

Grinding- and robotic hammer peening-induced modifications in the near-surface regions of laser-cladded Inconel 718 coatings

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Abstract: Inconel 718 coatings deposited by laser-cladding (LC) on low-alloy steel substrate have been grinded followed by robotic hammer peening (RHP). White layer consisted of nanograins, smaller than 100 nm, has been observed on the surface accompanied with a deformation layer underneath after grinding and RHP. By increasing the RHP intensity (i.e., both the impact energy and the dent overlap), the residual tensile stress measured by X-ray diffraction from the surface of the coating has been monotonically reduced and eventually converted to residual compressive stress with increased in-plane anisotropy, which was accompanied with monotonically increased surface hardening up to 40.5 HRC. Grain deformations have been induced in the white- and deformation-layer, they are dominated by twinning and slipping, respectively. In terms of depth-dependent distributions of low-angle ($< 15^\circ$) grain boundaries and hardening, the effective cold-working thickness induced by the RHP process is over 1000 μm , which is close to the thickness of a single-pass LC deposition. Onset of porosity closures occurred upon the RHP process, especially in the near-surface regions. These findings shed light on integrity enhancement for additive manufacturing of metal alloys by LC-based techniques through introducing interpass RHP process.

Keywords: Laser cladding; Additive manufacturing; Peening; Protective coating; Plastic Deformation, Integrity Enhancement; Inconel 718; White Layer

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1. INTRODUCTION

Inconel 718 is a typical Ni-based superalloy that has excellent mechanical performance for continuously serving, e.g., as aircraft engine components, at temperatures up to 650 °C. The combination of corrosion resistance and weldability, along with the excellent creep-rupture strength at elevated temperatures, makes Inconel 718 an excellent choice for surface finishing applications in various areas including aerospace, automotive, food service, housewares, etc. Laser-cladding (LC) is one of the most advanced direct energy deposition methods that has been extensively investigated in the past two decades.¹⁻⁵ By this technique, additive manufacturing (AM) of Inconel 718-based structural components,⁶⁻⁷ e.g., turbine blades,⁸⁻⁹ has now been entering industrial applications. However, post-AM treatments, e.g., heat-treatment to release the residual tensile stress, are still necessary at the current stage.¹⁰⁻¹³ A lot of efforts have been made in recent years to reduce the necessity of post-AM treatments. For example, S. Sui et al. have tried introducing in-situ laser heat-treatment for regulating the Laves phase in LC-deposited Inconel 718 coatings.¹⁴ Y. Kong et al. have investigated the effect of in-situ cyclic heat-treatment on microstructural and hardness properties of LC-deposited metallic components.¹⁵

Instead of in-situ heat-treatment by lasers during AM process, surface integrity enhancement has been proposed for modifying the microstructures of metallic components through monolithic integrations between AM and cold-working, e.g., rolling and peening.¹⁶⁻¹⁹ The general function of cold-working is to introduce plastic deformation in the surface and near-surface regions of the workpiece that, in turn, induce residual compressive stress, grain refinements, pore closures, and hardness enhancement. Lesyk et

al. have studied the effect of barrel finishing, shot peening, ultrasonic shot peening, and ultrasonic impact treatment on surface texture, roughness, hardness, porosity, and residual stress of Inconel 718 deposited by selective area melting.²⁰ They also compared the effect of ultrasonic impact on Inconel 718 prepared by hot isostatic pressing and laser powder bed fusion was also studied.²¹ Amanov et al. have studied the effect of ultrasonic nanocrystal surface modification on Inconel 718 prepared by laser powder-based direct energy deposition to improve its mechanical properties and fretting wear behavior.²² Likewise, Bianchetti et al. have studied the effect of shot peening on fatigue life improvement of Inconel 718 manufactured by laser-powder bed fusion.²³ By introducing interpass cold-working process into AM, the integrity of the entire AM component can be effectively enhanced in a layer-by-layer strategy. For example, J. Hönnige et al. have introduced interpass rolling into AM of Inconel 718 coatings by wire-arc method and addressed the rolling effect on residual tensile stress and microstructural evolutions.¹⁶ In a similar way, W Kan et al. have studied the effect of interpass rolling on microstructural and mechanical properties as well as corrosion resistance of LC-deposited stainless steel (SS) 316L.¹⁷ M. P. Sealy et al. have studied the effect of interpass laser-shock peening (LSP), an advanced laser-based cold-working process,²⁴⁻²⁵ on the global integrity of SS420 directly deposited by laser engineered net shaping (LENS).¹⁸ Distinct from rolling and LSP, robotic hammer peening (RHP) is a dynamic cold-working process besides shot peening that has long been developed for surface enhancements of metallic components in manufacturing and remanufacturing.²⁶⁻²⁸ Because of the process compatibility between RHP and LC on either robotic or CNC (computer numerical control) platform, RHP process has been introduced into LC of Ti-alloy to disrupt the interlayer epitaxial grain

growth, which greatly enhanced the mechanical performance.¹⁹ In comparison, the effect of RHP process on LC-based AM of Inconel 718 is still lacking in the literature.²⁹

In this work, we investigated RHP process on Inconel 718 coatings deposited by LC and addressed the effect of RHP intensity on the evolutions of residual tensile stress, micro hardness, and microstructural properties of the coatings by various characterization techniques. Both the LC and the RHP processes in this work were based on industrial robotic platform. It is revealed that the residual tensile stress has been effectively compensated and eventually converted into residual compressive stress while the hardness has been increased to 40.5 HRC, which is ideal for commercial Inconel 718 bulks after machine hammer peening.³⁰ More important is that the effective thickness of the studied RHP process is over 1000 μm , which is close to the thickness of a single-pass LC deposition (~ 1.5 mm). These results shed promising light on monolithic integrations between RHP and LC towards advanced AM process with reduced necessity of post-AM treatments.

2. EXPERIMENTAL METHODS

The Inconel 718 was deposited by LC on low-alloy steel (LAS) substrate. LAS coupons in 45 mm (L) $\times$ 45 mm (W) $\times$ 10 mm (H) were used as the substrate. Sand blasting and surface cleaning were processed in sequent before the LC process. Inconel 718 powder with the particle size distribution of $D_{10} = 56.99$ μm , $D_{50} = 78.75$ μm , and $D_{90} = 108.86$ μm was employed as feedstock during the LC process. Argon was used as the carrier- and the shielding-gas, flowing at 3.0 and 7.0 l/min, respectively. The spot size of the laser beam was ~ 4.9 mm in diameter; the working distance was 22 mm; the laser

power was 2500 W; the powder feeding rate was about 33 g/min; the scanning speed of the LC head with respect to the workpiece was 1100 mm/min; and the pitching width was 2.1 mm. These parameters led to a coating thickness of ~ 1.5 mm/layer. Two layers of coating, i.e., of ~ 3.0 mm thick, were processed in this work.

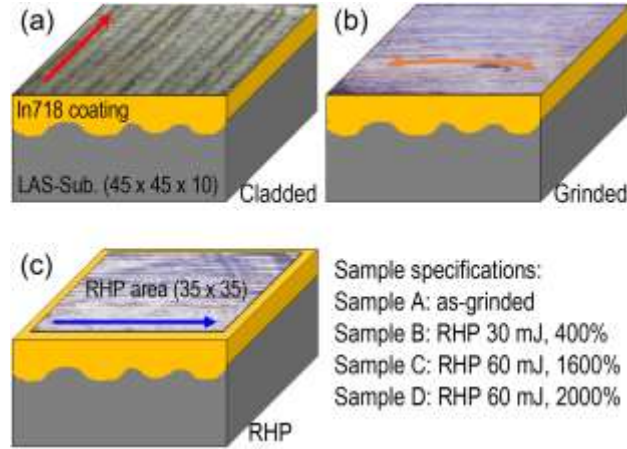


Figure 1: Grinding and robotic hammer peening (RHP) of laser-cladded (LC) Inconel 718 coatings. (a), (b), and (c) Schematic diagrams showing the dimensions (in millimeter) and orientation of the LC, the grinding, and the RHP processes, respectively.

After LC, the surface of the Inconel 718 coatings was grinded (at a speed of 200 m/s and cut depth of ~ 1.0 mm) and then RHP process was carried out with the longitudinal hammer head scanning direction perpendicular to that of the LC. The schematics in Figs. 1(a)-1(c) present the relative orientations of the LC, the grinding, and the RHP processes. To study the effect of RHP intensity on the Inconel 718 coatings, four typical samples were compared. They are the as-grinded sample and the RHP processed samples with the peening strength, i.e., the impact energy and the dent overlap, varied

from 30 mJ and 400%, through 60 mJ and 1600%, to 90 mJ and 2000%. For the sake of brevity, hereafter, they are referred to as samples A (as-grinded), B (30 mJ, 400%), C (60 mJ, 1600%), and D (90 mJ, 2000%), respectively. It is worth mentioning that the dent overlap is the area interfered between two neighboring impacts. The overlap of 100% means every point of the surface is peened once. Increased overlap greater than 100% means every point of the surface is peened multiple times. Table 1 presents the compositional comparisons in weight percentage (wt%) of the Inconel 718 powder feedstock employed in this work and the resulted Inconel 718 coatings as well as the typical specifications of commercial Inconel 718 bulk materials. The RHP processing parameters are also summarized in table 1.

Table 1: Chemical compositions of Inconel 718 powder feedstock and the LC-deposited coatings as well as the typical specifications of commercial Inconel 718 bulk.

	Element	O	C	Al	Ti	Mo	Nb	Cr	Fe	Ni
Powder	wt%	0.02	0.06	0.72	0.81	3.22	5.39	17.81	18.96	Bal.
Coating	wt%	-	-	0.64	0.80	2.79	4.81	18.15	21.35	Bal.
Typical Specification	Min %	-	-	0.20	0.65	2.80	4.75	17.00	15.00	Bal.
	Max %	0.20	0.08	0.80	1.15	3.30	5.50	21.00	20.00	Bal.
RHP processing parameters										
Frequency	Speed	Impact Energy			Stroke Length			Hammer Radius		
220 Hz	1 m/min	30-80 mJ			2 mm			3 mm		

To understand the morphological and structural modifications of the LC-deposited Inconel 718 coatings by the grinding and RHP processes, we have employed various material characterization techniques, including optical microscopy (OM), step-profilometer, X-ray diffraction (XRD), electron backscatter diffraction (EBSD), transmission electron microscopy (TEM). Micro- and nano-indentation were also

employed to validate the hardness improvements of the LC-Inconel 718 coatings induced by the RHP process. The measurements were made not only on the surface but also on the cross-section, targeting at both the near-surface and the coating/substrate interface regions. The cross-section, perpendicular to the longitudinal direction of LC, was prepared by electrical discharge wire-cut machining followed by grinding and polishing. A 4-circle-goniometer XRD system (Cu-K α) equipped with a general-area-detector was used for phase analysis of the Inconel 718 coatings. For residual stress measurement, a system with two separated detectors fixed symmetrically besides the X-ray (Cr-tube) nozzle in the same beam plane (formed by the incident and the diffracted beams) was employed, targeting at the (311) atomic planes with varied tilt angles away from the surface normal direction. This configuration measures the in-plane residual stress along the tilting direction. The sample was rotated above its surface normal direction to change the direction for the in-plane stress measurement. Commercial software and materials database (stresstech) were applied in this work. For TEM studies the specimens were prepared by focused ion-beam milling at the cross-section of the Inconel 718 coatings.

3. RESULTS AND DISCUSSION

3.1. Morphology and Topography

Figures 2(a)-2(d) present the OM images recorded from the surface of samples A-D, respectively. Likewise, presented in Figs. 2(e)-2(h) are the topographies collected by step-profilometer from the surface of samples A-D, respectively. The arrow indicates the longitudinal direction of the RHP process. Their comparisons show clear grinding lines on sample A; apparent RHP dents along with grinding lines on sample B; the RHP dents

decreased in size and aligned clearly along the longitudinal direction rather than the transverse direction on sample C; the dent distinctions, both the size and the alignment, are significantly reduced on sample D than on samples B and C; and the grinding lines completely disappeared from the surface of samples C and D. The surface roughness derived from the topographies in Figs. 2(e)-2(h) are 0.67, 1.62, 1.24, and 0.99 μm for samples A-D, respectively. Their variations exhibit a strong correlation with the XRD peaks, i.e., the intensity and the fullwidth at half-maximum (FWHM), measured from the surface (to be discussed later).

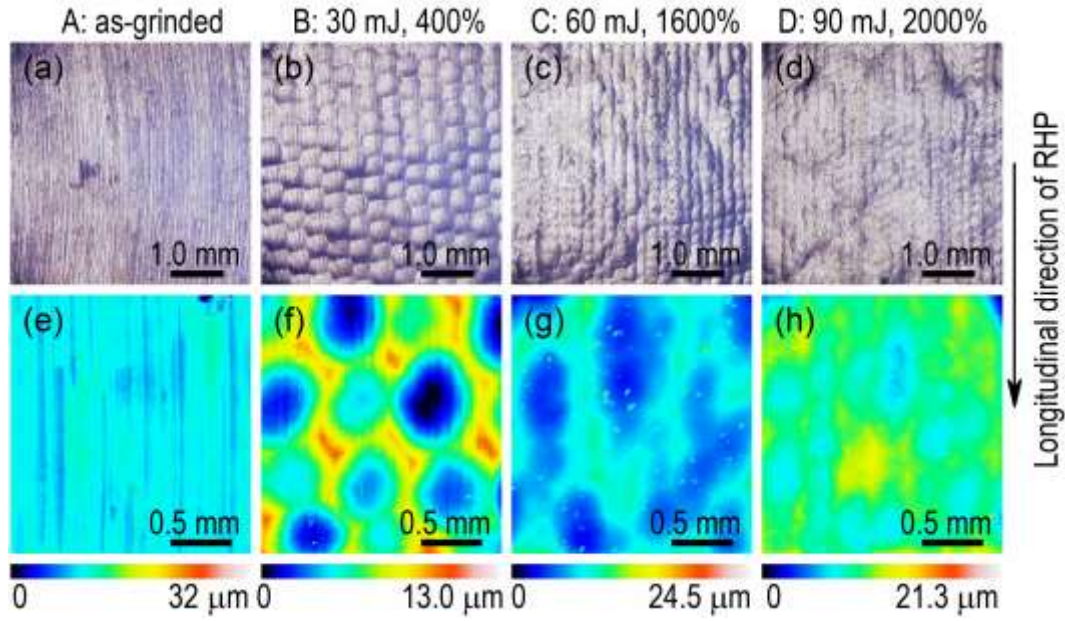


Figure 2: Morphology and topography of the as-grinded and RHP processed LC-Inconel 718 coatings. (a)-(d) Optical microscopic (OM) images recorded from samples A-D, respectively. (e)-(h) Topographic images recorded by surface height mapping using step-profilometer from samples A-D, respectively.

The peening-induced surface roughness increase followed by decrease at increased peening coverage has been frequently observed by different peening methods, including shot peening, LSP, and RHP.^{25-28, 31} It is generally believed that the increased surface roughness is a negative factor for the processed samples. However, the regularly distributed grinding lines [see Figs. 2(a) and 2(e)], having their peaks as the potential centers for stress concentrations, tend to be irregularly smeared by the peening-induced plastic deformations and eventually eliminated by increasing the coverage (termed by the overlap) of the peening dents [Figs. 2(c)-2(d) and 2(g)-2(h)]. It is worth mentioning that because of the controlled movement of the hammer head during RHP, especially the conformal movement along the surface of the workpiece, surface burrs or micron scales, which have been earlier observed in severe shot peening,²⁸ are apparently reduced from the processed surface. This is a desired advantage of RHP process as compared to shot peening in mitigating premature fracture initiations.³¹

3.2. Substrate Warping by LC and Residual Stress Compensation by RHP

Warping of the base plate has been earlier observed for steel coupons with the similar dimensions upon laser treatments, especially when the surface of the coupons was melted and resolidified.³² The distortion has been attributed to the expansion and contraction of the surface layer during the melting and resolidification, respectively. As a common result, residual tensile stress is induced in the surface layer and thus the coupon is warped with its edges bended up after releasing from its fixture. Such coupon warps have been frequently applied to estimate residual stress resulted from LC process.^{16-17, 32} In this regard, we have measured height profiles at the rear surface of the substrate for samples A-D along both the longitudinal and transverse directions of the LC process. The results

are presented in Figs. 3(a) and 3(b), respectively. One sees that the LC-induced warping (sample A) along the longitudinal direction [see Fig. 3(a)] is much more significant than that along the transverse direction [see Fig. 3(b)]. Also seen is that the RHP process tends to reduce the warping in both directions and the reductions increased with the RHP intensity from samples B to D. A general conclusion is that residual tensile stress has been built in the LC-deposited Inconel 718 layer with larger residual tensile stress along the longitudinal direction than that along the transverse direction and both were compensated by the RHP process. To quantify the comparisons, we have fitted the height profiles using circular arc with a typical example shown in Fig. 3(c), from which the curvature radius can be derived. Figure 3(d) plots the obtained results, which show that the curvature radius monotonically increased with the RHP intensity, especially along the transverse direction of the LC process.

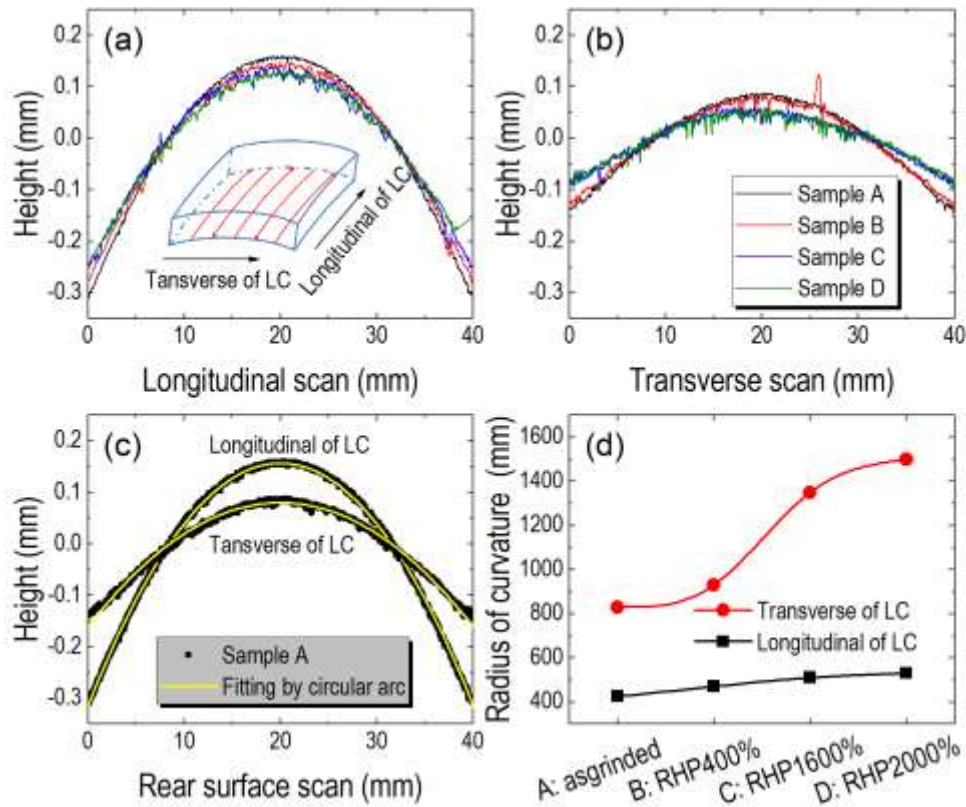


Figure 3: Substrate distortions induced by the LC process and their compensations by the RHP process measured by step-profilometer. (a) and (b) Comparisons of the surface height profiles measured from the rear surface of the substrate along the longitudinal and transverse directions, respectively. (c) Typical fittings of the surface height profiles using circular arc functions. (d) Comparison of the curvature radius derived from the fittings, showing the distortion compensations by RHP.

Figure 4(a) presents XRD patterns collected from the surface of samples A-D, showing a single-phase face-centered cubic (FCC) structure, i.e., the γ -phase, of the Inconel 718 coatings. The XRD peaks shifted to lower angles after the RHP process. The dashed line clearly indicates the angular shifts of the (311) peaks at about 90° . Such angle shifts indicate an increased lattice constant of the FCC crystal along the surface normal direction, which, in turn, implies that in-plane compressive stress, i.e., residual compressive stress, has been introduced by the RHP process in the near-surface region. To validate the RHP-induced residual compressive stress, stress measurements were carried out from the sample surface at the center by using the 2-detector XRD system [see section II above and the inset in Fig. 4(b)] and the results are presented in Fig. 4(b). It is clearly seen that the residual tensile stress in both directions monotonically decreased and eventually converted to residual compressive stress by increasing the RHP intensity from samples A to D. It is also seen that the stress difference between the longitudinal and transverse directions of LC monotonically increased with the RHP intensity.

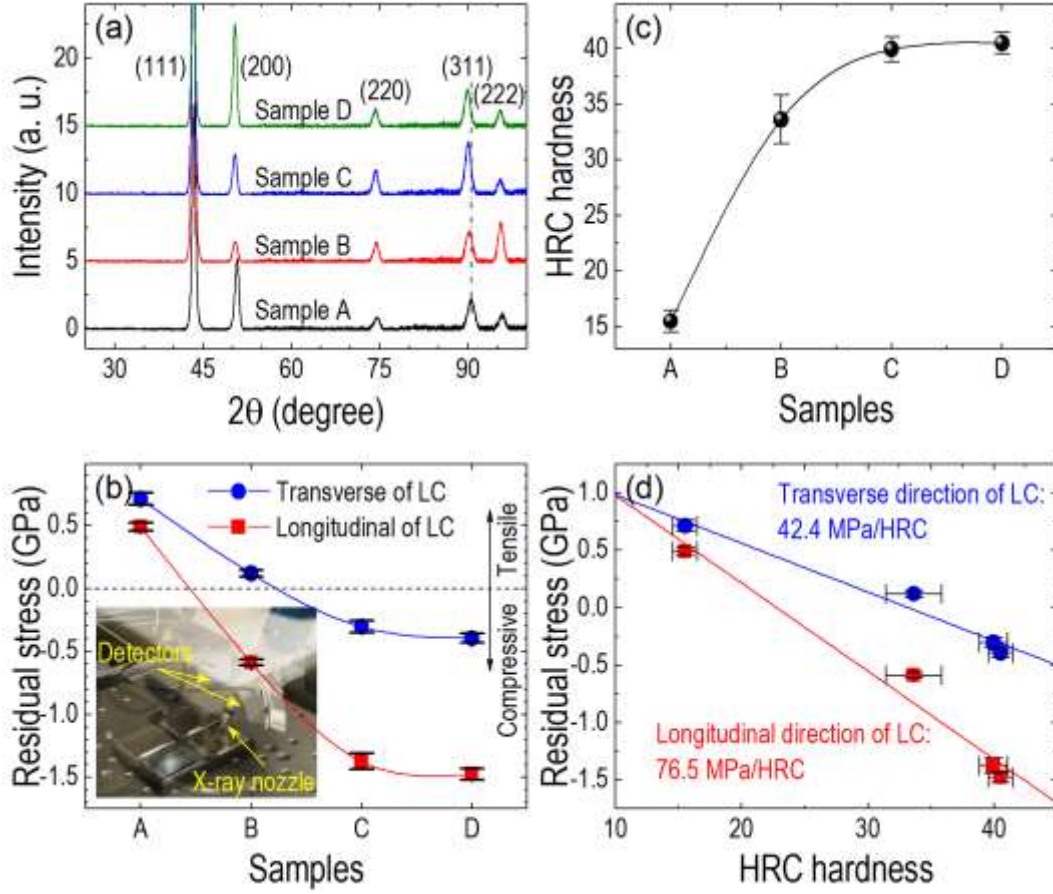


Figure 4: Residual stress evolutions and surface hardening induced by RHP. (a) XRD patterns, showing the peak shifts to lower angles and thus the compressive in-plane stress induced by the RHP process. (b) Residual stress measured by XRD in both the transverse and longitudinal directions of LC. (c) Surface hardness measured by micro-indentation (0.3 kg load) for sample A-D. (d) Plots of the residual stress as a function of the surface HRC hardness, exhibiting a near-linear correction.

Surface hardness was measured by micro indentation (Mitutoyo HM 810-125E) at a load of 0.3 kg and the results are plotted in Fig. 4(c). They show that the surface hardness of the as-grinded sample, i.e., sample A, is about 16 HRC, which has been monotonically increased by increasing the RHP intensity from samples A to D. A

comparison between samples C and D indicates a trend of saturation in the hardness improvement and the hardness increment by the RHP process is quite significant, i.e., over 40 HRC of samples C and D. This value, right in the maximum range of commercial Inconel 718 bulks after machine hammer peening,³⁰ provides evidence that the RHP process can effectively enhance the hardness of Inconel 718 components produced by LC-based AM methods without needing any post-AM treatments.¹⁶⁻¹⁷

After reviewing various cold-working methods, including cavitation peening, shot peening, and water jet peening, H. Soyama et al. noticed a linear correlation between the plastic deformation-induced residual compressive stress and the Vickers hardness, which is stronger than that between the residual compressive stress and the yield stress of bulk metals.³³ It is interesting to know whether this correlation is valid for RHP process. In this regard, we have plotted the residual stress as a function of the HRC hardness measured from the surface of samples A-D in Fig. 4(d). The solid lines are the best linear fittings, which show a near-linear correlation between the residual stress and the HRC hardness in both the longitudinal and the transverse directions of the LC process. This finding for RHP process agrees well with those reported by H. Soyama et al. for cavitation peening process.³³ Also seen in Fig. 4(d) is that the increase in residual compressive stress is faster along the longitudinal direction than that along the transverse direction of the LC process. It has been shown in Fig. 1 that the longitudinal scanning direction of the RHP process was rotated by 90° from that of the LC process. In this configuration, the residual compressive stress induced by RHP along its transverse direction is higher than that along its longitudinal direction, which is consistent with those reported on RHP process of steel coupons with the similar processing parameters.³⁴

3.3. Grinding-Induced White Layer and Swept Deformation

Figure 5 presents the band contrasts measured by EBSD from the cross-section perpendicular to the LC-direction of samples A, C, and D (see the orientation notation at the bottom of Fig. 5). The overall images in Figs. 5(a)-5(c) show columnar grain structures extending towards the surface of the Inconel 718 coating layer. Presented in Figs. 5(d)-5(f) are the band contrasts collected with higher magnifications from the near-surface regions of samples A, C, and D, respectively. A layer with darker contrast is seen on the surface as indicated by the arrows, which is similar to those so-called ‘white layer’ in the literature because of its white contrast under optical microscope.³⁵ Its darker contrast in EBSD mapping is caused by the zero-solution of the diffraction patterns,²⁸ i.e., unable to resolve the crystallographic structures from weak Kikuchi patterns due to the severely deformed grains. Figures 5(d)-5(f) also exhibit a deformed layer immediately below the featureless white layer. Swept features, as indicated by the dashed/arrowed curves, can be clearly seen in the deformed layer, extending from the white layer into deeper regions of the Inconel 718 coating. The morphological comparisons among Figs. 5(d)-5(f) indicate that the grinding-induced featureless skin layer (i.e., the white layer) in conjunction with its adjacent swept features-extending deformed layer [see Figs. 5(a) and 5(d)] is similar to those induced by mechanical turning on Inconel 718,³⁶ where Zhang et al. attributed the formation of white layer, i.e., the turning-induced featherless surface layer, to phase transition and dynamic recrystallization. In fact, machining-induced white layer (typically featureless) along with an adjacent deformation layer (exhibiting swept or dragged features) has been commonly observed not only in Inconel 718 but also in other alloys such as AISI 1045 annealed steel, Ti-6Al-4V, Ti-834, etc.³⁵⁻⁴⁰ To this end, the

layer with darker contrast observed on the surface in Fig. 5 can be assigned to a white layer while the deformation layer immediately under the white layer before RHP process [see Fig. 5(d)] can be attributed to grinding-induced severe plastic deformation.^{36-37, 40}

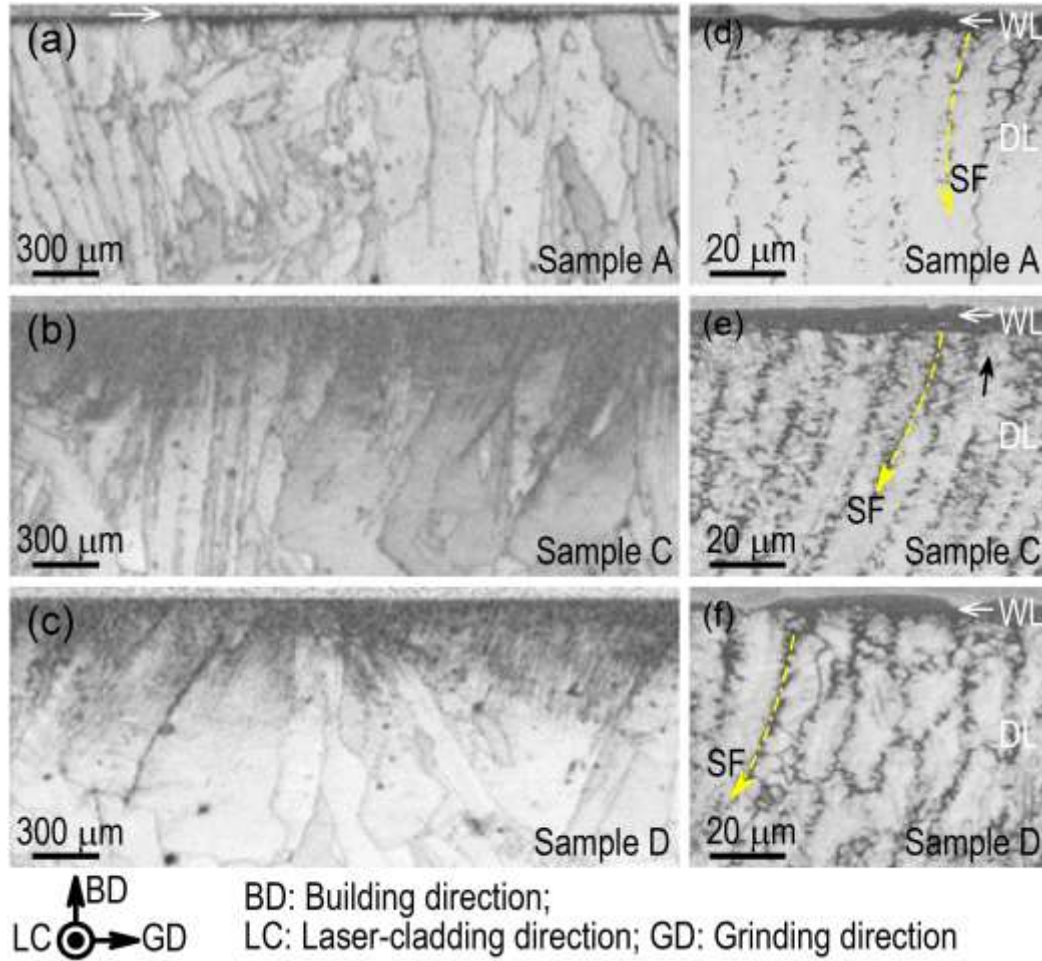


Figure 5: Band contrast measured by EBSD from the cross section of the studied Inconel 718 coatings. (a)-(c) Band contrasts measured with lower magnifications from samples A, C, and D, respectively. (d)-(f) Band contrasts measured with higher magnifications from samples A, C, and D, respectively. The horizontal arrows indicate the grinding-induced ‘white layer’ (WL); the black arrow in (e) indicates the presence of intercrossed linear structures in the deformation layer (DL); and the dashed curves indicate the swept features (SF) in the DL.

A comparison of Figs. 5(d)-5(f) reveals that the deformation layer was deformed not only by the grinding process but also by the RHP process. This is manifested by the increased thickness of the swept features (indicated by the dashed curves) in the deformation layer when the RHP intensity is increased from sample A to samples C and D. The regular distribution of the swept features, i.e., aligned along vicinal directions of the column grain growth [e.g., see Figs. 5(a) and 5(d)] with certain horizontal intervals [see Figs. 5(d)-5(f)], is similar to the elemental segregation-induced Laves phase in Inconel 718.⁴¹⁻⁴²

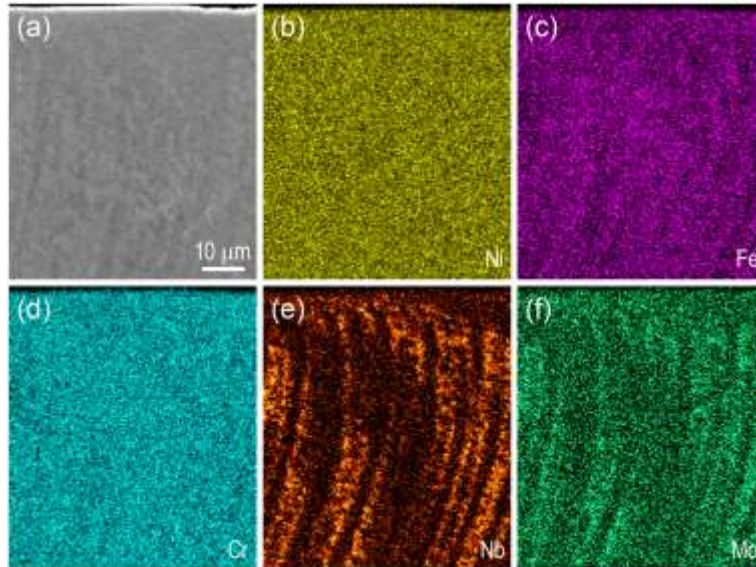


Figure 6: SEM and EDX mapping of elemental distributions collected from the cross section of sample D. (a) SEM image and (b)-(f) Elemental distributions of Ni, Fe, Cr, Nb, and Mo, respectively.

To validate the presence of elemental segregations at the swept features in the deformation layer, we have carried out SEM/EDX analysis. Figure 6 presents a SEM image and EDX mappings of Ni, Fe, Cr, Nb, and Mo distributions collected from the

cross section at the near-surface regions of sample D with the same orientation as that shown in Fig. 5. Segregations of Nb and Mo are clearly seen in Figs. 6(e) and 6(f), respectively. They are precisely following the distributions of SFs in the DL [see Fig. 5(f)]. In contrast, the distribution of Fe in Fig. 6 (c) is slight poor in the Nb- and Mo-rich regions while the other alloy elements do not exhibit any varied distributions. The same regular distribution the Nb/Mo-segregation as that of swept features in the deformation layer provides evidence that the unresolved dark regions, i.e., the swept features, in the deformation layer [see Figs. 5(d)-5(f)] consisted of Nb- and Mo-rich Laves phase.⁴¹⁻⁴² It has been reported that the segregation of alloying elements, typically Nb and Mo, on the front of solid-liquid interface in the dendrite growth of Inconel 718 tends to promote the nucleation and growth of Laves phase along with the γ -phase dendrites.⁴¹⁻⁴² In this light, the columnar growth of the Inconel 718 coatings [see Figs. 5(a)-5(c)], accompanied with Nb- and Mo-segregation along the columnar dendrite structures, gave rise to the regular distributions of the Laves phase in the swept feature regions [see Figs. 5(d)-5(f)].

Figure 7 shows the TEM images taken from the cross section of sample A near the surface regions with the specimen orientation shown in Fig. 7(a), where LC indicates the longitudinal direction of LC, BD indicates the building direction of the coating layer, and GD indicates the grinding direction (see Fig. 1). Figures 7(a)-7(f) are the bright-field image, reliability, phase map, inverse pole-figure (IPF)-X, -Y, and -Z, respectively, obtained by scanning the nanosized electron beam across the specimen and calculating diffraction patterns collectively. Figures 7(g)-7(i) are the TEM images showing the microstructural morphologies induced by the grinding process in the near-surface regions. The white layer is clearly seen in Figs. 7(a)-7(g) at the surface of the Inconel 718 coating,

consisted of nanograins due to dynamic recrystallizations induced by the severe plastic deformation and the localized heating, i.e., the thermal mechanical reactions, during the grinding process.⁴³⁻⁴⁴

A comparison between the white layer and its underneath region reveals grain refinement and grain elongations along the grinding direction within the white layer. Laves has been identified at the grain boundary (GB) of Inconel 718 [see Figs. 7(c) and 7(f)]. Figure 7(h) is TEM image taken with a higher magnification from the white layer. It shows the presence of SGs. In comparison, the TEM image in Fig. 7(i), which was taken from a location below the white layer, exhibits some dark crosslines. These crosslines are most likely twin deformations and/or shear bands induced by the grinding process in the deformation layer below the white layer as those observed for the as-grinded Inconel 718 coating, i.e., sample A, in Fig. 5(d). It is worth mentioning that stress relaxation alongside the warping-induced plastic deformation as those observed in Fig. 3 might also contribute to the formation of shear band.

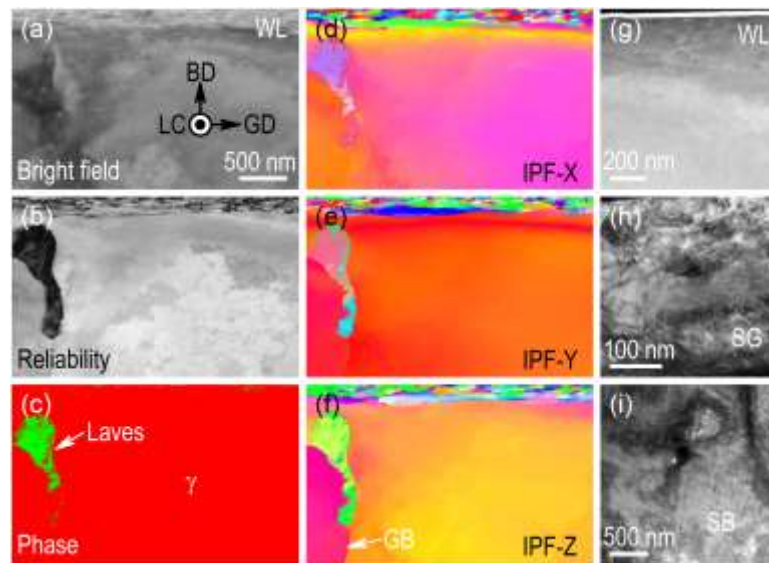


Figure 7: TEM images collected from the cross section of sample A at the near-surface region. (a) Bright field TEM image, (b) Reliability, (c) Phase map, (d)-(f) IPFX, IPFY, and IPFZ, respectively. (g)-(i) TEM images taken with higher magnifications from the white layer and its lower deformed regions.

Figure 8 presents the TEM results collected at a depth of $\sim 20 \mu\text{m}$ below the surface of sample D, i.e., in the deformation layer, where columnar grain growths were formed in dendritic structures [see Fig. 5(f)]. The TEM image in Fig. 8(a) and the SAED pattern in Fig. 8(b) show a single crystal γ -phase grain with a lot of slip bands. The comparison in Figs. 8(a) and 8(b) revealed that the slip bands are dominantly aligned along the orthogonal $[110]$ -axis directions as indicated by the arrows in Fig. 8(a). Figures 8(c)-8(e) are TEM images recorded with higher magnifications of the slip band ($\sim 70 \text{ nm}$ in width). The fast Fourier transformation (FFT) image in the bottom-left and top-right insets of Fig. 8(e) were transformed from the left and right at the top corners of Fig. 8(e), respectively. The top-right inset shows a single crystalline FCC structure with its (111) atomic planes parallel to the boundary of the slip band. However, the presence of twins and dislocations in the slip band resulted in a sharp line cutting across the (111) diffraction spots in the FFT image in the bottom-left inset. Figure 8(f) presents six slip bands, indicated by the arrows, at the left area seen in Fig. 8(e). A high-resolution TEM image of such a defected region is shown in Fig. 8(g) and its inverse FFT is shown in Fig. 8(h). They show an accumulation of dislocations and stacking faults along the slip band for the (111) atomic planes. These results suggest that crystallographic shear has been induced by the RHP process along a vicinal direction of the surface normal.

Figures 8(i)-8(m) are the phase and crystallographic orientation maps collected from the deformed layer of sample D. They show the presence of γ and Laves phases. The shear bands [see Figs. 8(i) and 8(j)] are dominated by γ -phase. However, the comparison in Figs. 8(k)-8(m) shows the same crystal orientations on both sides of the shear bands but crystal plane tilt can be clearly distinguished within in the shear bands. This orientation relationship as well as the dominant phase structure of the shear band in the deformation layer is mainly caused by the RHP process via plastic deformation controlled by the crystallographic shear.²⁶ It is worth mentioning that the TEM specimen in Fig. 8 was cut parallelly to the surface plane, i.e., in a top-view direction, from the deformed layer of sample D, which is different from the side-view specimen shown in Figs. 5 and 6. Therefore, the Laves phase, which is associated with the regularly aligned Nb- and Mo-segregation along the surface normal direction (see Figs. 5 and 6), does not show extending features in Figs. 8(i)-8(m).

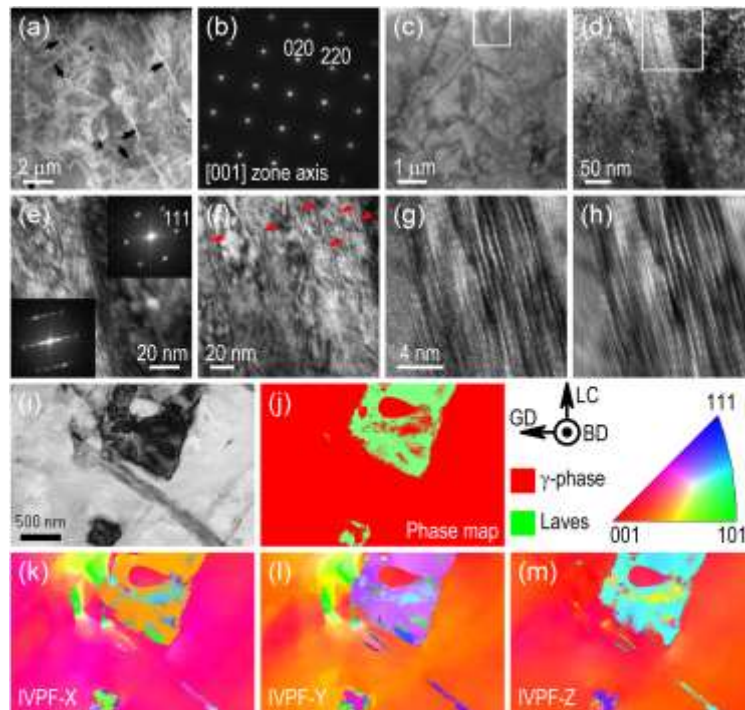


Figure 8: TEM images collected from the deformed layer (DL) of sample D. (a) TEM image, (b) SAED pattern, (c) and (d) TEM images with high magnifications for the linear defect structures, (e) and (f) TEM images identifying detailed defect structures of deformed grain structures, including dislocations and deformation twins, (g) and (h) TEM image and its IFFT of a typical deformation band, and (i)-(m) Band contrast, phase, and crystal orientation maps derived from TEM diffractions. The bottom-left and up-right insets in (e) are SAED patterns collected from the left and right parts in the TEM image, respectively.

3.4. Microstructures and RHP-Induced Integrity Enhancements

Figures 9(a)-9(d) present the SEM images taken from the cross-sections of samples A-D, respectively. Thin layer of columnar grains, as indicated by the arrows, has been formed in the Inconel 718 coatings at the interface. The formation of the columnar grains perpendicular to the interface is typically caused by the fast heat dissipation through the LAS substrate and thus the orientated cooling and solidification of the melt pool. The fluctuated interface indicates onset of substrate dilutions and thus the metallic interface bonding.^{3, 45} The substrate dilution is consistent with the compositional comparisons between the Inconel 718 coating and the powder feedstock in table 1. A more important observation is the presence of pores in the Inconel 718 coatings. However, the comparisons in Figs. 9(a)-9(d) show that the porous density is reducible by the RHP process, especially in the near-surface regions (i.e., up to 500 μm beneath the RHP processed surface).

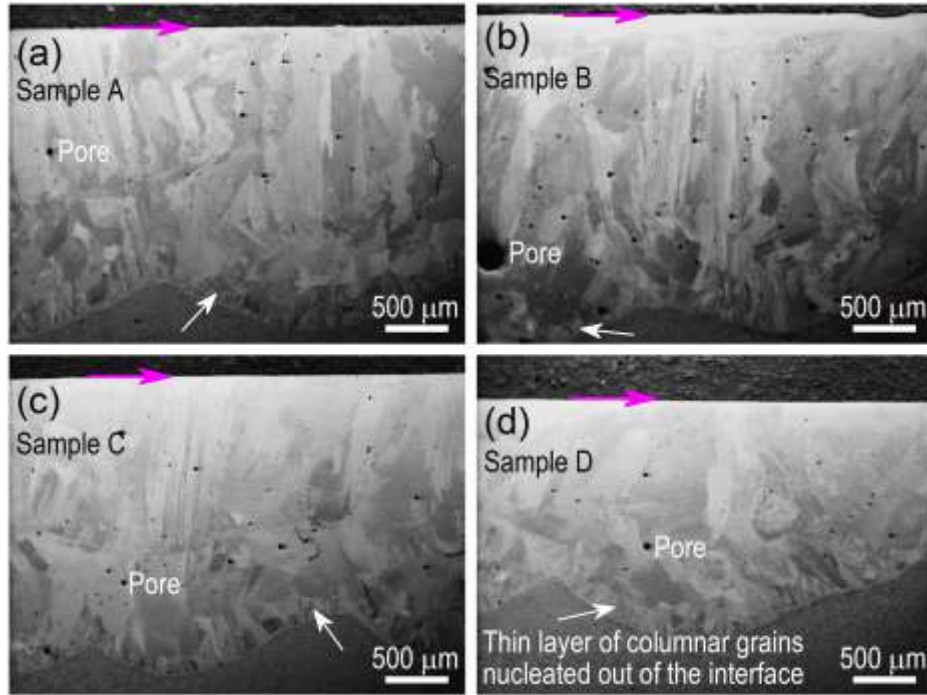


Figure 9: SEM images recorded from the cross section of samples A-D, showing the distribution and RHP-induced closure of the pore structures near-surface of the LC-deposited Inconel 718 coatings. (a) Sample A, (b) Sample B, (c) Sample C, and (d) Sample D. The horizontal arrows indicate the surface of the individual coupons.

Figures 10(a)-10(d) present the grain boundary structures recorded by EBSD from the cross sections of samples A-D, respectively. The grain boundaries with the angle smaller than 15° were highlighted in blue, i.e., the low-angle grain boundary (LAGB). It is seen that the distributions of LAGB increased in density and penetrated deeper into the base material of the Inconel 718 coatings by increasing the RHP intensity from samples A to D. Presented in Figs. 10(e)-10(g) are the relative densities as a function of depth derived from the EBSD results for the LAGB, the deformed grains, and the recrystallized grains, respectively. The dashed arrows indicate the evolutions from samples A to D.

Figure 10(e) shows that LAGBs have been significantly increased in the near-surface regions by the RHP process and the increasement monotonically increased with the RHP intensity from samples A to D. The transition points, i.e., from the increased LAGB in the near-surface region to the stable ones in the base material, shifted from $\sim 800\text{ }\mu\text{m}$ for sample B to $\sim 1100\text{ }\mu\text{m}$ for sample C and much deeper than $1200\text{ }\mu\text{m}$ for sample D. In comparison, the deformed grain distribution in Fig. 10(f), although following the similar trace of LAGB in Fig. 10(e), exhibits a similar transition point, i.e., at about $1000\text{ }\mu\text{m}$, for samples C and D. Likewise, the distribution of the recrystallized grains in Fig. 10(g) exhibits a same depth of transition point at $\sim 1000\text{ }\mu\text{m}$ for samples C and D. The comparisons in Figs. 10(e)-10(g) provide evidence that the effective depth of plastic deformations induced by the RHP process is about $1000\text{ }\mu\text{m}$ while the RHP effect on grain boundaries is much deeper than $1200\text{ }\mu\text{m}$ in the LC-deposited Inconel 718 coatings. This difference, depending on the definition of LAGBs, i.e., $\leq 15^\circ$ in this work, indicates that LAGBs with even lower angles corresponding to the deformation, recrystallization, and sub-structuring of the grains in the LC-Inconel 718 coatings. Nevertheless, the thickness of the surface layers where significant grain deformation occurred is quite close to the coating thickness deposited by a single-pass LC process (i.e., $\sim 1500\text{ }\mu\text{m}$). This finding, together with the RHP-induced residual compressive stress, pore closures, and hardening, provides evidence that RHP can be an effective integrity enhancement process when integrated, as an interpass process, with LC-based AM technologies.

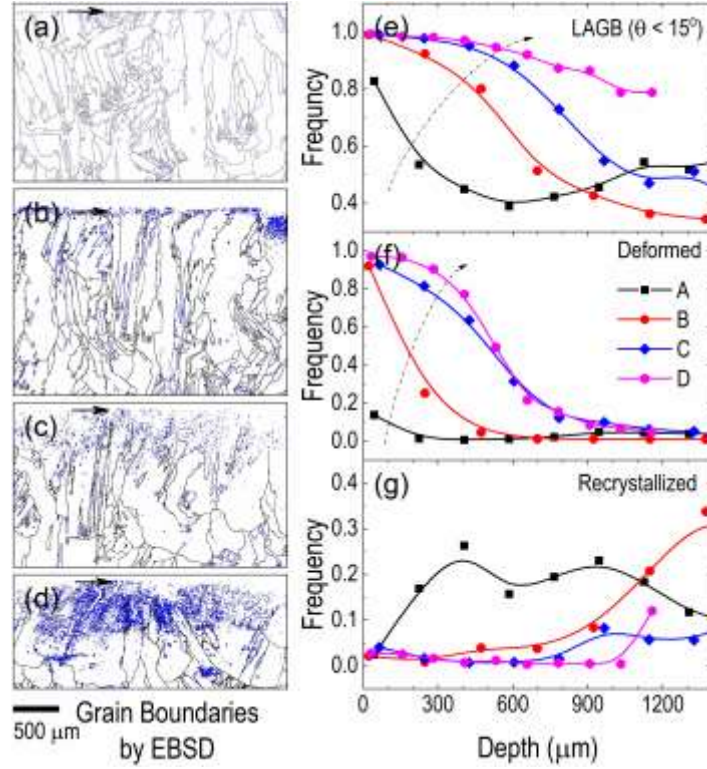


Figure 10: Grain boundaries and RHP-induced grain deformations measured by EBSD from the cross section of the LC-deposited Inconel 718 coatings. (a) Sample A, (b) Sample B, (c) Sample C, (d) Sample D. (e), (f), and (g) Comparisons of the depth dependent LAGBs ($< 15^\circ$), deformed grains, and recrystallized grains, respectively. The horizontal arrows indicate the surface of the coupons.

To validate the RHP effect on the LC-deposited Inconel 718 coatings, we have further carried out hardness and Young's modulus measurements as a function of depth by nano-indentation at the cross sections of samples A-D. The results are plotted in Fig. 11. The dashed curves are empirical fittings using an exponential decay function. It is seen that the hardness of the Inconel 718 coating monotonically reduced from ~ 6.0 GPa at the surface to ~ 3.3 GPa at the bottom of the coating, i.e., near the interface regions. A sharp drop occurred at the coating/substrate interface, i.e., from ~ 3.3 GPa in the Inconel

718 coating to ~ 2.5 GPa in the LAS substrate. In comparison, the modulus also exhibits a decrease when moving deeper into the coating layer, but the relative decrease rate is slower than that of the hardness. It is worth mentioning that both the thickness of the remaining Inconel 718 coating and the thickness of the grinding-induced white layer are unintentionally varied for the studied samples (see Figs. 5 and 9). They might lead to varied effects from the surface (white layer) and the interface (substrate dilution) regions on the nanoindentation measurements. Nevertheless, to demonstrate the depth-dependent mechanical changes, hardness-to-modulus ratios, i.e., H/E , have been plotted in Figs. 11(c), 11(f), 11(i), and 11(l) for samples A-D, respectively. They show that the effective thickness of the RHP process is about $1500\text{ }\mu\text{m}$, which is indeed close to the thickness of a single-pass LC deposition. This character of RHP process shed promising light on its application as an interpass process for integrity enhancement of metallic components fabricated by LC-based AM methods.

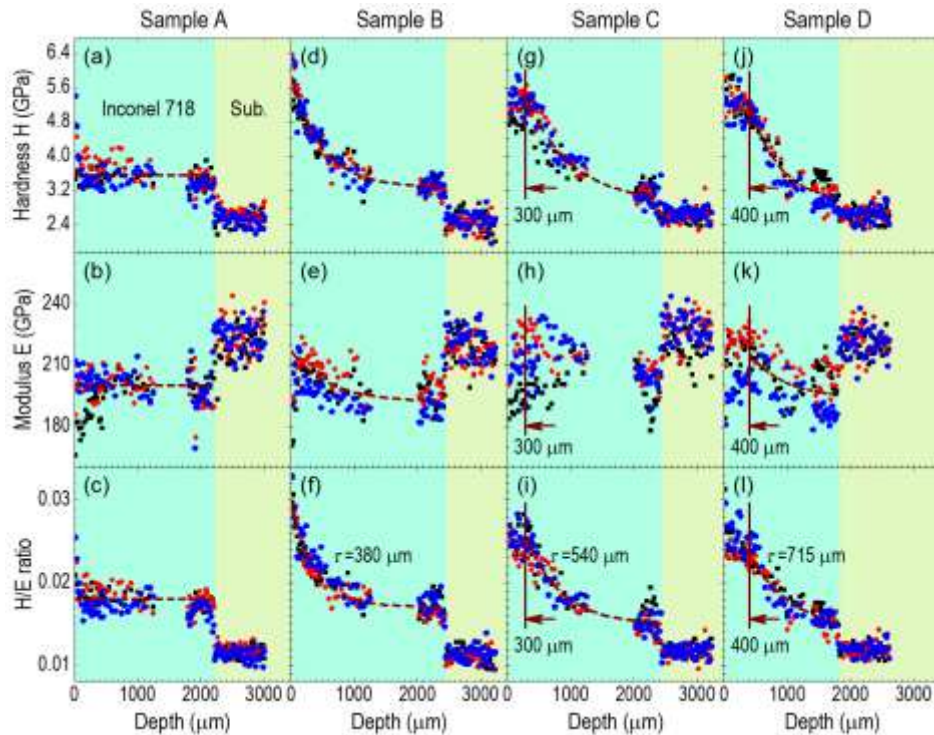


Figure 11: Hardness and modulus measured by nanoindentation from the cross section of samples A-D as a function of distance from the surface. Hardness-to-modulus ratios are also plotted for comparisons. The dashed lines are empirical fittings using exponential functions.

3.5. Discussion

Recent development in AM of Inconel 718 has shown its great potentials in aerospace industry, however, post-AM process is still required in the current stage to enhance the quality of AM-Inconel 718 approaching those manufactured by traditional methods. Cold-working has been recognized as a powerful method to introduce plastic deformation in the near-surface region of metallic alloys via introducing compressive stress, dislocations and their network, crystallographic shear, grain twinning and grain refinement, and therefore, integrity enhancement. Our experimental results of RHP process in LC-Inconel 718 show that the surface morphology and roughness can be modified by engineering the RHP processing parameters, typically the overlap of adjacent impact dents. Both hardness and compressive residual stress measured from the surface of the LC-Inconel 718 coupons apparently increased as a function of the RHP intensity. They also exhibited a linear correlation with each other upon the cold-working, which can be further developed for in-situ monitoring the surface hardness during cold-working process by non-destructively measuring the residual stress. Grain refinements have been observed not only in the white layer but also in the deformed layer below it, dominated by grain twinning and atomic plane slipping, respectively. Elemental segregations and the precipitate of Laves phase at the dendritic boundaries played an

important role in the plastic deformation, however, absence of phase transformation was observed. The RHP-induced shear strain near the porous structures tends to promote crystallographic shear and material transport into the pores, leading to the pore closures.^{24, 26, 46-48} Finally the analysis of both the grain deformation and the RHP-induced hardening as a function of depth of the LC-Inconel 718 coupons indicate that the effective depth of the RHP process is close to the coating thickness of a single-path LC process, which is also much thicker than the influenced thickness of the first LC layer by the second LC path. This comparison suggests that RHP process can contribute its functions as an interpass method, regardless of the surface melting, in LC process.

4. CONCLUSION

In conclusion, we have carried out RHP process on LC-deposited Inconel 718 coatings by increasing the impact energy and dent overlap. Through analyzing the distortions of the substrates and charactering the morphological, structural, and mechanical properties of the coatings, we found that the tensile stress built by the LC process in the Inconel 718 coating can be compensated and eventually converted to compressive stress. The residual stress modifications in the near-surface region apparently reduced the substrate distortions. Surface hardness of the LC-Inconel 718 coatings has been significantly enhanced by the RHP process from 16 HRC up to 40.5 HRC, i.e., to the range of commercial Inconel 718 bulks fabricated and treated by traditional methods. There is a linear correlation between the residual compressive stress increase and the surface hardening induced by the RHP process. The residual compressive stress increase along the longitudinal direction (i.e., 76.5 MPa/HRC) is faster than that along the transverse direction of the LC process (i.e., 42.4 MPa/HRC), which is attributable to the anisotropic

stressing behavior of both LC and RHP process. These linear correlations can be a guidance when design RHP process for LC-based AM. Grain deformations, dominated by nanosized crystalline domains in the white layer and crystallographic slip band/shearing in the deformation layer, along with the RHP-induced pore closures have been observed in the near-surface regions with an effective depth of over 1000 μm . This has been further validated by nano-indentation measurements at the cross-section, where the hardness was monotonically decreased from ~ 6.0 GPa at the surface to ~ 3.3 GPa at the bottom of the Inconel 718 coating after the RHP process. The effective thickness of the RHP-hardened near-surface region is ~ 1500 μm . Both the microstructural changes and the nano-indentation measurements provide evidence for an effective thickness of the RHP process close to that of a single-pass LC deposition. This result, together with the RHP-induced residual compressive stress and pore closure, sheds promising light on integration of RHP as an interpass process into LC-based technologies for advanced AM with reduced need of post-AM treatment.

Declaration of competing interest:

The authors are here declaring no conflict of interest in this work for publication.

ACKNOWLEDGEMENT

This work is supported by A*STAR RIE2020 advanced manufacturing and engineering (AME) programmatic grant through the structural metal alloys program (SMAP, Grant no. A18B1b0061, Project no. SC25/18-8R1715-PRJ6). TLM appreciates the financial support from Enterprise Singapore and Dura-Metal (S) Pte. Ltd. through a T-UP project on protective coatings by direct energy deposition.

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