

## Nanocrystallisation on the surface of superalloy In718 component with a commercial shot peening process

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### Introduction

Since the early of 2000s, there has been significant interest in surface nanocrystallisation using the so-called surface mechanical attrition treatment (SMAT) [1]. Ultrasonic shot peening is one of the most common processes that functions at a frequency ranging from 50Hz to 20kHz, and has been proved effective to create nanograin structures on a variety of metallic materials: stainless steel, Ti-6Al-4V, aluminium alloys and Ni-based superalloys etc.[2,3]. However, ultrasonic peening has process limitations where complex features exist. Recently the conventional shot peening process has also been found capable to produce nanostructures on a metal surface [4, 5, 6], however, most studies were carried out in a laboratory scale, and there is a lack of systematic study on how to adopt this surface nanocrystallisation process to a large scale production using a commercial shot peening process. In industry, the process of shot peening is usually set up and established to treat a component surface by imparting large compressive stress into the surface of parts therefore improving fatigue life. By development of shot peening parameters, specifically shot size, shot energy and coverage, a successful adoption of the current commercial process can achieve a Nanocrystalline surface producing a step change and further enhancing part performance. Inconel 718 is a workhorse Ni-based superalloy often made into structural components subjected to various service conditions such as dynamic loading, fretting fatigue, wear and corrosive environments, all of which are sensitive to the microstructure and properties of the component surface. A nanocrystallized surface with grain size of 12 nm up to a depth of about 90 microns was created with an ultrasonic peening process in a peak aged Inconel718 superalloy [3]. However, there is a lack of information regarding a commercial shot peening process to produce a nanocrystallized surface in Inconel 718.

### Objectives

The present investigation aims to produce a nanocrystalline structure by utilising the current commercial shot peening process for volume production of highly stressed components made of Inconel 718 superalloy used in the oil industry.

### Methodology

The as-received Inconel 718 superalloy was shot peened with a commercial shot peening process using a Sandwell 5 axis shot peening machine. Shot velocities were measured using a Tecnar G3 Shotmeter, the processing parameters are listed in the Table 1. Two levels of kinetic energy from 1.90 to 2.34 Joules and three levels of coverage from 200% up to 1500% were applied to two groups of samples. Chemical compositions (wt.%) of the as-received Inconel 718 material is composed of 53.55Ni, 18.5Cr, 17.37 Fe, 5.28Nb, 2.91Mo, 0.54Al, 0.98Ti with other trace elements.

The samples for SEM and Optical Microscopy study were polished to 0.05 microns with Struers's OPS polishing suspension and then electrolytically etched with saturated oxalic solution at 6V. Microstructure and deformation bands of the peened samples were examined with Zeiss Imaging microscopy (Observer M2m) using the circular differential interference contrast (C-DIC) function. A Hitachi TM3000 Scanning Electron Microscope (SEM) was used in back-scattered electron mode at 15kV. Transverse slices of electron transparent, vertical to shot peened surface, were sectioned with a FIB/SEM, the size of a foil specimen is around 5x6 microns. Detailed microstructure of the foil specimen was studied with a transmission electron

microscopy (TEM, FEI Talos) at 200kV. Atomic force microscopy (AFM, Bruker Multimode) was used to examine the peened surface, an area of 10 $\mu$ mX10 $\mu$ m was scanned. Microhardness tests were carried out across the transverse section at a test load of 15g holding for 15 seconds.

Table 1 Shot Peening Process parameters

Sample ID	Velocity (m/s)	Kinetic energy (J)	Coverage %	Shot Size*
E	68	1.90	200	ASH280
F	68	1.90	700	ASH280
H	68	1.90	1500	ASH280
I	75.5	2.34	200	ASH280
J	75.5	2.34	700	ASH280
L	75.5	2.34	1500	ASH280

\*Complies with SAE-AMS2431/2E

### Results and analysis

Fig. 1 (a) shows the typical Inconel 718 microstructure for the as-received material showing the generally equiaxed coarse grains with carbides and delta phase distributed along the grain boundaries. Fig.1 (b) and (c) demonstrate, respectively, the general microstructure of shot peened samples at lower and higher coverages. The high density of surface shear bands in various directions was revealed clearly and it reflected the repeated cross slipping in the subsurface region due to plastic deformation induced by shot peening process. The thickness of the strongly deformed surface layer was measured and averaged to be around 56  $\mu$ m, and 160  $\mu$ m, respectively, for the samples peened at 200% and 1500%. According to the simulation study of surface nanocrystallisation by Hassani-Gangaraj [5], the maximum plastic strain occurs in the immediate subsurface layer near the indentation edge where material piles up, as also observed in the present study (Fig.1d). The evidence of the deformation and sub-micron to nanometre sized cell structure were observed with back scattered SEM as shown in Fig.2.

Atomic force microscopy technique was considered a low cost and efficient non-destructive technique to perform the surface characterization with nanometrical resolution without expensive surface preparation. The characterisation of nanostructure on shot peened surface was successfully revealed using AFM as reported by Pompeo [8]. Similar work was conducted in the present study, as shown in Fig.3. The surface nanostructures were evident under AFM on a micro region of the sample peened at 2.34J and at a coverage of 700%, the size of the nanostructures varies from a few to 100 nanometres. Further observations across the section of the shot peened surface were carried out with TEM as described in a later paragraph.

Fig.4 shows the microhardness changes of all shot peened samples as a function of the distance from the top peened surface. It can be seen in general that the hardness value on the top peened surface is near 500HV which is over two times higher than the unpeened central part of the samples with average of hardness around 250HV. The hardness on the top surface of samples peened at higher kinetic energy seems to be only slightly higher comparing to the samples peened at a lower kinetic energy. The effect of the coverage on the hardness change at lower kinetic energy peening seems to be less significant than the samples peened at higher kinetic energy. However, the depth of the hardened layer does not seem to be affected by the coverage change in both low and high kinetic energy, the overall depth of microhardness change is around 400  $\mu$ m for all cases in the present study. Similar results were also found in other metallic materials as reported by Gonzalez [9] for Al6063 alloy where the depth of hardened layer is largely around 500 $\mu$ m, and by Bagherifard [7] for stainless steel AISI316L where the depth of the hardened layer is around 250 $\mu$ m, regardless of coverages changes in shot peening. The significant increase in hardness at the top surface is due to the higher plastic strain and strain rate applied to the top surface layer through shot peening treatment, suggesting the significant increase of the dislocation density at the top surface therefore resulting in the grain refinement in this region according to the Hall-Petch relationship [7, 10]. However, the increased microhardness produced by shot peening was also partly due to the

compressive stress in the peened area. The contribution of the compressive stress to microhardness would be up to around 12% for superalloy In718 [3].

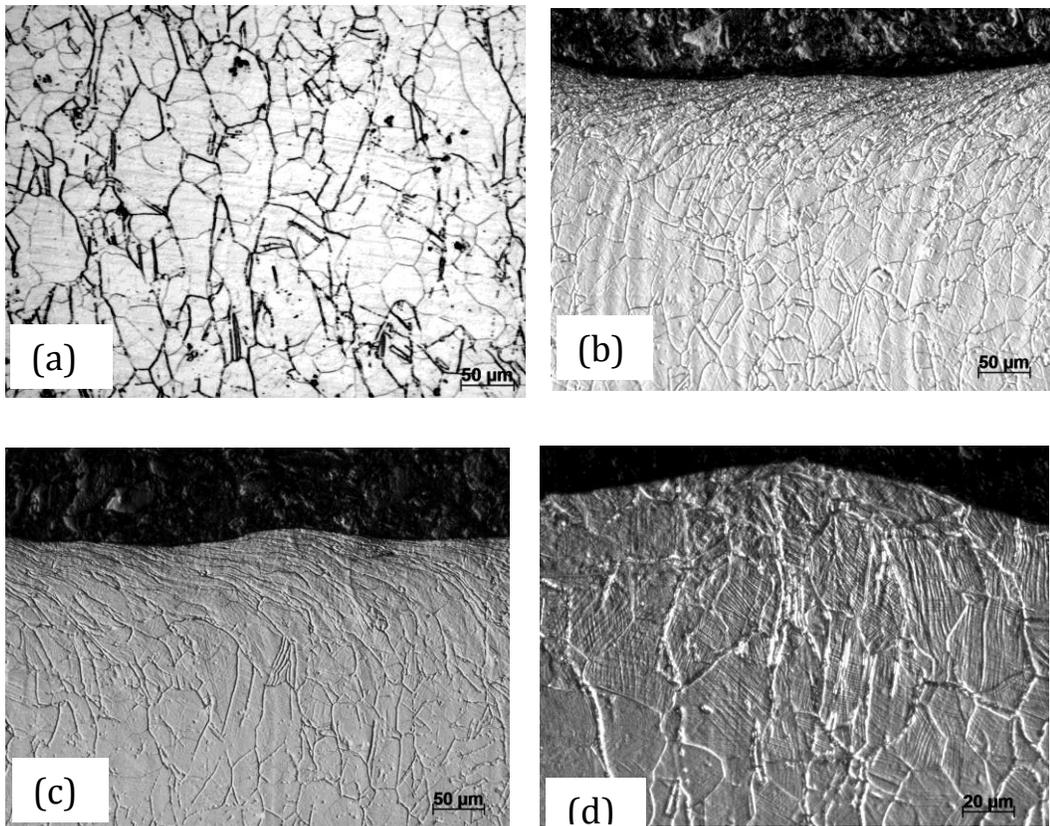


Figure 1 Typical microstructure in un-peened and peened samples at different coverages, (a) normal microstructure before peening, (b) microstructure peened at 200% coverage (2.34J), (c) microstructure peened at 1500% coverage (2.34J) and (d) microstructure at localised pile-up region (2.34J).

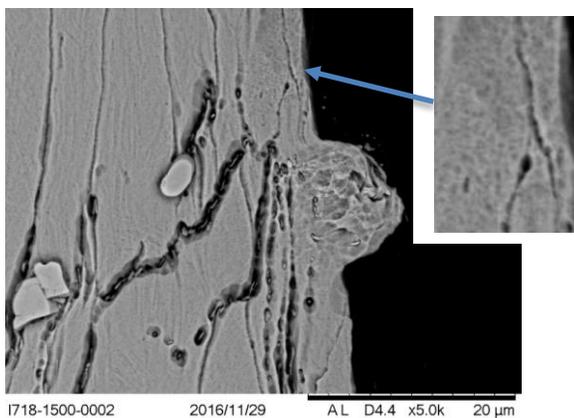


Figure 2 Back scattered SEM observations of the shear bands, (Sample H at a coverage of 1500%)

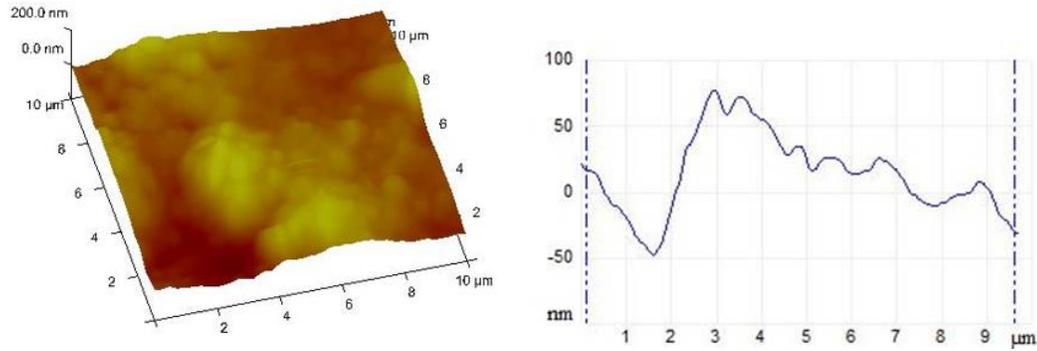


Figure 3 AFM Observations of 10µm x 10µm sized region on the sample J: peened at 2.34J at a coverage of 700%.

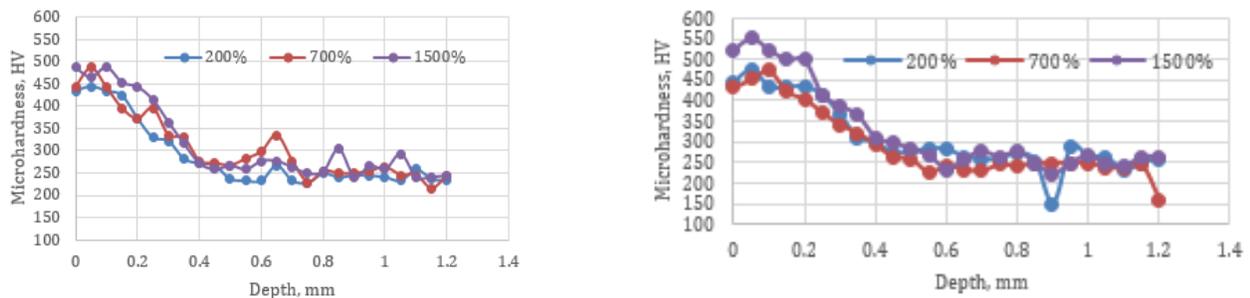


Figure 4 The microhardness distribution as a function of distance from the surface at different kinetic energy of (a) 1.90J and (b) 2.34J

Fig.5 shows the residual stress distributions of all samples peened at different parameters. As expected, a high level of compressive stress were achieved on the surface of all samples. In general, the highest compressive stress location is in the sub-surface at a depth of around 75-150 µm. it is largely consistent with other published data [5, 6]. For the group of samples peened at a lower kinetic energy level of 1.90J (Samples E, F and H), the sample E peened at low coverage of 200% seems to have slightly higher compressive stress than the specimen F and H, peened at 700% and 1500% coverage, respectively. The compressive stress existed in a broader range for the sample F, but this range was reduced for the sample H. This may be due to the stress relaxation caused by a temperature rise due to heat accumulation in localised region during higher coverage peening. When peening at a higher kinetic energy level of 2.34J, the range of compressive stress region was largely extended in particular for the sample L peened at a coverage of 1500%, however, sample J peened at a coverage of 700% seemed to have a peak value of the compressive stress. The variations in stress values across different samples might be partly caused by the stress measurement process. However, in general, the overall results suggests that the higher kinetic energy peening can create a more rapid accumulation of effective plastic deformation to overcome the stress relaxation due to the localised heat rise. Similar finding was reported by Hassani-Gangaraj [5]. Shot velocity, in another word, kinetic energy appears to be more influential in grain refinement and surface nanocrystallisation.

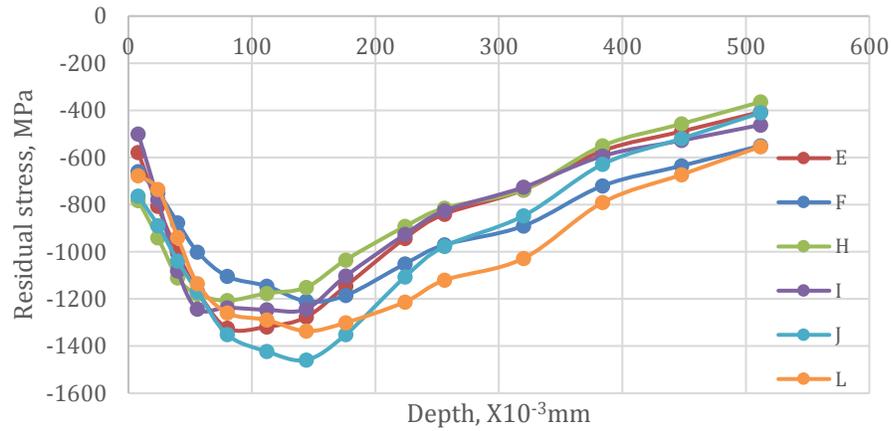


Fig. 5 Residual stress distributions as a function of the depth from the surface.

As shown in the Fig.6, the nanograin structure was further identified with a TEM-FEI. The overall thickness of nanograin structured layer is around 2.5  $\mu\text{m}$  for the specimen peened at a coverage of 1500% at a kinetic energy of 2.34J. The average grain is smaller than 50nm at an equiaxed morphology in the region of the top 1.5 $\mu\text{m}$  thick layer. However, the morphology of the grains was transferred to more deformed and longitudinal but still at a total length under 100nm at the depth of 2.5  $\mu\text{m}$  away from the top peened surface. More detailed TEM study will be carried out for other samples and published elsewhere.

### Conclusions

The present initial investigation has demonstrated that nanograin structures were produced with a commercial shot peening process on the surface of the highly stressed component made of Inconel 718 superalloy. The thickness of the nanocrystallization layer would be up to a few micrometers with the current shot peening process parameters in the present study. The effect of shot velocity on nanocrystallisation was more influential than the coverage.

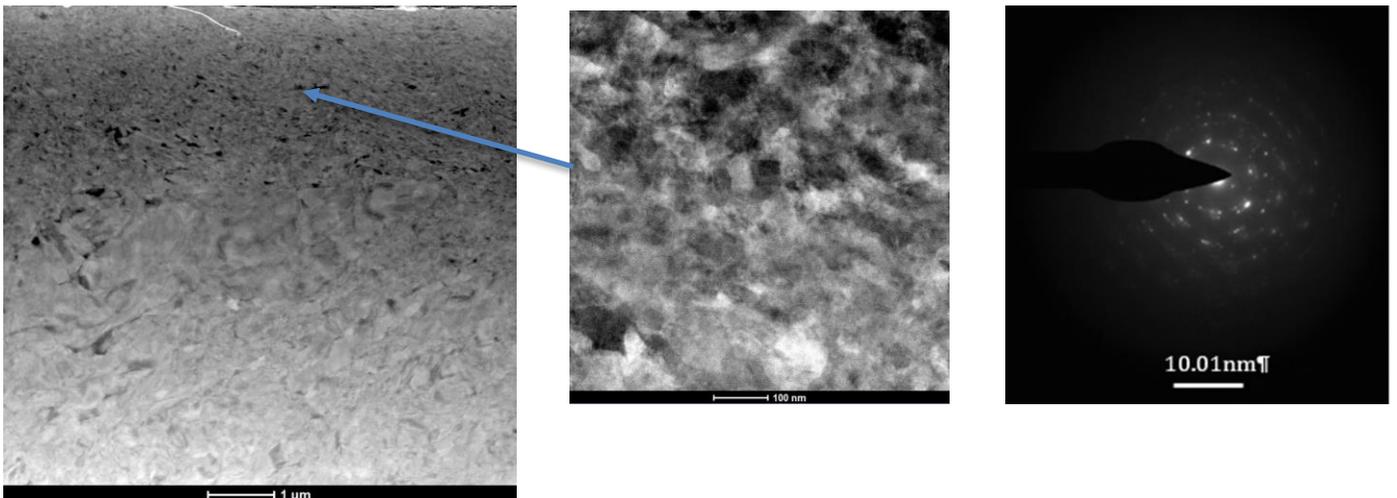


Fig.6 Typical TEM images at the treated surface and sub-surface region of sample L showing BF images and the corresponding SAED patterns.

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