High resolution imaging and analysis of residual elastic strain in an additively manufactured turbine blade

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Abstract: Using recently developed microchannel plate (MCP) neutron area detectors at a pulsed neutron source provides a method of fast non-destructive imaging and characterisation of advanced materials at unprecedented spatial resolution. The energy resolved neutron transmission spectrum can be found from the neutron time of flight (ToF), this provides valuable information about the crystallographic structure within the sample such as the average residual elastic strain in the incident beam direction. By measuring this spectrum at a number of sample orientations information is built up about the strain distribution throughout the specimen. The energy-resolved neutron transmission spectrum of an additively manufactured turbine blade was measured at the Engin-X instrument, ISIS, UK. A high resolution three-dimensional reconstruction of the neutron attenuation coefficient of the blade was recovered, then analysis of the energy resolved spectrum gave insight into the underlying residual elastic strain profile imparted by the advanced manufacturing process.

Keywords: tomographic imaging; residual elastic strain; neutron transmission; Bragg-edge measurement; microchannel plates; MCPs; additive manufacturing; advanced materials.


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Anton S. Tremsin received MSc from Moscow Institute of Physics and Technology in 1989, and PhD from Russian Academy of Sciences in 1992. He was an Honorary Visiting Postdoctoral Fellow of British Royal Society in 1994–1996 working at the University of Leicester, UK and is working at the Space Sciences Laboratory (SSL) at the University of California at Berkeley (UCB) since 1996. She has led the research programs at SSL which resulted in a substantial extension of detection technologies initially developed for space-based astrophysical applications into the fields of synchrotron research, biomedical imaging and non-destructive testing with epithermal, thermal and cold neutrons. In addition to his research activities, he was a Lecturer at the Physics Department. He has been presented the results of the research at many international conferences and published more than 200 technical papers is co-author on nine patents.
1 Introduction

Additive manufacturing (AM) technology has been in research and development for over 20 years. AM was initially used by engineers for rapid prototyping of parts and tools [1] from materials including metals, ceramics, polymers composites and biological systems [2] directly from digital models. It has now found itself in wide range of applications from micro-scale to large structural parts manufacturing in micro-electronics, tooling, automotive/industrial [3], aerospace [4] and biomedical fields [5,6]. The ability to efficiently fabricate parts often with geometric and material complexities that cannot be made with conventional e.g., subtractive manufacturing techniques, is driving its development. Recently, AM has begun to emerge as an important commercial manufacturing technology [2], though many challenges remain to be addressed. The current work is concerned with imaging and characterising the residual elastic strains
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imparted on such metal components during the thermally driven sintering process used during manufacturing. The results of a recent neutron tomography experiment are presented here. The wavelength-resolved transmission spectrum of thermal neutrons of a jet engine turbine blade manufactured with direct laser deposition (DLD) was measured. High resolution neutron attenuation coefficient maps were reconstructed and the residual elastic strain profile was examined via the wavelength dependence of the transmitted spectrum.

As the working environments of such additively manufactured components are pushed to the materials physical limits, better understanding of the thermal cycling processes and how they are linked to microstructure as well as the ability to accurately model them is crucial to their success as engineering components. The temperature driven manufacturing processes can generate significant residual strains in such components. Knowledge of these strain fields is crucial to the development of the free-form additive building process and the in-service success. Non-destructively mapping this strain state however, presents significant challenges.

Non-destructive strain measurement by X-ray or neutron diffraction relies on Bragg’s law, \( \lambda = 2d \sin(\theta) \), which relates the interplanar lattice spacing, \( d \), and the wavelength of the incident radiation, \( \lambda \), to the scattering angle, \( \theta \). The average lattice spacing is measured in a specific direction within a scattering gauge volume defined by collimating or focusing the incident and scattered beams. Typical spatial resolutions for diffraction based techniques are of the order \( \approx 1 \) mm for neutron or \( \approx 1 \) \( \mu \)m for X-ray synchrotron experiments [7–9]. The elastic strain, \( \varepsilon \), is defined as the relative shift in lattice spacing from the ‘strain-free’ value. Since diffraction is primarily sensitive to lattice spacing, the elastic strain component in one direction can be found by measuring the relative change in the diffracted peak position. By raster scanning the scattering gauge volume through the sample, the elastic strain fields can be mapped in two or three dimensions (depending on the collimators used). Diffraction methods generally measure only one strain component in a single voxel at a time, hence to fully characterise the strain state of a sample many measurements at multiple sample orientations are required. Therefore, obtaining a full strain map is often a laborious process.

Recent developments in high resolution Microchannel plate (MCP) neutron detection [10–12] are enabling neutron studies of material characteristics at unprecedented spatial and time resolution. Neutron sensitive MCPs coupled with a TimePix readout provide high neutron detection efficiency (\( \approx 70\% \) for cold neutrons [13]) at a temporal resolution of 1 \( \mu \)s and a spatial resolution of 55 \( \mu \)m. Though with event centroiding and longer measurement times a spatial resolution of up to 10 \( \mu \)m should be possible [14,15]. This efficiency, resolution and speed is superior relative to other current neutron detection methods, such as imaging plates and scintillator-based systems [16].

The high temporal resolution offered by MCP detectors allows wavelength resolved imaging at pulsed neutron sources. By tagging each arriving neutron with its time of flight (ToF), the wavelength can then be determined from the known time structure of the incident pulse and neutron flight path. The primary interaction of thermal neutrons with most metals is Bragg scattering, which is dependent on the materials crystal structure and incident neutron wavelength [17]. This type of imaging can therefore provide quantitative information about a materials microstructure and crystallographic texture from the measured wavelength resolved spectrum.
Bragg-edge neutron transmission [18–22] has recently emerged as a means for non-destructively evaluating the residual elastic strain state throughout engineering components and bulk materials. It utilises the fact that the transmission spectrum of thermal neutrons in polycrystalline materials displays sudden well-defined jumps in intensity as a function of neutron wavelength. These jumps in intensity are known as Bragg-edges and occur for a given wavelength as described by Bragg’s law. If we consider a lattice spacing $d_{hkl}$ for a particular $hkl$ set of lattice planes, the Bragg angle increases as a function of incident neutron wavelength until $\theta = \pi$. At wavelengths greater than this critical value, no diffraction takes place and there is a sudden increase in the transmitted intensity. The wavelength at which this occurs gives a direct measure of the average lattice spacing through the sample in the direction parallel to the incident beam. By recording the wavelength resolved neutron transmission spectrum on a pixelated area detector a two-dimensional map of the average lattice spacing in the incident beam direction can be obtained.

2 Experiment

The wavelength-resolved neutron transmission spectrum of an additively manufactured turbine blade was measured at the Engin-X beamline, ISIS, Rutherford-Appleton Laboratory, UK. The sample was mounted on a fine rotation stage directly in front of the high resolution MCP detector with a pixel size of $55 \times 55 \, \mu m^2$ (which defines the maximum spatial resolution) 50 m downstream from the pulsed neutron source. This flight path allows the neutron pulse to expand sufficiently in time to obtain a wavelength resolution of $\sim 1 \, mÅ$ with the MCP detector as well as a highly collimated incident neutron beam. For each incident neutron pulse 2734 intensity images were recorded to form a three-dimensional image stack, as shown in Figure 2. Each image recorded the transmitted neutrons every 12 µs and were accumulated over all pulses for the duration of each projection.

The nickel-base super alloy C263 turbine blade was produced via an additive manufacturing technique known as direct metal laser deposition (DMLD). The sample was built up in the vertical ($y$) direction on top of a steel supporting plate using a powder bed procedure in an EOS M270 laser-sintering system (EOS GmbH, Munich, Germany). Fabrication consisted of repeatedly spreading a thin layer ($\approx 20 \, \mu m$ thickness) of the C263 powder (average particle size $\approx 20 \, \mu m$) and line sintering using a focused 195 W Yb-fibre laser beam with a 100 µm spot size to incrementally build the sample. The sample was manufactured under atmospheric conditions with a raster speed of 900 mm/s with a line spacing of 80 µm. The laser sintering and build directions are described in Figure 1.

Two tomography scans were recorded, the first scan recorded 90 projections in $2^\circ$ rotation steps with an exposure time of 40 s each to allow a high resolution tomographic reconstruction of the turbine blade neutron attenuation coefficient. The second scan consisted of fewer projections, (45), with a longer exposure time for each projection of 2 hours. The extended exposure time was required to build up sufficient neutron counting statistics to accurately determine the positions of the Bragg-edges at each sample orientation. The incident neutron wavelength range was defined by the neutron choppers directly downstream of the moderator. The detector and choppers were set up to provide a neutron spectra with wavelength 1.3–4.4 Å.
Figure 1  Coordinate system used for the construction and measurement of the turbine blade. The blade was incrementally built up in the $y$-direction by line sintering each powder layer in the $x$-direction. For the tomography the blade was rotated by an angle, $\theta$, about the $y$-axis.

Figure 2  Transmission spectrum measured with the MCP detector for each neutron pulse is a three dimensional image stack. In this experiment an image was recorded every 12 $\mu$s throughout the incident neutron pulse.
3 Data analysis

The tomographic neutron transmission experiment generated a four-dimensional dataset; \( x \) and \( y \), the horizontal and vertical positions on the detector respectively, \( \lambda \), the neutron wavelength (calculated from the neutron ToF) and \( \theta \), the rotation of the sample relative to the incident beam. The rotation axis is parallel to the \( y \)-axis, where \( z \) is in the direction of the incident beam, Figures 1 and 3(a) show these directions relative to the sample. The neutron ToF is converted to neutron wavelength with:

\[
\lambda = \frac{ht}{m_n l},
\]

where \( h \) is Planck’s constant, \( m_n \) is the neutron rest mass and \( t \) is the time it takes each neutron to travel the length of the flight path, \( l \). Open beam measurements were made to correct for detector non-uniformities and the temporal and spatial intensity variations in the incident neutron beam, i.e., spectra with and without the sample in place were recorded. The spectrum was normalised by dividing each radiograph, \( I(\lambda) \) by the corresponding white field image, \( I_0(\lambda) \) to determine the transmission spectrum \( T(\lambda) = \frac{I(\lambda)}{I_0(\lambda)} \). Each incident neutron pulse is divided up into \(~3000\) bins in time, each containing \( 512 \times 512 \) pixels. A running total of the neutron counts in each detector pixel for each time bin is recorded for each sample orientation. The following section summarises the data processing of this four-dimensional dataset to obtain high resolution tomographic reconstructions of the neutron attenuation coefficient and to obtain information about the residual elastic strain state for each sample orientation.

3.1 Neutron attenuation coefficient reconstructions

The wavelength dependent measurement means that the three-dimensional neutron attenuation coefficient can be recovered as a function of neutron wavelength, thus providing a useful contrast imaging mechanism. However this imaging mode comes at the cost of a greatly increased imaging time. To obtain a high resolution absorption tomogram the wavelength resolved dataset was integrated over the ToF axis resulting in a two-dimensional map of the attenuation of neutrons through the sample with no energy dependence. The three-dimensional reconstruction of an object from a series of its projections is a well-established technique \([23,24]\). The filtered back projection algorithm was used here to obtain a three-dimensional map of the neutron attenuation coefficient.

3.2 Bragg-edge analysis

Four Bragg-edges were measured for each sample orientation over the incident neutron energy range. Fitting the Bragg-edges with an analytical function provides a way to accurately determine the edge position and shape to an accuracy which is better than the resolution of the ToF bins. Here we use a five parameter function to describe the shape of the Bragg-edge \([18]\):
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\[ T(\lambda) = c_0 + \frac{c_1}{2} \left[ \text{erfc} \left( \frac{\lambda - \lambda_{\text{bulk}}}{\frac{1}{2^2} \sigma} \right) - \exp \left( \frac{\lambda - \lambda_{\text{bulk}}}{\tau} \cdot \frac{\sigma}{2^2 \tau} \right) \text{erfc} \left( \frac{-\lambda + \lambda_{\text{bulk}} + \sigma}{\frac{1}{2^2} \sigma} \right) \right] \]

where \( T(\lambda) \) is the normalised transmission intensity and \( \lambda \) is the wavelength of incident neutrons. The wavelength at which the \( hkl \) Bragg-edge occurs is \( \lambda_{\text{bulk}} \) and \( \text{erfc} \) is the complimentary error function. The transmitted intensity before the edge is described by \( c_0 \) and the relative height of the edge is given by \( c_1 \). The symmetric and asymmetric broadening of the edge are described through parameters \( \sigma \) and \( \tau \), respectively. The main source of broadening is owing to the initial width of the neutron pulse leaving the moderator (neglecting any sample broadening effects) [19].

4 Results

4.1 Neutron tomography

A neutron radiograph of the turbine blade is shown in Figure 3(a). The internal cooling channels can be seen, the largest channel has a diameter of around 1mm as shown in the lineout plot in Figure 3(b), the lineout corresponds to the line in Figure 3(a) where \( Y = 10 \text{ mm} \). At the time of the experiment the channels were full of resin, which has a high neutron attenuation coefficient, hence the channels appear darker (highly absorbing) in the radiograph and lineout plot.

Figure 3  (a) Neutron radiograph of the additively manufactured turbine blade positioned perpendicular to the incident beam (\( \theta = 0^\circ \) position). (b) Line-out of the relative transmitted neutron intensity at position \( Y = 10 \text{ mm} \) from (a)

The reconstructed neutron attenuation coefficient from the high resolution tomography scan is displayed in Figure 4. The longest dimension of the blade is 21.4 mm. The reconstruction revealed that there were no significant pore structures in the blade from the additive manufacturing process at 0.3 mm resolution. The image shown in Figure 4(c) has been thresholded to show the inner cooling channels within the blade, the largest channel has a diameter of \( \approx 1 \text{ mm} \).
Figure 4 (a) and (b) High resolution reconstructed images of the turbine blade the from neutron tomography experiment at Engin-X. (c) Thresholded volume showing the cooling channels within the blade. (d) Top section of the blade has been translated, revealing the reconstructed cooling channels. The largest channel has a diameter of approximately 1 mm

4.2 Strain results

The results of the fitting analysis were compared. The 200 Bragg-edge gave the best results since it coincided with the brightest part of the incident neutron pulse (the neutron flux was approximately three times higher than for the 111 edge). The spectrum measured in each pixel was fit using the analytic function in equation (2). A two-dimensional average strain map for the $\theta = 0^\circ$ projection obtained from the fitting analysis is shown in Figure 5(b). The strains in this case show significant noise, i.e., owing to the lack of neutron statistics because of the relatively small sample thickness in the beam direction. However, by integrating over regions of pixels neutron statistics can be improved at the cost of spatial resolution. To enable an analysis of the residual elastic strain, the neutron counts in each ToF frame were summed over the whole area of the blade ($512 \times 512$ pixels) for each angular position. This resulted in a single Bragg-edge spectrum for each projection giving the average strain component in the whole sample in that direction. Each edge was then fit with the analytic function described in equation (2), an example of the data and corresponding fit are shown in Figure 6(a). The wavelength at which the Bragg-edge occurred was obtained as a function of sample rotation. The average lattice spacing, $d$, was then determined through Bragg’s law, which in the transmission geometry reduces to $d = \lambda/2$. The average strain component, $\varepsilon$, in the transmission direction was then determined as a function of sample rotation using:

$$\varepsilon(\theta) = \frac{d(\theta) - d_0}{d_0},$$

(3)
where $d(\theta)$ is the average lattice spacing in the transmission direction as a function of sample rotation and $d_0$ is the ‘strain-free’ lattice parameter. The mean value of $d(\theta)$, $d_0 = 1.79$ Å, was used instead of the theoretical lattice spacing value, $d_0^{\text{theory}} = 1.76$ Å, in order to account for any systematic errors that may be applied to the lattice spacing measurement using neutron TOF (for example uncertainties in the path length will induce a systematic offset without altering the relative lattice measurements). Using an experimental (as opposed to theoretical) $d_0$ accounts for any linear offset in the strain profile associated with the experiment. The strain calculated with the experimental lattice spacing value gives better agreement when compared with strain measurements made in a separate X-ray diffraction study [25]. The X-ray study showed the magnitude of the elastic strain was $<500$ µε, which corresponds to a maximum change in lattice spacing of $|\Delta d| < 8 \times 10^{-4}$ Å. The average strain as a function of rotation is shown in Figure 6(b). The height of the Bragg-edges corresponds to the number of crystallites being orientated such that the backscattering condition is fulfilled [26]. Therefore analysis of the edge heights provides information about texture and preferred orientation of crystallites within the specimen. The height of the Bragg-edges as a function of rotation are also shown in Figure 6(b). Though this is the result of integrating over the whole specimen, there appears to be a clear relationship between the residual elastic strain, the crystallographic texture and the direction of the laser deposition. The heights of the Bragg edges measured parallel to the direction of laser scanning (at angles $\theta = 0^\circ$ and $180^\circ$) are larger than in any other direction indicating an increased number of crystallites oriented along this axis. This suggests that there is a preferred crystallite orientation (texturing) parallel to the direction of the lasing (the $x$-direction in Figure 1); however this has only been measured in the $x$-$z$ plane. Further measurements are required to determine the preferred orientation in three-dimensions. This preferred orientation appears to influence the angle dependent strain profile, this relationship is not well-understood and requires further investigation. However, from this we may conclude that microstructure effects induced by the fabrication of the AM blade influence the crystallographic and elastic strain properties of the component.

**Figure 5** (a) Neutron radiograph and (b) two-dimensional average strain map. The result of the Bragg-edge fitting to the projection at $0^\circ$ (see online version for colours).
5 Conclusions

Neutron transmission measurements made at a pulsed neutron source using an MCP detector have been applied to the characterisation of small engineering components. A high resolution volumetric map of the neutron attenuation coefficient was recovered verifying the internal structure of the additively manufactured component. We also presented results from a longer energy-resolved tomography scan which provides a measure of the average residual elastic strain in the transmission direction through the sample. Owing to the low interaction of the sample with the neutron beam in the thinnest parts of the sample, neutron counts were averaged across the whole blade to enable accurate Bragg-edge fitting. This analysis provided evidence that the component contains a residual elastic strain profile which (due to the orientation dependence) is likely related to its microstructure formed during the additive manufacturing process. This analysis shows that the microscopic effects present in this advanced manufacturing process may have some influence on the macroscopic properties of such components. Further work will be carried out to understand the nature and origins of these effects. Longer measurement times would provide better neutron statistics and accuracy in the Bragg-edge fitting, but the combination of this analysis with small area, high spatial resolution characterisation techniques such as X-ray diffraction and hole drilling offer the greatest promise of a complete interpretation.

References

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