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Short communication

Mechanical property heterogeneity in additively manufactured nickel superalloy

A.N. Forsey\textsuperscript{a,⁎}, Y.B. Das\textsuperscript{b}, T.H. Simm\textsuperscript{b}, D. Clark\textsuperscript{c}, J. Boswell\textsuperscript{c}, S. Gungor\textsuperscript{c}, R.J. Moat\textsuperscript{a}

\textsuperscript{a} Open University, MK7 6AA UK
\textsuperscript{b} Swansea University, SA1 8EN UK
\textsuperscript{c} Rolls Royce Plc, DE24 8BJ UK

Metallurgical additive manufacturing (AM) techniques do not produce a homogenous structure. Digital image correlation is used to quantify significant variations in mechanical properties in an AM nickel superalloy. Non-uniform properties at multiple length-scales are demonstrated, which could explain the poor mechanical properties common with AM alloys.

Extensive investigation over the last 10 years has shown that laser-based AM of nickel superalloys exhibit non-optimal microstructures and hence mechanical properties. High crystallographic texture\textsuperscript{[1–3]}, columnar grain structure\textsuperscript{[4–6]} and intergranular defects\textsuperscript{[7]} are common due to epitaxial growth. The alloy’s use is also limited by high residual stresses\textsuperscript{[8,9]} suboptimal aging\textsuperscript{[10]} and anisotropic mechanical properties\textsuperscript{[11–15]}. Efforts are being made to mitigate these problems with varied levels of success. Notably, the alloy CM247 has shown promise in controlling the microstructures formed during AM\textsuperscript{[5,13]}. Hence this alloy was used in the following work, however other Nickel base alloys have shown similar scope\textsuperscript{[11,16]}.]

Two tensile specimens were EDM machined from a sheet of nickel superalloy CM247\textsuperscript{[4]} manufactured using selective laser melting. Test piece dimensions were based on ASTM E8M (Fig. 1a and b) and orientated with the loading axis perpendicular to the z-axis (the direction of build). Two different samples were measured to provide information about strain at different length scales, and are referred to meso-scale and micro-scale throughout. The tensile specimens were ground for equal times on both front and back faces, to remove surface roughness, and polished to a mirror finish. Final polishing was performed using an oxide suspension (OPS) to eliminate plastic damage from the surface. Optical microscopy and electron backscatter diffraction (EBSD) were used to characterise the microstructure of the specimen surface (Fig. 2a and b). The microstructure observed with optical microscopy, although not equiaxed shows a significantly less directional microstructure compared to that seen with EBSD. Furthermore, EBSD reveals bundles of grains with very similar grain orientation. The colour scale in Fig. 2b shows misorientation from a (001) orientation, and it is clear that this is the most prevalent growth orientation, as would be expected from epitaxial growth in face-centred cubic nickel.

The meso-scale sample (Fig. 1a) was pulled at a strain rate of 0.01 s\textsuperscript{−1} at room temperature and 850 images were taken at 1 Hz using a Nikon DS-Qi2 monochrome camera. The optics used were a Nikkor 200 mm f4 IF ED micro lens spaced by macro bellows, producing an image with a pixel size of 2.6 μm. A 30 s exposure to Kalling’s reagent produced a surface speckle pattern suitable for digital image correlation (DIC) on the polished surface. DIC is a computational technique that tracks the movement of small regions of a surface. The displacements of small subregions of the images are obtained by pattern tracking between displacement steps\textsuperscript{[18]}. This produces a regular grid of displacement vectors from the sample surface for each load step and consequently maps of 2D surface strains. Images were taken during deformation and displacements were obtained using the commercial DIC package DaVis 8.3\textsuperscript{[17]}, using a least squares based algorithm\textsuperscript{[18]}. A subregion size of 31 × 31 pixels with a 10 pixel step size. The 2D plots (Fig. 3b and c) were obtained using a sliding bilinear least square fit to differentiate the displacement field, using a differentiation length of 159 μm.

For mechanical property calculation, a separate high density DIC analysis was performed. This analysis had a subregion size of 21 × 21 pixels and a stepsize of 1 pixel, and was used to maximise the amount of data contained within each averaging stripe. The elastic region is calculated for each stripe by minimising the difference between the tangent and secant moduli, with tangent modulus used as the reported value. The intersection of this modulus, offset by 0.002, with a cubic spline fit to the raw data, is used to calculate the proof stress. The error bars in Fig. 3d and e are calculated by splitting the DIC data into four separate datasets and performing the same calculation process on these distinct vector maps.

When loaded perpendicular to the z-axis (see Fig. 1a and b), grains close to a (001) orientation (white in Fig. 2b) have a theoretical stiffness of ~170 GPa\textsuperscript{[19]} while those with an orientation close to (011) could have a stiffness as high as 200 GPa (Data reported by Dye et al.\textsuperscript{[19]}, used for comparison purposes here is for the Nickel superalloy, Waspaloy, however orientation dependence of different superalloys is not expected to vary significantly). In a similar manner, plastic flow is also orientation dependent, with different slip systems and hence strain expected in different orientations. Hence, clusters of grains with similar
orientations are not expected to behave the same as a typical polycrystal. This potentially large anisotropy has significant implications for structural integrity and component life prediction, as stress localisation inevitably leads to plastic deformation mismatch.

To investigate the influence of this microstructure, in situ tensile tests were conducted in combination with DIC. The image magnifications were optimised for the length scales of microstructural variation observed in these samples. The preparation required a gold remoulded surface to be produced on the sample to function as a speckle pattern for DIC [20,21]. Images were taken using a Zeiss Supra 55VP field emission gun scanning electron microscope (FEG SEM) using backscatter electron mode, an accelerating voltage of 20 kV and a working distance of 16 mm, with a pixel size of 91 nm. The strain was applied by a 4.5 kN ADMET mini-tensile testing machine in situ, at a strain rate of 0.01 s\(^{-1}\). The loading was halted during imaging so that three images could be acquired and averaged at each load step. The images were analysed using a DIC subregion of 11 \(\times\) 11 pixels and a step size of 5 pixels and the strain is plotted using a differentiation length of 3.3 \(\mu\)m.

For a global strain of 0.075, Fig. 3b and c show spatially resolved maps of the correlations made from meso-scale images; the reference image is shown in Fig. 3a. In Fig. 3b, a banded strain response is observed with a range of approximately 0.1 strain, and a periodicity of \(~400\ \mu\)m. Similarly, Fig. 3c has a banded pattern perpendicular to the loading direction with a similar periodicity. This figure shows the range in principal strain angle with respect to the loading direction. The range in angle is 0.3 rad, which is an unexpected range for a uniaxial tensile test.

From Fig. 2b, the vertical regions of epitaxial growth can be seen, which should lead to relatively consistent material properties in the y direction. This was the key assumption to enable the calculation of material properties from the DIC maps, by averaging the strain across the width of the test specimen (y direction). The strain was calculated by fitting a bilinear polynomial to stripes of the image perpendicular to the loading direction, using the optical flow approach [22]. By taking stripes across the full width of the sample (y direction), a constant stress assumption can be made for each region. This enables stress versus strain plots to be made for each stripe and these are then interrogated to obtain the elastic modulus and proof stress. For this study, the constant stress assumption is only approximate and a number of the stripes will cross material property boundaries and in others local constraint effects will alter the stress state. In both cases this is likely to result in an underestimate of the variation in material properties along the sample.

Fig. 3d and e show line plots calculated from rectangular, 65 \(\mu\)m in x and 4000 \(\mu\)m in y, striped regions calculated from a separate high density DIC analysis. Fig. 3d shows the elastic modulus, and Fig. 3e the 0.2% proof stress extracted from the stress-strain curves produced from the rectangular stripe strain calculation. Both are observed to have a similar periodicity to that found in the 2 dimensional plots. A range of \(~75\ \text{GPa}\) in elastic modulus and \(~60\ \text{MPa}\) in proof stress offer extreme uncertainties compared to the values calculated from global strain measurements. These values are likely to be underestimates because the procedure averages out extremes of behaviour.

To link the mesoscopic deformation to microstructural features, a similar second test was performed using SEM imaging. This enabled electron backscatter diffraction (EBSD) to be performed on the same area prior to straining; this region is marked in Fig. 2b. Fig. 4a shows an EBSD image of the micro-scale sample with a gold remoulded surface and Fig. 4b shows the EBSD IPF map of the area imaged prior to straining. Fig. 4c and d show EBSD and the principal strain angle relative to the loading direction respectively. Fig. 4 is plotted at the same global strain value as Fig. 3 to enable direct comparison between the length scales. In the meso-scale test (Fig. 3), a similar range in strain and principal strain offset angle are observed. However, the sudden changes in both strain and principal strain angles seen at grain boundaries can only be resolved at the higher magnification. Conversely, at this higher magnification it is impossible to compare the scale and periodicity of these features. However, the spread in measured values at both length scales is precisely comparable. It is clear to see that the variation in strain accommodation seen in Fig. 3b is radically smoothed by the resolution of the camera. At the scale seen in Fig. 4c, the 400 \(\mu\)m frequency periodicity of strain localisation is revealed, as discontinuous regions of significant deformation. The interfaces of these regions are between grains close to a \{011\} orientation and those closer to \{001\} (Fig. 4b), confirming the cause of the variation to be crystallographic.

Variations in elastic modulus and proof stress of the order observed here pose significant issues for component performance and life predictions. Invariably a single value for proof stress and elastic modulus are used for such calculations. In special cases one might consider using anisotropic mechanical properties, such as single crystal turbine blades and highly texture weld metal, but spatially resolved variations in anisotropy would be unprecedented. This is more the reserve of the microstructural modelling community and not the structural integrity or lifting community.

The extent of the considerations that must be made are illustrated in Fig. 5, with part a showing the global tensile stress-strain curve for this sample. A proof stress of 818 MPa and an elastic modulus of 169 GPa is comparable to properties reported by Geiger [16] and more than adequate for as deposited SLM materials. Fig. 5b shows two tensile curves taken from neighbouring locations indicated in Fig. 3d and e (19 \(\mu\)m apart). One is located in a low strain (red) and one from a high strain...
(blue) region and plotted alongside the global stress strain curve. The difference in elastic modulus is plainly obvious, but similarly, the proof stress between the 3 curves varies by 50 MPa. This effect is further exacerbated by the proposition from Fig. 4, that these steps in material properties are discontinuous; this implies extreme states of local

Fig. 3. Meso-scale DIC results, a) an example image used for analysis with insert showing pixel definition, b) spatially resolved map of loading direction strain (Exx) difference to global value at 0.075 strain, c) spatially resolved map of principal strain angle with respect to the loading direction at the same strain value, d) variation of elastic modulus along the length of sample gauge and e) variation of 0.2% proof stress along the length of sample gauge. Red and blue vertical lines in d and e correspond to locations of data extraction for Fig. 5. Red bars on parts d and e indicate the measured systematic error of the property extraction process. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Fig. 4. Micro-scale DIC results, a) an example image used for analysis with insert showing pixel definition, b) inverse pole figure EBSD orientation map of area tested using DIC c) spatially resolved map of difference in loading direction strain (Exx) to sample average (of 0.075 strain) and d) spatially resolved map of principal strain angle with respect to the loading direction.
constraint for the weaker sections of the material. Such localisations in deformation will result in regions prone to cracking and premature failure, when compared to macroscopic stress-strain data. Furthermore, as a result of this crystallographic heterogeneity, usual post processing techniques, such as heat treatment or HIPing (hot isostatic pressing), will have little influence [13,23].

Digital image correlation has been used to measure the variations in mechanical properties in SLM nickel superalloys. Whether considering the tensile curves plotted in Fig. 5 or the strain maps shown in Figs. 3 and 4, the heterogeneity in mechanical properties are clear. Grain-to-grain variations in mechanical properties, such as those presented in Fig. 4 are traditionally only of interest of material scientists because they become aggregated over large volumes. The results presented in this manuscript do not appear to show the same aggregation, with variations in mechanical properties appearing to be related to the crystallographic heterogeneity inherent to AM materials. The current inability to crystallographically randomise additively manufactured metallic structures is a current obstacle in the AM revolution; not just inability to crystallographically randomise additively manufactured nickel based superalloys, but also for relatively mundane applications. The techniques and results presented in this manuscript do not appear to show the same aggregation, with variations in mechanical properties due to compositional inhomogeneities, in: T.M.S. Annu. Meet, 2007.

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