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2019

Kumar, D., Idapalapati, S., Wang, W., Child, D. J., Haubold, T., & Wong, C. C. (2019).
Microstructure-mechanical property correlation in shot peened and vibro-peened Ni-based
superalloy. *Journal of Materials Processing Technology*, 267, 215–229.
[doi:10.1016/j.jmatprotec.2018.12.007](https://doi.org/10.1016/j.jmatprotec.2018.12.007)

<https://hdl.handle.net/10356/144788>

<https://doi.org/10.1016/j.jmatprotec.2018.12.007>

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Microstructure-mechanical property correlation in shot peened and vibro-peened Ni-based superalloy

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Abstract

In this experimental research, shot peening (SP) and vibro-peening (VP) processes were employed to modify the surface and sub-surface of a Ni-based superalloy Udimet®720Li. The effect of these treatments on the surface topography, microstructure evolution, and mechanical properties was investigated using instrumented tests. The compressive residual stresses and plastic strain generated through surface treatment were characterized using X-ray diffraction (XRD). The depth of plastically strained region was estimated using a quantitative analysis of grain orientation spread (GOS) data from electron back-scattered diffraction (EBSD) technique. It was observed that the magnitude of compressive residual stresses induced by VP is relatively low compared to SP, but the converse is true for depth of influence (DOI) for the same peening intensity. After thermal exposure at 650 °C ($\sim 0.4T_m$) for 5 h, the relaxation of compressive residual stresses and plastic strain was less pronounced in vibro-peening when compared to shot peening representing an improved thermal stability of the former process. Furthermore, 5.24 GPa surface micro-hardness of the untreated sample (O) was increased by $\sim 30\%$ and $\sim 25.2\%$ following SP and VP respectively. This resultant micro-hardness enhancement is due to grain refinement, plastic strain effects, and the introduction of low angle grain boundaries (LAGBs). Finally, a positive correlation among microstructural features and mechanical properties was fitted from the experimental data.

Keywords: Ni-based superalloys, Surface treatments, Microstructure, Residual stresses, plastically strained region, Electron back-scattered diffraction (EBSD)

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Nomenclature:

O	:	Untreated sample
SP	:	Shot peening
SPV	:	Combination of shot peening and vibro-polishing
VP	:	Vibro-peening
SPHT	:	Shot peening after exposed to 650 °C for 5 h
SPVHT	:	Combination of shot peening and vibro-polishing after exposed to 650 °C for 5 h
VPHT	:	Vibro-peening after exposed to 650 °C for 5 h

1 Introduction

Ni-based superalloys are commonly used in aero-engine components due to their excellent corrosion and creep resistance at elevated temperatures. For example, Udimet720Li is being used in high-pressure turbine discs at an operating temperature of 650 °C as stated by Foss et al. (2013). It undergoes cyclic loading during the operation so high fatigue performance is desirable. In general, mechanical components fatigue performance can be improved through the good surface finish and inducing compressive residual stresses which inhibit crack initiation. Surface mechanical treatments like shot peening (SP) as reported by Altenberger et al. (1999), laser shock peening as presented by Peyre et al. (2000), and deep cold rolling as investigated by Wong et al. (2014) can be employed to induce compressive stresses. Among these techniques, SP is most widely used in the industry because of its operational simplicity, reliability, cost-effectiveness, and capability to treat complex geometries as demonstrated by Chattopadhyay et al. (2014). However, the surface roughness generated by SP is far beyond the requirement of aero-engine components, hence an additional process like vibro-polishing or drag finishing is required to achieve targeted roughness for improved aerodynamic efficiency, as stated by Ahluwalia et al. (2017) and Ciampini et al. (2007). Recently Feldmann et al. (2014) and Ardi et al. (2016) have demonstrated vibro-peening (VP) process as an alternative surface treatment method for introducing compressive residual stresses with the good surface finish.

Vibro-peening utilizes the effect of high-frequency vibrational energy for generation of plastic deformation, which is different from the conventionally used processes in terms of mechanism of plastic deformation, strain rate, and strain gradient. These parameters drive the dislocation multiplication mechanism, microstructure refinement, boundary spacing which in turn governs strength and other mechanical properties as reported by Liu et al. (2013). Thus, it is worth to investigate the microstructural evolution and mechanical properties under VP. Feldmann et al. (2014) reported preliminary results on surface roughness, residual stress, and high cycle fatigue life of blade integrated disk (blisk) of Inconel IN718 subjected to VP using media with different hardness values. For the relatively hard media, the magnitude of compressive residual stresses was comparable to SP, but the depth of influence (DOI) was higher, so higher fatigue life is anticipated. Later, Ardi et al. (2016) investigated the residual stresses and surface integrity generated by vibro-peened Ni-based superalloy RR1000. It was concluded that desirable compressive stresses with low plastic strain could be generated using VP to potentially optimize the fatigue performance of the components.

Although these methods can improve the fatigue resistance by inducing beneficial compressive residual stresses, the mechanical performance could be compromised during severe operating conditions, such as elevated temperature and corrosive environments. This is a significant concern for aerospace engineers as performance may not be optimized. Kim et al. (2005) investigated the stress relaxation behavior of shot peened Udimet720Li under elevated temperature exposure and isothermal fatigue. It was found that almost 50% of compressive residual stresses relaxed during the first few hours of the thermal exposure at 650 °C. Prevy (2000) described the mechanism of stress relaxation and correlated it with the plastic strain of the material treated by SP. However, there is still a lack of knowledge regarding the stress relaxation mechanism and its correlation with the microstructure parameters.

Kumar et al. (2018) revealed that compressive residual stresses and plastic strain are crucial to enhanced fatigue performance. Hence, it is important to characterize both these parameters accurately and validate them using various competing techniques for high level of confidence. Kumar et al. (2015) have used XRD technique, Schajer (1988) hole-drilling method and whereas Withers and Bhadeshia (2001) have employed neutron diffraction to measure the compressive residual stresses. However, there is still a lack of understanding on the quantification of plastic strain and its depth of influence in severe plastic deformation processes. Plastic strain can be measured using XRD peak broadening as in the work of Prevy (1987) or employing micro-hardness measurements as studied by Soady et al. (2013). The measured values of depth of plastically strained region invariably vary from one method to another one. For example, Soady et al. (2013) reported that micro-hardness overestimates the depth of plastically strained region when compared to the XRD technique. Ungr (2004) showed that XRD peak broadening is not only influenced by the plastic strain but also by other factors like crystalline size, dislocations, grain/sub-boundaries, twinning, stacking faults and instrumental broadening. Recently, different approaches using electron back-scattered diffraction (EBSD) characterization technique were utilized to evaluate the depth of plastically strained region as follows: image quality (IQ) by Wilkinson et al. (2010), kernel average misorientation (KAM) by Badiola et al. (2007), grain average misorientation (GAM) by Yoda et al. (2010), grain mean misorientation (GMM) by Foss et al. (2013) and grain orientation spread (GOS) by Child et al. (2011). In the GOS method, the depth of plastically strained region is defined as the GOS cut off value exceeding mean plus standard deviation, which underestimates it due to orientation noise effects as demonstrated by Foss et al. (2013) on shot peened Udimet720Li alloy. Later, Lain et al. (2017) estimated the depth of residual stresses for shot peened and laser shock peened Ti-6Al-4V using GOS data curve fitting. However, the estimated depth is not compared and correlated with the X-Ray peak broadening and micro-hardness values. Hence, this new approach was used in the current work

to quantify the depth of plastically strained region for shot peened and vibro-peened Udimet720Li and to correlate it with micro-hardness and FWHM. To the best of authors' knowledge, there is no comprehensive study on the effect of VP on the microstructure evolution and the mechanical properties on Ni-based superalloys.

In the current study, a detailed investigation on the effect of SP and VP (at the same Almen intensity) on surface topography, microstructural response, and mechanical properties were carried out with and without thermal relaxation. The surface modification effect on grain size distribution across the depth is investigated. Special attention was paid to the residual stresses analysis and plastically strained region after surface treatments. Finally, following the same experimental and analysis protocols for both the shot peened and vibro-peened sample, a mutual comparison of the processes has been made through correlations between various experimental measurement parameters such as FWHM and GOS, FWHM and micro-hardness.

2 Experimental Materials and Methods

This section details the sample preparation techniques and experimental method used for the surface modification techniques and characterization of surface topography, microstructural features, and mechanical properties.

2.1 Material Preparation

Commercially available Ni-based superalloy (Udimet720Li) was employed for the experimental study. It is a fine-grained (1-20 μm) polycrystalline material produced through forging followed by hot rolling. The samples were machined and polished using a series of emery papers from coarse to fine grit sizes, diamond paste and finally by colloidal silica suspension (0.04 μm) until a highly smooth surface (R_a of 0.02 μm) was achieved. Test coupons were heat treated in a vacuum furnace at 760 $^{\circ}\text{C}$ for 8 h to relieve residual stresses in the material arising from the

polishing. The chemical composition of Ni-based superalloy Udimet720Li, which has predominantly Co and Cr as alloying elements, is measured through electron dispersive spectroscopy (EDS) listed in Table1.

Table 1: Chemical composition of Udimet720Li in wt%

Cr	Co	Mo	W	Al	Ti	Zr	B	C	Ni
16	14.7	3.0	1.25	2.5	5.0	0.03	0.015	0.010	Bal.

2.2 Surface Mechanical Treatments

Shot peening (SP) and vibro-peening (VP) (as shown in Figs. 1(a), (b)) were employed for the surface treatment to the same Almen intensity (4-5 A). Almen intensity is a widely recognized industrial practice to quote the level of plastic deformation induced to the workpiece surface, see the work of Chattopadhyay et al. (2014). Additionally, SP followed by vibro-polishing (SPV) samples were also prepared to understand their combined effect on surface topography and microstructure evolution. In SP, spherical cast steel shots (S110H, 0.29 mm diameter) of 5.2 GPa hardness with 30 ms^{-1} shot velocity were employed. In the vibro-polishing process, plastic media ($\sim 0.3 \text{ GPa}$ hardness as reported by Ahluwalia et al. (2017)) at 6 mm vibration amplitude was employed. In VP, test specimens were rigidly fixed to a trough and submerged in a vibratory chamber filled with steel media of hardness approximately 5 GPa and circulating water (to act as a coolant) as shown in Fig. 1(c). The trough is subjected to the vibration amplitude of 5.5 mm by connecting it to an unbalanced motor. The peening and burnishing actions developed during the movement and flow of media against the specimen surface through normal and shear forces. The experimental set-up is also explained by Feldmann et al. (2014). In the current experimental work, a commercial VP machine supplied by Walther Trowal[®] 175/85 was used. The strain rate was significantly higher in SP ($\sim 10^3 \text{ s}^{-1}$) when compared to VP ($\sim 10^{-2} \text{ s}^{-1}$).

Fig. 1: Schematic diagram of (a) SP, (b) VP and (c) VP test setup (both types of media were significantly larger than the grain size (1-20 μm))

For the thermal relaxation experiments, the surface treated samples were heated to 650 $^{\circ}\text{C}$ with a ramp rate of 5 $^{\circ}\text{C min}^{-1}$, dwelled for 5 h and slowly cooled (5 $^{\circ}\text{C min}^{-1}$) to room temperature in a vacuum furnace at 10^{-8} MPa. The temperature was monitored throughout using a Pt thermocouple with an accuracy of ± 0.1 $^{\circ}\text{C}$. This thermal exposure state represents that of a typical operating temperature condition of high-pressure turbine discs as reported by Foss et al. (2013).

2.3 Surface Topography

Taylor Hobson[®] profilometer was used for surface roughness (R_a) measurement. Areal topographic measurements (S_a) were performed using Taylor Hobson CCI white light interferometry with 20X objective lens. A raster scan was performed using the X-Y stage on 2x2 mm^2 surface area followed by map stitching. Three repeat measurements were made at each

location to ensure the accuracy and precision of values and later used to calculate the standard deviation of each topographic descriptor.

2.4 Phase and Dislocation Density Analysis using XRD

Phases were identified using X-ray diffractometer (PANalytical Empyrean[®]) using a copper (Cu) source with an incident X-ray wavelength (λ) of 1.54 Å. From the XRD pattern, the full-width half-maximum (FWHM) value was calculated for every peak from 30° to 100° 2θ angles and the contribution of instrumental broadening was removed from the FWHM values. To ascertain the anisotropic behaviour of polycrystalline Ni-based superalloy, Waspalloy, Stone et al. (1999) found that elastic constant of (311) plane is more representative of bulk behaviour and insensitive to intragranular strain as compared to any other planes. A similar observation was made by Aba-Perea et al. (2016) while measuring elastic constants at high temperatures for Udimet 720Li and IN718. Thus, it can be said that the FWHM and residual stresses calculation from (311) plane is representative of the bulk defect concentration. Further, high 2θ angle of (311) plane has greater sensitivity to peak broadening with influencing factors when compared to those at low 2θ angles. Hence it is considered for residual stress and FWHM analysis.

Dislocation density, micro-strain and crystalline size were calculated from the XRD data using modified Williamson-Hall method as described by Ungár et al. (1998). According to them the FWHM line profile can be represented as:

$$\Delta K = \frac{0.9}{D} + \Delta K^D \quad (1)$$

where ΔK^D is the strain contribution to line broadening and D is the average crystallite size, $K = \frac{2 \sin \theta}{\lambda}$, $\Delta K = \frac{2 \cos \theta (\Delta \theta)}{\lambda}$, θ and λ are the diffraction angle and the wavelength of X-rays, respectively. The strain contribution (ΔK^D) in terms of Burgers vector (b), the dislocation

density (ρ), the average contrast factor ($\bar{C}$), by neglecting the higher order terms is given by (see Ungár et al. (2001)):

$$\Delta K - \beta' W(g) = \frac{0.9}{D} + \sqrt{\frac{\pi \rho A}{2}} K \sqrt{\bar{C}} b \quad (2)$$

where A is anisotropic factor which is 0.66 as calculated from the approach presented by Wilkens (1970). Guided by the work of Ungár et al. (1998) and Warren (1959) for FCC Ni, the values of $K \sqrt{\bar{C}}$ are taken as 1.608, 3.240, 3.064 and 4.088, and the values of $W(g)$ are 0.43, 1, 0.71 and 0.45 for $\{111\}$, $\{200\}$, $\{220\}$ and $\{311\}$ reflections respectively. The values of $K \sqrt{\bar{C}}$ are based on the assumption that the dislocation population is 50 % edge and the remaining screw in nature and β' is taken as 0.005 for the correction of stacking faults from FWHM.

2.5 Residual Stresses and FWHM Analysis

Residual stresses were measured using Stresstech XStress G3 XRD equipment via the d vs $\sin^2 \Psi$ method. A manganese radiation ($\lambda = 2.1031 \text{ \AA}$) was used to get the diffraction from (311) crystal lattice plane positioned at a 2θ angle of 156° which represents the bulk properties of the γ/γ' structure, as reported by Ardi et al. (2016). Kim et al. (2005) have measured the Udimet720Li Young's modulus (E) and Poisson's ratio (ν) to be 208 GPa and 0.31, respectively. A plot between interplanar spacing (d) vs $\sin^2 \Psi$ was obtained by varying Ψ values from -36° to 39° and linear regression was used to quantify residual stresses. Electrolytic polishing using a mixture of 90% methanol by vol. and 10% by vol. perchloric acid at 25 V was employed to remove the layer successively to get the residual stress distribution through the thickness (up to 200 μm). A stylus profilometer (Talysurf®) was used to measure the depth of the thickness removed. The diffraction peak profile at each depth was fitted using Pseudo-Voigt function and the FWHM value is calculated after correcting for the instrumental broadening using the relation shown in equation (3) (see the work of Suryanarayana and Norton (1998) for further details):

$$w^2 = (w_{total} - w_i) \sqrt{w_{total}^2 - w_i^2} \quad (3)$$

where, w is the sample peak broadening due to crystallite size and lattice strain, w_i is the instrumental broadening and w_{total} is the combination of sample and instrumental peak broadening.

2.6 Micro-hardness Testing

Micro-Vickers hardness was measured using a Qualitest QV-10000 DM hardness machine. A Vickers diamond pyramid with an included angle of 136° (standard one) has applied 200g load over a dwell time of 10s. Hardness along the through-thickness direction (i.e., from the peened surface to the bulk of material) was measured by making indentations spaced approximately 10-20 μm apart from the polished surface. No two indents were spaced less than 2.5 times the length of the indent diagonal during the test. Three repeat measurements were made to calculate the average hardness value.

2.7 Microstructural Investigation of Plastically Strained Region using EBSD

The EBSD analysis was carried out using Oxford Instruments[®] detector positioned within FESEM JEOL-JSM 7600F. All the samples were cut using electric discharge machining from the work-piece to a size of 5 mm x 5 mm with 3 mm thickness and subjected to fine polished using diamond paste (detailed EBSD sample preparation method is described in detail by Child et al. (2011)). Additionally, electrolytic polishing by the A2 etchant consisting of 90% by vol. methanol and 10% by vol. perchloric acid at 25 V for five secs was employed for EBSD analysis. A scanning area of 150 x 150 μm^2 was obtained using a step size of 0.3 μm , generating 3×10^5 data points. AZtec[®] software and Tango map were used for the analysis of GOS, grain size distribution, and interface boundary data. For the grain size measurement using EBSD, the

critical misorientation is defined as 10° and special boundaries such as twins in the grains were disregarded.

EBSD was also utilized for estimating the depth of plastically strained region using grain orientation spread (GOS) values. High GOS values are analogous to severe plastic deformation of the material as mentioned by Foss et al. (2013) (illustrated in Fig. 2(a)). Hence, the GOS values of each grain with respect to their depth from the treated surface were plotted (see Fig. 2(b)). Within the experimental scatter, the variation of GOS with depth is fitted to a fourth order polynomial. The depth of plastically strained region is defined as the point where the slope of the polynomial function is zero i.e. first order derivative of a polynomial equation is zero (see Fig. 2(c)). The raw data was analyzed with Tango map and exported in the text file which includes x , y position of grains and corresponding GOS value. Four different areas were scanned on two samples with the same process intensity to calculate the mean depth of plastically strained region and its standard deviation.

Fig. 2: Schematic diagram of (a) EBSD scan area (b) GOS vs depth and (c) first derivative of GOS vs depth

2.8 Plastic Strain Estimation using XRD

Plastic strain of treated specimens was estimated using the measured variation of FWHM with plastic strain from a uniaxial tensile tests, as suggested by Prevy (1987). In this method, tensile

specimens of untreated material as per ASTM E8 standard were tested for known plastic strain values and corresponding FWHM values were noted (see Fig. 14). Three tensile specimens at each of the plastic strain value were used to calculate the average FWHM value. An empirical equation was obtained by fitting the data points using second-order polynomial function and used for calculating the plastic strain in shot peened and vibro-peened samples. However, this method is based on the assumption that change in FWHM due to plastic strain is independent of deformation mode and crystallite size variation. A similar strategy was adopted by Li et al. (2009) and Kim et al. (2005).

3 Results

In this section, the experimental measurements of surface topography, phase and microstructural characterizations, compressive residual stresses and plastically strained region analysis are presented.

3.1 Surface Topography

Inherent to the surface and sub-surface modification processes of SP and VP, the surface roughness increases. The initial surface roughness of $R_a = 0.01 \mu\text{m}$ increased to $0.71 \mu\text{m}$ and $0.26 \mu\text{m}$ respectively, after SP and VP processes as shown in Fig. 3. Roughness is higher in the case of SP because the impingement of high-velocity shot particles generates overlapping micro-indentations on the surface (see Nordin and Alfredsson (2016)). In VP, roughness is relatively lower as it involves both the peening and burnishing effect. The burnishing effect (shear action) is due to the movement and flow of steel media working against the specimen surface. Additionally, the media is larger in size for VP, so indents are shallower. Vibro-polishing on shot peened coupons (SPV) has been performed to achieve roughness comparable to vibro-peened coupons of $R_a = 0.26 \mu\text{m}$, which is close to surface roughness recommended for good aerodynamic efficiency. As listed in Table 2, the measured R_a and S_a values are very close to one

another, which show a uniform surface finish. Child et al. (2011) have also measured similar S_a value ($\sim 0.77 \mu\text{m}$) for shot peened Udimet720Li at an Almen intensity range of 4-6 A.

Fig. 3: Surface profilometry (3D) images of (a) O (b) SP (c) SPV (d) VP (e) SPHT (f) SPVHT and (g) VPHT sample conditions

Table 2: Surface roughness R_a and S_a values after the original and peened samples

Process	R_a (μm)	S_a (μm)
O	0.01 ± 0.013	0.01 ± 0.011
SP	0.71 ± 0.012	0.72 ± 0.026
SPV	0.27 ± 0.021	0.29 ± 0.012
VP	0.26 ± 0.011	0.28 ± 0.018
SPHT	0.54 ± 0.023	0.55 ± 0.021
SPVHT	0.30 ± 0.014	0.29 ± 0.023
VPHT	0.28 ± 0.017	0.27 ± 0.013

After thermal exposure, the surface topography remained the same as before. The slight variation that is observed in the SPHT condition could be due to the formation of thin oxide layer during the post heat treatment. There is no significant variation in SPVHT and VPHT condition because the peak to valley height is low, hence thin oxide layer did not affect the roughness or within the experimental scatter.

3.2 Phase and Microstructural Characteristics

FWHM represents the XRD peak broadening which signifies the presence of plastic strain because of the total dislocation density arising from the geometrically necessary and statistically stored dislocations in the material. The peak broadening was observed through diffraction pattern analysis for all crystallographic planes, i.e. (111), (200), (220) and (311). The percentage increment in FWHM is higher for high 2θ angle planes such as (311) compared to those at low 2θ angle planes such as (111). Similarly, the percentage reduction in FWHM after thermal exposure is significant in (311) plane when compared to (111) plane (see Fig. 4). The change in FWHM of (311) after thermal exposure is 22%, 27% and 15% in SP, SPV, and VP respectively.

After thermal exposure, dislocation annihilation and recovery of microstructure could lead to decrease in FWHM. Overall, the change in FWHM is remarkable at high 2θ angle plane so the (311) plane was selected for residual stress analysis. Furthermore, no additional phases were observed after any of the surface treatments and thermal exposure.

Fig. 4: XRD pattern of untreated and treated Udimet720Li samples

Table 3 summarises the calculated dislocation densities, micro-strain and crystallite sizes using modified Williamson-Hall method based on the (111), (200), (202), and (311) peaks. Herein, the slope and intercept of modified Williamson-Hall plot of FWHM corrected for stacking faults as a function of $K\sqrt{C}$ were used for the analysis (see Figs. 5(a)-(c)). There is maximum 86% increase in dislocation density after SP as compared to the untreated condition ($\sim 1.23 \times 10^{15} \text{ m}^{-2}$). Amongst the treated samples, the dislocation density of SP ($\sim 1.19 \times 10^{16} \text{ m}^{-2}$) is comparable to SPV ($\sim 1.18 \times 10^{16} \text{ m}^{-2}$) and is slightly higher than that of VP ($\sim 8.60 \times 10^{15} \text{ m}^{-2}$). However, following the heat treatment, dislocation densities reduced by 43 %, 56 % and 35 % after SP, SPV and VP respectively. Ortiz et al. (2010) reported the dislocation density of $\sim 1.11 \times 10^{14} \text{ m}^{-2}$ after SP of single phase Ni-base C-2000 alloy when shot peened at 55 ms^{-1} with 350-400% coverage. The dislocation density can reach as high as $\sim 2 \times 10^{17} \text{ m}^{-2}$ after surface mechanical

attrition treatment of Ni-based alloy using vibration frequency of 20 kHz with stainless steel shots of 3 mm diameter as reported by Tao et al. (2004). Similarly, the micro-strain is increased after all surface treatments and reduced slightly after heat treatment. Crystalline size is reduced by 30-35% after all the surface treatments from the pristine sample and slightly increased by 2-6 % after heat treatment. It is to be noted that a grain may consists of several crystallites in a polycrystalline material. Hence, it can be clearly observed that crystallite size reported here is remarkably smaller than EBSD based grain size reported in Fig. 6. Further, XRD crystallite size is calculated from a top few micron layer ($\sim 3 \mu\text{m}$ XRD penetration depth) but the grain size is calculated from top $50 \mu\text{m}$ depth. It is illustrative to compare the crystallite data from the relevant literature. Li et al. (2017) reported that after ultrasonic shot peening of pure nickel at $70 \mu\text{m}$ amplitude and 20 kHz frequency for 10 min, the surface average crystallite size was $\sim 40 \text{ nm}$ measured using TEM. Very recently, Zhao et al. (2018) reported the crystallite size of 40 nm, 58 nm and 81 nm after SP of nickel-aluminium bronze alloy at the Almen intensity of 0.15 mmA, 0.20 mmA and 0.25 mmA, respectively at 200 % coverage using 0.35 mm ceramic shot balls.

Table 3: Dislocation density, micro-strain and crystallite size under different treatment conditions

Process	Dislocation density (m^{-2})	Micro-strain (%)	Crystallite size (nm)
O	$\sim 1.23 \times 10^{15}$	~ 0.89	~ 43.47
SP	$\sim 1.19 \times 10^{16}$	~ 2.76	~ 28.30
SPV	$\sim 1.18 \times 10^{16}$	~ 2.75	~ 28.48
VP	$\sim 8.60 \times 10^{15}$	~ 2.35	~ 30.50
SPHT	$\sim 6.73 \times 10^{15}$	~ 2.08	~ 29.80
SPVHT	$\sim 5.16 \times 10^{15}$	~ 1.82	~ 30.30
VPHT	$\sim 5.62 \times 10^{15}$	~ 1.90	~ 31.03

Fig. 5: The modified Williamson-Hall plot of FWHM corrected for stacking faults as a function of $K\sqrt{c}$ for (a) O, (b) VP and (c) VPHT samples

Grain Size and its Distribution: The surface treatment strengthens the material locally due to the grain refinement. The grain size and its distribution along the depth were quantified through EBSD technique by equivalent circle diameter method. As depicted in Fig. 6, there is a reduction in average grain size through the thickness: after the surface treatment, overall, the grains with 1-2 μm increases as shown in the frequency map. It can be observed from the Figs. 6(b), (c) that the image quality on the top edge is lower and not all points are indexed, it could be due to the accumulation of plastic strain and localised defects. After thermal exposure, there is no significant change in grain size distribution is observed or within experimental scatter.

Fig. 6: Grain size distribution map of (a) O (b) SP and (c) VP (inset histogram represents the grain size data from top 50 μm)

Interface boundaries: Interface boundaries play a critical role in determining the mechanical properties of the material. The strengthening is not only contributed by grain refinement but also due to the controlled creation of internal defects and grain boundaries to resist the dislocation motion, though they consist a small fraction of material's volume as reported by Seita et al. (2016). Tan et al. (2006) have shown that coincidence site lattice (CSL) boundaries improve the mechanical performance as they are less susceptible to damage, creep cavitation, or corrosion resistance.

Fig. 7: Cross-sectional EBSD images showing LAGBs and HAGBs in (a) O (b) SP and (c) VP

Fig. 8: Fraction of (a) LAGBs and HAGBs and (b) special boundaries (Twins (CSL $\Sigma 3$) and low- Σ CSL($\Sigma 3 - 29$))

Furthermore, the fraction of low angle grain boundaries (LAGBs) ($1^\circ < \theta \leq 15^\circ$) was 12% in the pristine samples which increased to 37%, 54% and 53% after SP, SPV and VP respectively with the corresponding decrease in high angle grain boundaries (HAGBs) ($\theta > 15^\circ$) as shown in Figs. 7(b), (c) and 8(a). This increase in LAGBs contributes to the hardening effect after treatment. Valiev (2004) had reported that grain boundary sliding is relatively more difficult in LAGBs, and hence leading to lower ductility. Alyousif et. al. (2008) have reported 21% increase in LAGBs from an initial 4% value on the surface of 304 stainless steel after SP. After thermal exposure, the fraction of LAGBs remains unaffected or within the experimental scatter of 54%, 36%, and 51% in SPHT, SPVHT and VPHT conditions, respectively.

Figure 8(b) shows the percentage of twin boundaries (TBs) (CSL $\Sigma 3$) and Low- Σ CSL ($\Sigma 3 - 29$) boundaries reduces in all the surface treatment conditions considered in this work from the original raw material. Lu et al. (2009) and Tan et al. (2006) have shown that TBs (CSL $\Sigma 3$) are more mechanically and thermally stable than traditional HAGBs due to their low energy, which is $1/50^{\text{th}}$ of random boundary: hence, this is detrimental to the surface treatment.

3.3 Compressive Residual Stresses

Surface compressive residual stress value 91 MPa of pristine sample increased to 826 MPa, 907 MPa and 640 MPa for SP, SPV and VP surfaces respectively, giving a difference of approximately 180 MPa (between SP and VP) (see Table 4). The variation of residual stresses across the specimen depth is shown in Fig. 9(a) for all the three processes SP, SPV and VP: the peak compressive residual stresses of 1094 MPa, 1099 MPa, and 874 MPa respectively occurred at a depth of approximately 20 μm which is almost $1/20^{\text{th}}$ of shot diameter used in SP. Residual stresses of SP and SPV are higher compared to VP up to changeover point ($\sim 60 \mu\text{m}$), the reverse is true after this point. The depth of influence (DOI) is 140 μm , 160 μm and 200 μm in SP, SPV and VP respectively, the transition to balancing tensile stresses takes place beyond this point. A similar residual stress distribution trend was observed for the vibro-peened IN718 material by Feldmann et al. (2014).

In VP, the higher DOI could be due to the intense plastic deformation which is induced by the energy transfer of high-frequency vibratory media, working against the surface as explained by Kudryavtsev et al. (2002) or larger media size when compared to SP. This energy is dissipated by stress waves which usually propagate from the initiation site deep into the material and generates minor indents on the affected surface. Compressive residual stresses are maximum at sub-surface rather than the top surface in both vibro-peened and shot peened samples as per the Hertzian contact theory predominantly, as explained by Schiffner and Helling (1999). The position of peak residual stresses depends on the shot size, shot velocity and hardness of the workpiece. The magnitude of compressive residual stresses decreases after reaching peak-value and turns to balancing tensile stresses.

Fig. 9: Depth profiling of compressive residual stresses (a) before and after thermal exposure and (b) % stress relaxation

It can be observed from the depth profile, the distribution of compressive residual stresses decreases gradually from sub-surface into bulk of the material. But, the rate at this drop occurs in SP is higher than the VP (see Fig. 9(a)). This difference could be due to the nature of the residual compressive stress generation mechanism, i.e., kinetic energy transfers in SP and vibrational energy transfer in VP.

Table 4: Values of surface residual stresses, peak residual stresses, peak residual stress position, and depth of influence (DOI)

Processes	Surface residual stresses (MPa)	Peak residual stresses (MPa)	Peak residual stress Position (μm)	DOI (μm)
O	-91	-159	~20	~75
SP	-826	-1094	~21	~140
SPV	-907	-1099	~22	~160
VP	-640	-874	~37	~200
SPHT	-328	-715	~39	~140
SPVHT	-361	-715	~34	~160
VPHT	-376	-655	~55	~200

After thermal exposure, surface residual stresses and peak residual stresses are relaxed in all treated conditions. The relaxation in surface residual stresses is 60.30%, 60.20%, and 41.25% and peak residual stress is 34.64%, 34.94%, 25.05% in SP, SPV and VP respectively. Surface

residual stress relaxation is higher when compared to peak residual stress relaxation (see Fig. 9(b)) because the top surface is exposed directly to the high-temperature environment and has higher plastic strain (see Fig. 10). High plastic strain percentage indicates a large amount of stored energy which is an unstable material state. Hence, it relaxes the residual stresses to attain the equilibrium state. In other words, the dislocation density is higher at the surface compared to the bulk material so dislocation annihilation rate is higher at the surface. Additionally, variation in the rate of stress relaxation from the surface to sub-surface induces a peak shift towards the bulk material. Peak shifts from 21 μm to 39 μm , 22 μm to 48 μm and 37 μm to 55 μm in SPHT, SPVHT, and VPHT respectively. A similar observation on the thermal stress relaxation was reported in shot peened superalloys RR1000 by Foss et al. (2013) and IN718 by Khadhraoui et al. (1997). Further, the rate of stress relaxation is higher in SP with depth as compared to the VP. However, the DOI of residual stresses remains the same because the plastic strain percentage in bulk material is relatively less, so there is negligible stress relaxation taking place as shown in Fig. 9(b).

3.4 Plastically Strained Region Analysis

The hardening effects of the target material are examined through four competing methods namely GOS, micro-hardness, FWHM and plastic strain.

Grain Orientation Spread (GOS): High GOS values are related to the increased plastic deformation and vice versa. The data can also be displayed visually using orientation imaging microscopy (OIM) to show maps of the scanned area (see Figs. 10(a)-(g)). Using statistical analysis as explained in section 2.7, depth of plastically strained region was estimated (see Fig. 2 and Figs. 11(a), (b)).

Fig. 10: GOS images of (a) O (b) SP (c) SPV (d) VP (e) SPHT (f) SPVHT and (g) VPHT sample conditions by EBSD

Fig. 11: GOS distribution of VP (a) fourth order polynomial fitting and (b) first derivative of a polynomial function

The depth of plastically strained region is almost negligible in the original sample, and it is 60 μm , 70 μm , 85 μm under SP, SPV, VP conditions respectively as depicted in Fig. 10. Child et al. (2011) reported 47 μm DOI for shot peened Udimet720Li at 4-6 A intensity. In SPV process, 16% increase in depth of plastically strained region when compared to SP is due to high vibrational amplitude (~ 5.5 mm) employed in vibro-polishing similar to VP process; surprisingly the polishing process is hardening the microstructure in addition to polishing. However, in the conventional low amplitude (2-3 mm) vibro-polishing process depth of plastically strained

region should be negligible. Furthermore, the GOS depth variation after thermal relaxation was within the measurement scatter range for the SPHT process, whereas for SPVHT and VPHT it was reduced by approximately 5-8% which is 55 μm , 68 μm , 80 μm respectively. There could be a significant generation of dislocations after surface treatment across the cross-section, hence the variation in depth of plastically strained region can be easily observed.

Micro-Hardness: The micro-hardness variation through the thickness is similar to the GOS distribution, it is mainly because both originate from the dislocation generation and interaction effect. Even at the low peening intensities employed, there is a slight refinement of the grain structure, and hence we expect strengthening effects according to the Hall-Petch relationship. The average surface micro-hardness of the original sample is 5.24 GPa which increased to 6.81 GPa, 6.57 and 6.56 GPa after SP, SPV and VP respectively as depicted in Fig. 12(a). This implies 30%, 25.4% and 25.2% increase in hardness after SP, SPV, and VP respectively from the original sample. The micro-hardness value decreases steeply from the surface to sub-surface in each condition, i.e., shot peened, vibro-peened reaching different DOI.

The DOIs of micro-hardness values and compressive residual stresses of the material are comparable to each other, i.e., it can be observed that both micro-hardness and compressive residual stresses both are effective upto $\sim 150\ \mu\text{m}$ depth.

Fig. 12: Depth profiling of micro-hardness (a) before and (b) after thermal exposure

After thermal exposure, the surface hardness reduced to 6.52 GPa, 6.51 GPa and 6.47 GPa in SPHT, SPVHT and VPHT respectively as shown in Fig. 12(b). The reduction in surface hardness could be due to thermal relaxation of stored strain energy and dislocation annihilation mechanism. However, the depth of influence remains the same at 150 μm , 145 μm and 205 μm (within experimental scatter) after thermal exposure in SPHT, SPVHT and VPHT respectively.

Full-width half maxima (FWHM): FWHM is obtained from the XRD measurements and represents the peak broadening under plastic deformation and could be analogous to plastic strain or micro-hardness variations. From Figs. 13(a), (b), show that the surface has higher FWHM than the sub-surface as peening generates higher plastic strain and dense dislocation network on the surface. The thermal exposure causes a reduction in FWHM value which could be due to the recovery of plastic strain and dislocation annihilation mechanism. Compressive residual stresses and FWHM distribution throughout the depth is not similar which signifies both are not directly correlated with each other. Furthermore, the micro-hardness value and FWHM of peaks follow the similar decreasing trend from a peak value to a saturation value in the bulk material.

Fig. 13: Depth profile of FWHM (a) before and (b) after thermal exposure

Plastic Strain: It was calculated based on the empirical equation fitted to the calibrated plastic strain versus FWHM response from the uniaxial tension test as shown in Fig. 14 (refer to section 2.8 for a detailed explanation). Fig. 15(a) show that SP (13.71%) and SPV (13.63%) induces a higher plastic strain in the material compared to VP (8.15%). Additionally, the depth of plastically strained region is high for SPV (~60 μm) and VP (~60 μm) when compared to SP (~50 μm). It can be observed from Fig. 15(b) that thermal exposure relaxes the plastic strain significantly and its values are relatively close to one another in all heat-treated conditions except for the top surface of VPHT (~6.35%). The depth of plastically strained region after thermal exposure is highest in VPHT at ~65 μm , and is ~55 μm and ~50 μm , respectively for SPVHT and SPHT processes. The plastic strain relaxation is low in VP (22%) compared to SPV (66%) and SP (69%), which shows a better thermal stability. A minimum plastic strain is required in the material for stress relaxation to occur by thermal energy.

Fig. 14: Empirical curve relating FWHM of (311) to plastic strain for Udimet720Li

Fig. 15: Plot showing plastic strain (a) before and (b) after thermal exposure

Overall, a variation of GOS, FWHM, micro-hardness, and plastic strain with sub-surface depth shows similar trend lines, though their influencing zone differs slightly from one another.

4 Discussion

A comparative study of the depth of influence (DOI) of individual parameters is discussed here in detail. It can be observed from the Table 5 that the DOI of individual parameters is different for the same process using various assessment techniques. The DOI of micro-hardness and FWHM is similar and showing a good correlation to each other. Similarly, GOS and plastic

strain also shows a similar pattern. The DOIs for GOS, plastic strain, micro-hardness, and FWHM is calculated based on the value where it saturates or reaches equal to the untreated condition. However, the DOI for compressive residual stresses is calculated based on its absolute value where its magnitude becomes close to zero.

It is to be noted that the EBSD technique has some limitations for indexing in the highly deformed zone. The GOS value underestimates the depth of plastically strained region as compared to micro-hardness or FWHM method. GOS is highly sensitive to each pixel orientation and average grain misorientation and limited to index low misorientation which could be due to the presence of dislocations and strains generated by the surface treatment process. It is observed that GOS never reaches zero for both untreated and treated samples, and further, the saturated GOS value is slightly higher for treated sample when compared to the untreated one. The non-zero GOS value shows that there is always the presence of small misorientation within the grain. Humphreys (2001) reported non-zero GOS value to the influence of orientation noise. Another reason could be due to the limitation of EBSD to accurately index the low grain misorientation angles ($\theta < 1^\circ$). On the other hand, in relatively small grain sizes material, residual stress measurement using XRD is quite reliable since the incident probe samples over a significant number of randomly oriented grains. As a result of this greater confidence could be placed on the DOI obtained through the FWHM technique.

Comparing DOI of compressive residual stresses, the SP and SPV show almost similar trend but the VP shows higher DOI for each parameter. For example, compressive residual stresses are effective till 200 μm in VP while it is 140 μm and 160 μm in SP and SPV respectively. After thermal exposure, DOI value remains unchanged or within experimental scatter for all three processes i.e. for SP, SPV and VP.

Table 5: Comparison of depth of influence (DOI) using various methods

DOI (μm)					
Processes	GOS	Plastic Strain	Micro-Hardness	FWHM	Compressive residual stresses
O (Baseline)	~ 0	~ 0	~ 0	~ 60	~ 75
SP	~ 60	~ 50	~ 140	~ 110	~ 140
SPV	~ 70	~ 60	~ 145	~ 140	~ 160
VP	~ 85	~ 60	~ 200	~ 155	~ 200
SPHT	~ 55	~ 50	~ 150	~ 110	~ 140
SPVHT	~ 68	~ 55	~ 145	~ 140	~ 160
VPHT	~ 80	~ 65	~ 205	~ 150	~ 200

Possible correlation among the micro-hardness, FWHM and GOS for all treated surface and the thermal exposed surface are investigated at equivalent depth from the treated surface ($\pm 5 \mu\text{m}$) for each sample. Using these correlation relationships (see equations (4) and (5)), one parameter value can be calculated from another parameter within 10% deviation from the actual measurement. It can be observed from the curve fitting with the power law regression between micro-hardness and FWHM plot (Figs. 16(a)-(f)) that both are analogous to each other. However, the slope of the fit is changing from the surface to the bulk material. The correlation function is,

$$\text{Micro-hardness} = a (\text{FWHM})^b \quad (4)$$

where the values of a and b are influenced by surface treatment conditions and varying in the range of $5.58 \leq a \leq 5.92$ and $0.11 \leq b \leq 0.13$

The correlation between micro-hardness (HV) and FWHM (major contribution from micro-strain) is good as the peened sample strengthens due to generated dislocations. It is to be noted that the slope of the fit between micro-hardness and FWHM, $(d(HV)/d(w))$, decreases as

FWHM value increases. This is due to the influence of peak compressive residual stresses on micro-hardness at the sub-surface ($\sim 25 \mu\text{m}$ from the surface), as reported by Nagarajan et al. (2018). Furthermore, the GOS and FWHM are correlating well and also fitted using a power law regression with a constant term possibly due to orientation noise. A reverse trend was observed for the slope of the curve the variation of GOS with FWHM i.e. $d(\beta)/d(w)$ when compared to micro-hardness versus FWHM curve. The correlation function is defined as:

$$\text{GOS} = c + d (\text{FWHM})^e \quad (5)$$

where, the values of c , d and e are influenced by surface treatment conditions and varying in the range of $0.26 \leq c \leq 0.50$, $10^{-4} \leq d \leq 0.02$ and $2.48 \leq e \leq 8.25$

At the top surface, GOS values are relatively high which may be easily detected by EBSD and low at the bulk material. This could be due to the inability of EBSD tool to resolve the zero or low misorientation ($\theta \leq 1^\circ$) data points especially at bulk material where strain is relatively low or zero. Also, GOS misorientation data is based on the crystal rotation, and may not be considering the effect of dislocations and stacking faults for the strengthening. Overall, it shows that FWHM is not only the result of micro-hardness or GOS, several other factors such as dislocations, stacking faults contribute to it. However, considering systematic error involved in each method used to analyze the data, no value should be considered as being absolute. Some other researchers also studied the correlation, for example, Soady et al. (2013) correlated the plastic strain with hardness and FWHM using power law regression for shot peened martensitic steel. Similarly, Srikant et al. (2006) correlated hardness with plastic strain using a power law regression. In both studies, the slope of the curve is changing between plotted parameters, but the correlation is still existing. Whereas Foss et al. (2013) reported a linear correlation among GMM, FWHM, and micro-hardness for shot peened Ni-based superalloy RR1000.

Fig. 16: Correlation among Hardness, FWHM and GOS of (a) SP (b) SPV (c) VP (d) SPHT (e) SPVHT and (f) VPHT

Fig. 17: Correlation among compressive residual stresses, micro-hardness, FWHM, GOS and plastic strain of (a) SP and (b) VP

Overall, the trend lines for plastic strain, FWHM, micro-hardness, and GOS are almost similar which shows higher value at the surface and gradually decreasing towards the depth as shown in Figs. 17(a), 17(b). However, compressive residual stresses show a slight increment in the sub-surface up to 50 μm deep and then starts to decrease.

5 Conclusions

In this experimental research, we assessed the effect of SP and VP on the evolution of microstructure in Ni-based superalloys and properties at the surface and sub-surface level for the same peening intensity. Peening influence on grain size distribution, grain boundaries such as LAGBs, HAGBs, and TBs was presented. The depth of plastically strained region was measured from the GOS variation with the depth as explained in section 2.7 and Fig. 2(c).

The major conclusions from this study are as follows:

1. The surface roughness of SP ($S_a = 0.72 \mu\text{m}$) was remarkably high compared to SPV ($S_a = 0.29 \mu\text{m}$) and VP ($S_a = 0.28 \mu\text{m}$) and remains unaffected by thermal exposure.
2. The microstructural analysis reveals the significant addition of LAGBs (CSL $\Sigma 1$) and grain refinement after SP and VP process which could be contributing for high micro-

hardness. However, there was slight reduction in the twin grain boundaries (CSL $\Sigma 3$) and low- Σ CSL ($\Sigma 3 - 29$) boundaries in the material.

3. It was observed that the magnitude of compressive residual stresses induced by VP (~640 MPa) is relatively low compared to SP (~826 MPa), but the converse is true for depth of influence (DOI). Compressive residual stresses after reaching a peak at sub-surface drop more gradually in the case of VP than SP. After thermal exposure, the stress relaxation is higher in SP (~60%) when compared to VP (~41%) without any change in the DOI.
4. Plastic strain obtained from VP (~8%) was found to be significantly lower than SP (~14%). However, depth of plastically strained region is higher in VP when compared to SP.
5. The surface micro-hardness increases by 30% and 25.2% in SP and VP respectively and decreases gradually from the surface to the sub-surface depth. However, the rate of decrease is higher in the SP process than VP process; it shows that the hardening effect is gradually distributed towards the bulk material. After thermal exposure, a slight reduction in micro-hardness is observed accompanied by a reduction in dislocation densities for all type of treatments SP, SPV and VP.
6. The micro-hardness, GOS, plastic strain, and FWHM correlate well with each other irrespective of treated conditions and fitted using power law regression.

Overall, a combination of deeper compressive residual stresses with low plastic strain and good surface finish is obtained by vibro-peening (VP) process when compared to shot peening (SP) process for the same intensity. Additionally, the VP process shows a better residual stress stability at elevated temperatures. Therefore, the VP process is a viable alternative to treat the aero-engine components operating at elevated temperatures.

Acknowledgments: Authors thank Nanyang Technological University Singapore (NTU), Advanced Remanufacturing and Technology Centre (ARTC) and Rolls-Royce for providing

resources, facilities and financial support (Project agreement number SKMBT 42316032113520 between ARTC and NTU). Dharmesh Kumar thanks NTU Singapore for the financial assistance in the form of research scholarship. Authors' thank Mr. Iain Parr from Rolls-Royce, Dr. Dennise Tanoko Ardi and Dr. Shashwat Shukla from ARTC for scientific discussions, Dr. Ayan Bhowmik from Rolls-Royce@NTU Corporate Lab for XRD data analysis and Dr. Wu Jiang from Oxford Instruments for valuable inputs in the EBSD characterization work.

Conflict of Interest

The authors declare that they have no conflict of interest.

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