprayed IN718 coating porosity levels remained relatively similar after heat treatment at 950 °C, 1010 °C and 1060 °C for 2 hours. However, the porosity is significantly reduced and the microcracks are in particular found to be almost totally healed after IHT. Significant consolidation, therefore, occurs during the induction heat treatment process, as shown in Fig. 3(c). It is noticeable the coating porosity level decreases to 0.2% after only 10 mins of hold at 900 °C under the copper induction coil in the bell-jar system. Comparing the coating microstructures after IHT and FHT, although the surface temperature is the same for both cases, IHT additionally induces eddy currents within the coatings. Fig. 3(d) shows the schematic illustration of the eddy currents flowing through

the splat/splat interfaces, the currents are forced through the narrow contact areas between the splats, thus resulting in simultaneous high current densities and temperatures at the narrow contact areas. The effect of an electric field on mass transport can be evaluated from the electromigration theory [22]:

$$J_i = -\frac{D_i C_i}{RT} \left[\frac{RT \partial \ln C_i}{\partial x} + F z^* E \right] \quad (2)$$

where J_i is the flux of diffusing i_{th} species, D_i is the diffusivity of the species, C_i is the concentration of the species, F is Faraday's constant, z^* is the effective charge on the diffusing species, E is the current field, R is the gas constant, and T is the temperature.

It can be seen that current field can contribute to the mass transport and material flux between the splats. Besides, Trzaska et al. [14] have proved that electric current triggered the diffusion and densification of TiAl powder particles during spark plasma sintering process. They also observed that pronounced increases in dislocation and twin densities in the neck regions with respect to the centers of the particles, which showed that intense plastic deformation occurred in the neck regions and that the particle centers were less affected by this electric current [14].

After IHT, the coating/substrate bonding at the interface is more intimate to the extent that the interface line is indistinguishable as shown in Fig. 3(c), indicating superior adhesion strength of the coating. On the other hand, the coating/substrate interface following FHT still shows some microcracks at the selected sites in Fig. 3(b). The reason for this is believed to be the enhanced atomic diffusion of the constituent elements across the coating/substrate interface is significantly promoted by the applied eddy current field.

Fig. 3 (a-c) Optical micrographs showing microstructures of cold sprayed IN718 coatings: (a) as-sprayed, (b) furnace-heated at 900 °C for 10 mins, and (c) induction-heated at 900 °C for 10 mins and (d) schematic illustration of the eddy current flowing through deformed particles.

The surface morphologies of the as-sprayed and heat-treated IN718 coatings are subsequently observed by SEM, which are shown in Fig. 4. From Fig. 4(b & d), the unbonded interparticle regions are obvious for the as-sprayed and furnace heat treated coatings. However, the induction heat treated coating (Fig. 4(f)) shows that interparticle regions are strongly bonded. This is a clear evidence that the IHT causes rapid and efficient electromigration-assisted material diffusion across the splat/splat interfaces. This enhanced diffusion within the interparticle regions decreases the coating porosity level and potentially improves the cohesive strength of the coatings.

Fig. 4 SEM micrographs showing surface morphologies of cold sprayed IN718 coatings: (a & b) as-sprayed, (c & d) furnace-heated, and (e & f) induction-heated. The circled areas in (b, d & f) show the separated and bonded particles in the coatings.

3.2 X-ray diffraction analysis

The XRD analyses were carried out for the powder and subsequently as-sprayed and heat-treated coatings. The XRD profiles are shown in Fig. 5(a). The three peaks are identified as (111), (200) and (220) reflections from single-phase FCC γ Ni-solid solution structure for both the IN718 powder and IN718 coatings. Compared to the IN718 powder peaks, the as-sprayed coating peaks show significant peak broadening, indicating the presence of higher microstrain in the coating structure [24]. After heat treatment, the peaks become shaper due to residual stress relaxation

within the coatings. A modified Williamson-Hall (W-H) method is used to extract the average crystallite size and amount of microstrain from the XRD fitting curves as shown in Fig. 5(b). The modified W-H equation is expressed in the form of [24]:

$$\Delta K = \frac{0.9}{D_s} + \varepsilon K C^{\frac{1}{2}} \quad (3)$$

where, D_s is the average crystallite size, ε is the average microstrain, $K = \frac{2\sin\theta}{\lambda}$ and $\Delta K = \frac{(2\Delta\theta)\cos\theta}{\lambda}$, θ is the Bragg's angle of diffraction, $\Delta\theta$ is half of the full-width-half-maxima (FWHM) of the diffraction peak, λ is the X-Ray wavelength and C is the average contrast factor for a particular diffraction peak [23]. The intercept and slope of the plot determines the crystallite size and presence of microstrain in the material, which are shown in Table 1.

On the other hand, microstrain ε is mainly induced by dislocation, from which dislocation density can be calculated by following formula [25],

$$\varepsilon = (BM^2)^{\frac{1}{2}}\rho^{\frac{1}{2}} \quad (4)$$

where, $B = \frac{\pi b^2}{2}$, b is the Burgers vector (for typical $a/2\langle 110 \rangle$ dislocations on $\{111\}$ slip planes in IN718, $b = 0.25$ nm) as discussed by Syarif et al. [26], M is a constant (taken to be 1.5 [25]) which is related to the effective dislocation cut-off radius R_e and dislocation density ρ [25]. The calculated dislocation densities for the powder and cold sprayed coatings are shown in Table 1.

Fig. 5 (a) XRD patterns of IN718 powder and IN718 coatings at the state of as-sprayed, furnace-heated and induction heated at 900 °C for 10 mins showing a single-phase FCC solid solution and (b) a modified W-H plot illustrating the relationship between microstrain and crystallite size.

A steeper slope of the modified W-H plot indicates that the as-sprayed IN718 coatings contain a significant amount of microstrain due to large plastic deformation. This microstrain is related to the presence of defects, particularly dislocations [27] which are nucleated during the cold spray deposition process. Table 1 shows that the dislocation densities for the IN718 feedstock powder and as-sprayed IN718 coating are $2.9 \times 10^{14} \text{ m}^{-2}$ and $3.7 \times 10^{14} \text{ m}^{-2}$, respectively. The average crystallite size of the as-sprayed coating is around 46 nm, which is smaller than that in the as-received powder, i.e. ~67 nm. The reduced crystallite size in the as-sprayed IN718 coating is a consequence of the severe plastic deformation that occurs in the powder particle upon impact the substrate surface during the cold spray process. Presence of smaller crystallites and a sizable microstrain indicates the formation of sub-grains in the severely deformed microstructure of the individual splat in the coating [28].

After FHT, the dislocation densities reduce from $1.3 \times 10^{15} \text{ m}^{-2}$ to $3.7 \times 10^{14} \text{ m}^{-2}$ which is indicative of initiation of recovery process in the coating. As a direct consequence of this, the crystallite size is observed to increase to $\sim 113 \text{ nm}$. After IHT, the dislocation densities further reduce to $4.1 \times 10^{13} \text{ m}^{-2}$ with the least microstrain. Therefore, by comparison to FHT, eddy current field in IHT promotes a higher degree of relaxation of the microstrain possibly through recovery mechanisms such as dislocation annihilation and polygonization also subsequent growth of the crystallite [41]. The crystallite size obtained from the W-H plot is in agreement with the dislocation cell size (defect free regions bounded by dislocation walls) obtained from the analysis of the TEM images of the as-deposited coatings.

Table 1 Crystallite size, microstrain and dislocation density of IN718 powder and coatings as calculated from the W-H plot shown in Fig. 5b.

IN718	Crystallite size (nm)	Microstrain	Dislocation density (m^{-2})
As-received powder	~ 67	8×10^{-3}	2.9×10^{14}
As-sprayed coating	~ 46	1.7×10^{-2}	1.3×10^{15}
IHT coating	~ 157	3×10^{-3}	4.1×10^{13}
FHT coating	~ 113	9×10^{-3}	3.7×10^{14}

3.3 TEM analysis

3.3.1. As-sprayed IN718 coating

Fig. 6(a) shows a TEM bright field image of a splat within the as-sprayed IN718 coating. Firstly, as an observation of the phases that are present in the microstructure reveals that the coating comprises simply of the γ -grains and interestingly no strengthening intermetallic phases like the cubic $\text{L}_{12}\text{-}\gamma'$ (Ni_3Al) or metastable tetragonal γ'' (Ni_3Nb); even the stable DO_{18} orthorhombic δ -

Ni₃Nb precipitates are absent. The absence of these major strengthening precipitates could be traced back to the original microstructure of the powders and how they were synthesized in the first place. The IN718 powder used for spraying is synthesized by plasma atomization technique where wire feedstock is fed into multiple plasma torches under argon gas environment and the molten droplets are rapidly atomized as spherical powders - the quenching rate experienced by molten metal as it solidifies is 10^5 °C/s [19]. Under such extraordinarily high quenching rates, no precipitates would be expected to form in IN718 powder. In a systematic study carried out by Geng et al. [29], it had been clearly shown that no precipitates resulted during water quenching of IN718 from solutioning temperature of 1100 °C and this also simultaneously resulted in a much-reduced hardness of the alloy in absence of the strengthening phases, primarily the γ'' precipitates.

As a result of the extremely high stresses induced within the as-sprayed coating, as evident from the residual stress analysis previously [40], a large amount of strain contrast is visible in the TEM images. The heavily streaked reflections in the selected area diffraction (SAD) pattern (obtained from the area) of the near $\langle 110 \rangle$ -zone axis is also a testimonial to the high degree of strain in the sectioned thin foil. The corresponding dark field image is provided in the inset of Fig. 6(a), which shows that the coating microstructure is the very high density of dislocations present in the sample that result from the severe plastic deformation experienced within individual spats of the as-sprayed coating to the extent that resolving individual dislocations within this sample is nearly impossible. In addition, Fig. 6(d) shows an example of formation of the dislocation wall and sub-grain boundary due to high dislocation densities and the SAD patterns obtained from the region C and D are inserted in Fig. 6(d).

Besides this large density of dislocations, another important feature is the occurrence of narrow twin bands as shown in Fig. 6(b). These twins are characterized as the conventional $\{111\} \langle 11\bar{2} \rangle$ -type. The twin bands are around ~ 10 nm wide and can be substantially long with the one imaged in Fig. 6(b) is approximately $> 2.1 \mu\text{m}$, which is comparable to the grain size within the splat. From the microstructural observations, the twins almost invariably nucleate from the grain boundaries and once they initiate in a particular grain, it is not unreasonable to assume that they traverse across the whole grain. Thus, in addition to the generation of high density of geometrically necessary dislocations within the splats during the impact, formation of twins also becomes crucial in order to accommodate the high amount of plastic strain. Considering plastic deformation on $\{111\}$ planes during the impact of the particles, the dislocations are likely to be the $a/2\langle 1\bar{1}0 \rangle$ type. A closer look at the twinned grain, Fig. 6(c) reveals a set of parallel slip traces. When looking down the $\langle 110 \rangle$ zone axis the angle between these slip traces and the twin interface is $\sim 54^\circ$ as shown in Fig. 6(e). Considering the twin interface is $\{1\bar{1}1\}$ -type plane, these slip traces are parallel to $\{001\}$ planes. Also, interestingly, the slip traces are imaged to be at equal distances. This suggests that slip activities also occur on $\{001\}$ planes apart from dislocation nucleation and glide on $\{111\}$ planes in the splats. The slip traces result from a series of dislocations traversing on parallel $\{001\}$ planes at regular spacing. This is clear that high local stresses result in cross-slip of the regular spacing.

Fig. 6 TEM micrographs showing (a) a field image of splat microstructure with a dark field image in the insert, (b) deformation twins, (c) a set of parallel slip traces, (d) an example of formation of a sub-grain, where SAD diffraction patterns correspond to Regions C and D, and (e) the angle between these slip traces and the twin interface.

3.3.2 Furnace heat treatment at 900 °C

Fig. 7 shows the TEM images of the IN718 coating after FHT. Compared to the as-sprayed coating microstructure, the FHT coating is characterized by the presence of larger grains and a significant reduction in dislocation density, indicating some recovery has occurred in the microstructure during FHT. Clearly, the dislocation structure could be resolved via imaging in proper diffraction conditions as seen in Fig. 7(a & b). Some reordering of the dislocations into cell networks is evident in Fig. 7(b) when viewed near the $[111]$ zone axis. Some of the grains show a high density of dislocations (Fig. 7(b)) while a number of small grains could be observed to have nucleated at

the grain boundaries (Fig. 7(c)). These clusters of fine grains are typically <50 nm in sizes. These are strain-free recrystallized grains that nucleate as a result of rearrangement of dislocations into small networks as seen in Fig. 7(a) and eventually these networks, through accommodation of excess dislocations, result in grain boundary misorientation angles in exceeding 15° (the generally accepted criterion for a high angle boundary) to form distinct grains. As the dislocations rearrange to form these recrystallized grains, there is a natural reduction in the dislocation density that results in the networks to be identified easily in TEM – this theory also agrees well with the observation from XRD results whereby an almost an order of magnitude reduction in the measured dislocation density is noticed following furnace heat treatment of the as-sprayed coating.

Besides this gross rearrangement of dislocations from a high density in the as-sprayed condition, another interesting observation is the growth of the nano-twins as seen in the as-sprayed condition - coarse $\{111\} \langle 11-2 \rangle$ twin bands are visible in the heat-treated sample. The growth mechanism of these nano-twins as observed in Fig. 6(b & c) upon annealing has been caught in act in Fig. 7(e). This clearly shows that the twins grow by the ledge mechanism [30]. It is envisaged the annealing twins in FCC metals alloys form due to growth accidents on $\{111\}$ propagating steps present on migrating grain boundaries. As a result of the accidents, Shockley partials are generated contiguous to grain boundaries. These partials repel each other and glide away from the boundary to produce a twin [30, 42]. The lateral thickening of the twin bands occurs as the coherent twin boundary (CTB), i.e. $\{111\}$ plane, is pushed out by the progression of the incoherent twin boundary (ITB). The ITB generally progresses by the glide of Shockley partials on parallel $\{111\}$ planes [30, 42]. With the aid of thermal energy, high density of stored dislocations on the $\{111\}$ planes in the as-sprayed coating can easily glide on the parallel slip planes at 900°C . The propagation of an ITB is essentially halted at the grain boundaries as seen in the Fig. 7(e) when the twin band attains its

equilibrium thickness – any further lateral coarsening of the twin would require a new ledge to be nucleated.

The microstructure in FHT condition is also additionally characterized by the occurrence of δ -Ni₃Nb precipitates – these precipitates typically have blocky, disc-like morphology. One such precipitate was imaged near the $[010]_{\delta}$ zone axis with a diameter of ~350 nm as shown in Fig. 7(f). Precipitation in this condition tends to be localized at pre-existing grain boundaries. As seen in Fig. 7(f), the δ precipitate has a flat and coherent interface with the matrix. From the $[010]$ diffraction pattern, it is obvious that the interface is parallel to the $[001]$ direction (i.e. the c-axis of the precipitate). Also, a series of planar fault lines are observed along the c-axis of the precipitate. These precipitates are almost all observed to form at the γ -grain boundaries after annealing. The precipitation of δ -Ni₃Nb phase in IN718 occurs at around 900 °C, which has been confirmed by differential scanning calorimetry results [31]. Annealing at 900 °C previously has also shown precipitation of the δ at grain boundaries, however, aging for 20h led to profuse precipitation as long, needle-shaped δ within the grains too [32].

Fig. 7 TEM micrographs showing (a & b) bright field images of dislocation structure that can be resolved via imaging in proper diffraction conditions with SAD pattern illustrated in (a), (c) recrystallized grains, (d) thicker deformation twins, (e) twins grown by the ledge mechanism, and (f) a precipitate (δ -Ni₃Nb) and its corresponding SAD pattern.

3.3.3 Induction heat treatment at 900 °C

Fig. 8 shows the TEM images of the IN718 coating after IHT. In this condition, the microstructure is generally characterized by the presence of larger grain structures and also by a significant reduction in dislocation density, indicating that significant recovery has occurred in the microstructure during IHT. In comparison to the regular furnace heating where the heating is through radiation in the furnace chamber, IHT involves heating through resistive Joule heating. IHT comprises of a cumulative effect of resistive Joule heating and the imposed eddy currents.

Based on this, an analogy of the induction heating process could be drawn with spark plasma sintering (SPS) process [14-15, 22]. However, it is to be noted that SPS is typically employed to compact powder aggregates and is assisted by external loading. But in the present case of induction heating, no external loading is applied, so the prospect of mechanical compaction of the porous coating structure can be discounted. Hence, in this case, the thermally assisted processes play a more critical role in densification of the coating. In SPS, Joule heating and current field are also believed to be the major contributors to the mass diffusion that result in sample densification. Mass flux due to electromigration, which is forced atomic diffusion process with the driving force arising from an electric field and associated electric current in metals, has also been identified to have a significant role in densification [33]. As in SPS, the electric field can provide an additional driving force for atomic diffusion by electromigration that fuses the splats together. However, no direct correlation of densification, so far, has been drawn with regards to Joule heating associated with IHT. From the microstructure, it is clear that IHT promotes processes such as grain growth and thermally assisted dislocation motion within the splats. The eddy current field assisted annealing causes significant recovery of the severe plastically deformed splats through (i) annihilation of stored geometrically necessary dislocations, (ii) recrystallization of strain-free grains. Besides the TEM microstructures, the former XRD results also demonstrate that a reduction of dislocation density by two orders of magnitude is noticed in comparison to the as-sprayed coating. At 900 °C, besides annihilation of dislocations with opposite Burgers vector on glide planes, non-conservative movement of dislocation such as climb process is also possible under the effect of electric field. Such drastic reduction in the dislocation density would entail a natural reduction in the residual stresses both in the coating as well as the coating/substrate interface. Besides recovery of

dislocation as already seen in the furnace heat-treated samples, formation of recrystallized grain is also seen in the microstructure.

Also, another interesting finding is the very fine distribution of precipitates in grain interiors, as shown by white arrows in Fig. 8 (b & c), besides the usual precipitation localized at pre-existing grain boundaries. The grain boundary precipitates are identified from their diffraction patterns to be the δ -phase. The EDS results of the precipitates as shown in Fig. 8(e) also reveal a higher Nb and Ni content than surrounding matrix and hence could be attributed to the δ phase. These fine precipitates are probably attributed to the eddy electric field in line with similar observations made in SPS [34, 35]. In SPS, the free energy forming critical nuclei size in the electric field is given as [36]:

$$\Delta G_c = \frac{16\pi\sigma^3}{3[|\Delta G_V| + \frac{1}{2}E^2(\varepsilon_1 - \varepsilon_2)]^2} \quad (5)$$

where σ is interfacial energy, ΔG_V is driving free energy, E is electric field, ε_1 and ε_2 are the dielectric constants of the matrix and precipitating phase, respectively. Obviously, electric field can promote the precipitating by reducing the free energy needed to form critical nuclei [36]. The field-assisted heating additionally results in the δ precipitation from the supersaturated Ni-based matrix – while conventional heat treatment is unable to relieve this supersaturation. Although the exact sites of nucleation of these precipitates could not be exactly determined, with the high dislocation density in the parent coating and elevated temperature, short-circuit diffusion of the solute atoms through the dislocation cores could be a possible mechanism. Since the major strengthening phases in IN718 are the γ'' and δ phases [30-32], it is anticipated that significant strengthening of the coating could result through these profuse precipitates following IHT in

comparison to the as-sprayed coating. Also, these precipitates would be the potent pinning sources to dislocation motion through the matrix.

Fig. 8 (a-c) TEM bright field images of fine precipitates (δ -Ni₃Nb) in the interior of grains as indicated by the arrows, (d) TEM image showing twin bands, and (e) EDS results of the fine precipitates and surrounding matrix.

3.4 Mechanical properties

Fig. 9(a) schematically illustrates the three-point bending test configuration used in this study and Fig. 9(b) shows the representative stress-strain curves. The coating flexural strength is represented by the maximum stress experienced by the coating before it cracks. In this study, it was found that the coating cracks initiated from the coating bottom mid-point due to the maximum tension stress there, which propagated upwards through the coating, leading to coating failure. From Fig. 9(b),

the as-sprayed IN718 sample can survive under ~ 540 MPa and the furnace heat-treated sample can survive until ~ 923 MPa. Interestingly, the induction heat-treated sample can survive till the highest strength (i.e. ~ 1215 MPa) before the coating cracking and ultimate failure. Thus, it can be deduced that the splat/splat cohesive strength is significantly improved after IHT, which is probably due to the release of strain and improvement of diffusion between splats. Fig. 9(b) also shows that the as-sprayed coating fails abruptly, which indicates that the as-sprayed coating is brittle and fractures without elongation. However, ductility of the coatings improves significantly after IHT.

Fig. 9 (a) Schematic illustration of the three-point bending test configuration used in this study and (b) stress-strain curves of as-sprayed and heat treated IN718 coated samples.

Fig. 10 shows the coating fracture surface morphologies after three-point bending tests. For the as-sprayed coating, fracture apparently occurs between the splat/splat interface with a clean break-out of the individual particles, leaving smooth and faceted surfaces (red arrow in Fig. 10(a)), which represents more of a brittle-like fracture behavior. It is likely that the de-cohesive fracture occurs in the as-sprayed IN718 coating since the cohesive strength of splat/splat is relatively weak with a significant amount of residual stress remnant in the as-sprayed coating layer. The nature of the in-

plane or out-of-plane residual stresses also play an important role in determining the overall strength of the coating layer with the underlying substrate [39]. The out-of-plane residual stress at the interface of a cold-sprayed layer is tensile in nature while the in-plane component is compressive [39]. Thus, the out-of-plane tensile normal component assists in the de-bonding of the coated layer that eventually results in an intergranular (or intersplat)-type fracture. After FHT, as shown in Fig. 10(b) the fracture surface shows brittle facets (red arrow) and some ductile-type dimples and river-pattern marks, which implies the improvement of coating cohesive strength and ductility at the interface. However, the coating still fractures at the splat/splat interface and this failure can still be considered as a de-cohesive rupture. In comparison to the furnace heat-treated sample, the induction heat-treated sample shows plenty of dimples uniformly spread at the fracture surface (Fig. 10(c)). Compared to the as-sprayed IN718 coating, it can be seen that splat/splat interface becomes less visible in induction heat-treated coating due to diffusion-assisted bonding. The overall fracture surface indicates a characteristic intragranular (or intra-splat) fracture with a rough dimpled surface (yellow dash arrow). This, besides the cross-section microstructural observations, also suggests a great improvement of cohesive strength as well as coating ductility is brought about by the diffusion of the splats with overall reduction in the porosity levels.

Fig. 10 SEM micrographs showing fractured morphologies of (a) as-sprayed sample, (b) furnace heat-treated sample, and (c) induction heat-treated sample.

3.5 Proposed mechanism

Based on the results of microstructures, XRD and TEM analyses and mechanical properties, a plausible mechanism for microstructure evolution of IN718 coating during cold spray deposition and after FHT and IHT processes is proposed. Initially, a supersonic stream of spherical IN718 particles with a given size distribution and a moderate η value approaches IN718 substrate. Subsequent layers are built up to form the as-sprayed IN718 coating. Concurrently, a very high dislocation density is present in the as-sprayed coating that result from the severe plastic deformation and strengthening precipitates are absent. Besides, the grains become smaller due to the subsequently particle impingement and dislocation accumulation. In addition, formation of twins also becomes crucial in order to accommodate the high amount of plastic strain.

After FHT, the grains become larger and the dislocation density reduces due to some recovery has occurred in the microstructure during FHT. Besides, the dislocations reorder into cell networks and form some recrystallized sub-grains. Moreover, twin bands grow thicker by the ledge mechanism, which occurs as the coherent twin boundary is pushed out by the progression of the incoherent twin boundary by the glide of Shockley partials on parallel $\{111\}$. Additionally, δ - Ni_3Nb precipitates form in the IN718 coating, which have flat and coherent interfaces with the matrix. From the $[010]$ diffraction pattern, it is obvious that the interface is parallel to the $[001]$ direction.

After IHT, the IN718 coating porosity significantly reduces and the coating strength is improved, which is because the eddy current field promotes atomic motion and material flux at splat/splat interfaces. The microstructure is characterized by the presence of larger grains and a significant reduction in dislocation density and significant recovery of the severe plastically deformed splats.

The eddy current field assisted heating additionally results in the fine δ precipitates from the supersaturated Ni-based matrix due to the reason that electric field can reduce the free energy needed to form critical nuclei.

While the proposed model adequately describes the IN718 coating microstructure evolution during cold spray deposition and HT in the eddy current field, its applicability to other materials is not straightforward as the material behavior in the eddy current field is governed by different material properties such as conductivity, magnetic permeability etc. Further study involving different materials and different process parameters such as current densities and temperatures will lead to a more universal model for eddy current effects on cold sprayed coatings.

4. Conclusions

In this study, IN718 coatings were successfully coated on IN718 substrates via a high-pressure cold spray process. A novel local induction heat treatment (IHT) method was applied to heat treat the cold sprayed IN718 coatings. For comparison, traditional furnace heat treatment (FHT) was also used at the same temperature for the same duration. The effects of eddy current were investigated by analyzing the coating microstructures using optical microscopy, XRD and TEM and measuring the coating mechanical properties with three-point bending test.

- The as-sprayed IN718 coating presented high density of dislocations and $\{111\} \langle 11-2 \rangle$ -type twin bands due to a high degree of plastic strain. The twin bands were around 10 nm wide and could be substantially long.
- After FHT, the IN718 coatings presented larger grains and a significant reduction in dislocation density. Besides, the FHT IN718 coatings showed that the twins grew thicker through the ledge mechanism and the occurrence of δ -Ni₃Nb precipitates.

- During IHT, the eddy current field assisted annealing caused significant recovery of the severe plastically deformed splats. Besides, the eddy current could promote the atomic motion and resulted in more significant reduction in dislocation density within the cold sprayed IN718 coatings. The eddy current also promoted the δ -Ni₃Nb precipitation by reducing the free energy needed to form critical nuclei, resulting in the formation of fine precipitates in the interior of grains.
- IHT could increase the flexural strength and ductility of the cold sprayed IN718 coatings due to that the eddy current promoted mass transport between the particles.

Acknowledgements

This work was financially supported by the National Research Foundation of Singapore (NRF), Rolls-Royce (RR), and Nanyang Technological University (NTU), Singapore, with the research grant number of M-RT3.1. Wen Sun was grateful for the Ph.D. scholarship from the Rolls-Royce @ NTU Corporate Lab and the Nanyang Technological University, Singapore.

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