ANALYSIS OF STRAIN-INDUCED PEAK BROADENING IN TITANIUM ALLOYS

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Abstract

X-ray diffraction measurement in the bulk of engineering components allows evaluation of lattice spacing variation by application of Bragg law and hence, by comparison with a strain-free reference specimen, computation of elastic strains and stresses. Diffraction peak position is related to average elastic strain within the scattering grains, whilst diffraction peak shape is related to crystallite size and so-called root mean square (RMS) - strain, which refers to the spread of lattice parameter values around the average. This spread arises as a result of the anisotropic elastic and plastic properties of the individual crystallites, which, under macroscopic plastic deformation, lead to a non-uniform distribution of inter- and intra-granular elastic and plastic strains. It has been shown in the literature, that, based on monochromatic diffraction experiments, a correlation of peak width and equivalent plastic strain can be established for a given material. In this paper a correlation of plastic strain versus peakwidth is established based on energy dispersive Synchrotron X-ray diffraction measurements of a Ti-6Al-4V 4 point bend specimen. The correlation is then used to map equivalent plastic strain within a plate specimen of the same material in which two semicircular notches act as stress concentrators during tensile loading, inducing a known, non-uniform equivalent plastic strain field. The resulting map is compared with measurements of equivalent plastic strain by digital image correlation. The extent of the plastic zone observed shows qualitative agreement, however the equivalent plastic strain values predicted by peakwidth analysis are lower than those deduced from digital image correlation.

Key words: Peak-width, Plastic Strain Mapping, X-ray Diffraction, Digital Image Correlation

1 Introduction

In material modeling it is often desirable to separate total strain into plastic and elastic contributions. However, for all but the simplest geometries experimental measurement of these quantities is a considerable challenge.

One means of measuring elastic strains in small grained bulk metallic specimens is by Bragg \cite{1} powder diffraction. Measurements can either be carried out with a monochromatic beam, collecting a radial pattern of peaks corresponding to a section through the powder Debye-Sherrer rings \cite{2} or with a polychromatic (white) beam, collecting a pattern of diffraction peaks in energy at a fixed diffraction angle. In both cases diffraction plane spacing can be computed from diffraction peak position and elastic strain deduced by reference to a previously established "$d_0$" strain free
This document was presented at the Denver X-ray Conference (DXC) on Applications of X-ray Analysis.

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reference spacing. The balance of measurement accuracy required and time available determines the method of choice - monochromatic setup offers better resolution, whilst polychromatic beam allows more rapid measurements with a trade off in accuracy. The time advantage and the need to map elastic strains over large areas within engineering components make the white beam setup attractive for engineering applications. It was hence chosen as the method of interest in this paper.

Diffraction peak shape is related to crystallite size and so-called root mean square (RMS) strain, which refers to the spread of lattice parameter values around the average. This spread arises due to the anisotropic elastic and plastic properties of the individual crystallites which, when macroscopic deformation is applied, lead to inter and intra granular variation of elastic and plastic strain. In this context dislocations act as nuclei of inelastic deformation and the elastic strain fields associated with them give rise to the spread of lattice parameter around the mean. Based on the diffraction peak line profile analysis of monochromatic measurements, dislocation density can be estimated (Ungar et al. [3], Castelnau et al. [4], Dragomir et al. [5] or Leoni et al. [6]).

The primary mode of deformation in metals at room temperature is by dislocation motion. The evolution of dislocation density can be linked to plastic strain as illustrated by Bulatov et al. [7], based on dislocation dynamics modeling in single crystal molybdenum.

In fact it is not even necessary to consider dislocation density in order to link diffraction peak width variation to plastic deformation. Ji et al. [8] empirically correlated monochromatic diffraction peak width with plastic strain in INCONEL 600. Pantleon et al. [9] showed a similar correlation of plastic strain to peak width in an aluminium alloy, considering a single grain in the bulk, whilst Dawson et al. [10] investigated a polycrystalline aluminium alloy. Tang et al. [11] considered diffraction peak full width at half maximum (FWHM) variation as a function of plastic strain in copper foil under in-situ loading. They all show a clear increase in peak-width with plastic deformation using a monochromatic diffraction setup. To the authors knowledge the only mention of correlation of plastic strain and peak width in energy dispersive diffraction setup was presented by Zhang [12]. The aim of this paper is to investigate the feasibility of plastic strain mapping using peak width analysis on energy dispersive diffraction measurements.

2 The Specimen and Material

To assess the feasibility of mapping equivalent plastic strain by analysis of peak width variation in energy dispersive diffraction measurements a specimen with a well known plastic strain distribution was required. This was achieved by designing a plate specimen with two semicircular notches (Fig. 1), such that tensile loading in the axial direction gives rise to a non-uniform field of plastic deformation in the central gauge region (grey in Fig. 1) with deformation concentrated around the two notches. The specimen was manufactured from 2 mm thick rolled titanium Ti-6Al-4V alloy sheet, with the rolling direction aligned with the specimen axis. As the presented methodology is independent of mechanical material properties, they are here omitted for brevity.

3 Peak-Width to Plastic Strain Correlation using a 4 Point Bend Sample

The correlation of equivalent plastic strain to peak width is established using a 4mm high beam cut from the same sheet of titanium alloy as the double notched specimen, with its axial direction
aligned with the rolling direction. The beam was loaded in a 4 point bend configuration about the major axis to a total axial strain of 2.5% in tension on the bottom surface. On unloading, the mid section of the beam remained bent. Figure 2 shows the deformed center section of the beam with a superimposed diagram of elastic and plastic strain distribution.

Diffraction measurements were carried out in transmission at the Synchrotron Radiation Source (SRS) in Daresbury, UK, on powder diffraction beamline 16.3. The incident beam contained a smooth spectrum of photon energies from 0 to 100keV. Line spectra of counts versus photon energy were collected using a Canberra Li-drifted Ge energy dispersive detector fixed a 2θ angle of 10 degrees in the vertical plane. For the measurement of the 4 point bend calibration sample, the incident beam was collimated to a width of 0.1mm in the horizontal and a height of 1mm in the vertical plane. The 4 point bend calibration sample was mounted vertically such that the scattering vector was aligned along its axial direction. Then a line measurement was recorded along the major axis of the Ti beam with 0.1mm spatial resolution. Elastic strains were computed both by single peak fitting and full pattern refinement using the GSAS Rietveld refinement software [13]. Their variation is shown in figure 3. The difference between the values computed by single peak fitting and the GSAS refined data is small. In both cases strain error was lower than $10^{-4}$ which is consistent with the results presented by Zhang [12] for in situ loading of Ti-6Al-4V on the same instrument. The elastic strain error bars in figure 3 are smaller than the plotted markers.

In the region from 1.7 to 2.7 mm in figure 3 no plastic deformation occurred. Here total strain equals elastic strain. From the simplifying assumption that yield in compression and tension occurs symmetrically, one would expect this region to be centered about the 2 mm position. The asymmetry of the elastic region in figure 3 can be explained in terms of the asymmetry of yield in tension and compression displayed by Ti-6Al-4V (Neeraj et al. [14], Golshan et al. [15]). Assuming a linear variation of total strain in the bent beam specimen and using classical strain decomposition, $\epsilon = \epsilon_p + \epsilon_e$, variation of plastic strain across the beams crossection can be deduced as discussed by Golshan et al [15] (Fig. 3).
Figure 2. Sketch of bent beam total, elastic and plastic strain profiles superimposed on an image of the central part of the 4 point bend specimen.

Figure 3. Plot of the measured elastic strain profile in the 4 point bend specimen based on either Single Peak fitting (SP) or GSAS refinement. Also shown is the straight line approximation representing total strain and the plastic strain profiles computed by strain decomposition.

Figure 4 shows a plot of plastic strain versus FWHM deduced from single peak fitting of the 004 reflection. FWHM values were normalized with respect to the averaged FWHM values from depths between 1.8 to 2.6 mm. Normalized FWHM is denoted by FWHM$_n$. Apart from the 004 reflection, 100, 002, 102, 110 and 104 reflections were also considered. However here correlation was not as good as in the case of the 004 reflection and hence the results are here omitted.

As a fitting function a second order polynomial of the form

$$\epsilon_p = A \ast (FWHM_n)^2 + B \ast (FWHM_n) - A - B$$  \hspace{1cm} (1)

was chosen. It passes through point (1,0) which corresponds to the case of no plastic strain. Values of A and B were found by least squares fitting as $1.543 \ast 10^5$ and $-2.556 \ast 10^5$ respectively, where equivalent plastic strain, $\epsilon_p$ is in micro-strain, $10^{-6}$. The resulting fit is shown in figure 4.

The correlation in figure 4 is similar to that found by Ji et al. [8] for INCONEL 600 and monochromatic diffraction setup. This suggests that it should be possible to use the method of correlating peakwidth with equivalent plastic strain in polychromatic diffraction measurements also.

4 Plastic strain mapping

Plastic deformation was introduced to the central area of the double notched Ti plate specimen (Fig. 1) by tensile loading in the axial direction until the macroscopic load displacement curve showed some non-linearity and reduced gradient. Then the specimen was slowly unloaded and removed from the loading rig.
Figure 4. Plot showing the 2nd order polynomial fit to the equivalent plastic strain (Fig. 3) versus FWHM data for the 004 reflection.

4.1 Plastic Strain from Diffraction measurements

Diffraction measurements were carried out in transmission on the 10x10mm gauge region of the double notched specimen (Fig. 1). The measurement region was mapped with a grid of 10 x 10 points, with the incident beam collimated to 1x1 mm. The rest of the diffraction setup remained as described above. Two components of strain, specimen axial and transverse, were collected at each point. To reduce noise, the quarter symmetry of the Ti specimen was exploited and the measured data averaged to cover one quarter of the measurement area (Fig. 1). As such results plots only show a quarter of the measurement region, corresponding to the shaded area (1) in figure 1.

Figure 5. Equivalent plastic strain map from 004 reflection FWHM analysis with scattering vector reflection FWHM analysis with scattering vector aligned in the specimen axial direction.

Figure 6. Equivalent plastic strain map from 004 reflection FWHM analysis with scattering vector reflection FWHM analysis with scattering vector aligned in the specimen transverse direction.
The 004 peak was fitted with a Gaussian and hence peak position and FWHM were extracted. Elastic residual strains were computed using Bragg’s law. FWHM was normalized against the lowest FWHM recorded in the 10x10mm gauge region to give \( FWHM_n \). The assumption here is that the lowest FWHM value in the gauge region corresponds to the case of only elastic deformation. Based on the equivalent plastic strain versus \( FWHM_n \) correlation found previously (Fig. 4) equivalent plastic strain at each measurement point was deduced.

2 maps of equivalent plastic strain were thus generated from FWHM analysis of the 004 peak. Figure 5 shows the predicted map of equivalent plastic strain when the scattering vector is aligned in the specimen axial direction, whilst figure 6 shows the case where the scattering vector is in the transverse direction.

4.2 Digital image correlation

To provide a comparison for the equivalent plastic strain maps computed from FWHM variation, digital image correlation (DIC) was used as a direct method of measuring total strain introduced into the sample during loading and unloading. Throughout the specimen’s deformation, high resolution images were recorded of the 10x10mm gauge region. Fields of deformation increment in the specimen axial and transverse direction were computed between consecutive images by correlation of subregions from the both images. For each deformation increment, the field of total strain increment was found by differentiation. To obtain accumulated total strain in the sample after unloading all total strain increments computed between consecutive images were summed up. The plastic strain field in the unloaded specimen was found by subtracting the residual elastic strain field (from diffraction measurements) from the DIC computed total strain field. Then the equivalent plastic strain field was calculated (Fig. 7).

For easier comparison of predicted equivalent plastic strain values predicted by FWHM analysis and DIC, values along a line from (0,0) to (0,5) were extracted from figures 6 and 5 (computed from FWHM variation) and figure 7 (from DIC). These profiles are shown in figure 8.

5 Discussion

The overall shape of the equivalent plastic strain distributions generate by FWHM analysis (Figs. 5 and 6) agrees with that found by digital image correlation (Fig. 7). As expected from the specimen geometry and loading, plastic deformation in figures 5 to 7 is concentrated in the lower left hand corner, close to the tip of the semicircular notches, bearing in mind that the maps only show the top left hand quadrant (labeled (1) in Fig. 1) of the measurement region. On the left hand side of the top edge of the maps shown in figures 5 to 7 deformation was purely elastic. Zero equivalent plastic strain is shown in this area.

It is interesting to see that the plastic strain maps predicted by FWHM analysis (Fig. 5 to 6) show lower values of equivalent plastic strain than the map found by DIC (Fig. 7). DIC as a direct optical measurement technique can be assumed to only introduce a small error to the measured plastic strain field. By comparison with the DIC generated map, the values of equivalent plastic strain predicted by FWHM analysis are 23% lower in the case of figure 5 and 74% lower in the case of figure 6. This is confirmed by the line profiles in figure 8 which also show however, that the overall profile shape is similar for DIC and FWHM computed equivalent plastic strain.
Figure 7. Map of equivalent plastic strain (micronstrain, $10^{-6}$) computed from digital image correlation total strain and X-ray diffraction elastic strain maps.

Figure 8. Line profile of equivalent plastic strain (micronstrain, $10^{-6}$) in the gauge region along the line (0,0) to (0,5) as predicted by FWHM analysis (extracted from Figs. 6 and 5) and DIC (Fig. 7)

There are a number of possible factors which might explain this discrepancy. The correlation of plastic strain to peakwidth variation was carried out on the basis of a bent beam specimen (Figs. 4). An important assumption is that the material at each point in the section is exposed to a state of uniaxial loading. The strain gradient effect, as a first approximation, is ignored. A more in depth investigation should take this effect into account. The equivalent plastic strain versus FWHM$_n$ data was fitted using a second order polynomial as a fitting function. This was seen as appropriate to demonstrate the methodology. It is however possible that a different choice of fitting function might provide a better estimate of equivalent plastic strain. Another factor is that the horizontal collimation of the incident beam was changed from 0.1mm on the calibration specimen to 1mm in the double notched specimen. However since this change in dimension was perpendicular to the scattering vector, its effect on peak broadening should not be significant.

The plot of equivalent plastic strain computed from FWHM variation in diffraction profiles with the scattering vector aligned in the specimen axial direction (Fig. 5) shows significantly higher values than the plot based on diffraction measurements with the scattering vector aligned in the specimen transverse plane (Fig. 6). This suggest that peak broadening exhibits a certain directionality, with a larger amount of broadening occurring in the principal loading direction than perpendicular to it. A more detailed analysis will be required to investigate this interesting observation.

6 Summary

In this paper the feasibility of equivalent plastic strain mapping based on peak broadening analysis from white beam diffraction measurements was investigated. Maps of equivalent plastic strain in a double notched plate specimen with tension induced non-uniform plastic strain distribution were computed based on a peak width to equivalent plastic strain correlation established from a 4 point bend sample. The maps generated from FWHM variation were compared with a map of equivalent plastic strain found by digital image correlation. The overall shape of the equiva-
lent plastic strain distribution is similar. However predictions of equivalent plastic strain magnitude from FWHM analysis are lower than from DIC and show a dependance on the scattering vector direction.

Overall the methodology presents an interesting means of mapping equivalent plastic strain, especially since the method can easily be extended to allow measurements in the bulk, where digital image correlation is not possible. More in depth investigation is required to reduce the variability of the predicted equivalent plastic strain maps and to render the methodology sufficiently robust for quantitative analysis.

References