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High-resolution strain mapping in bulk samples using full-profile analysis of energy dispersive synchrotron X-ray diffraction data

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Abstract

The feasibility of high-resolution strain mapping in bulk samples with both high-spatial and strain resolution is demonstrated using high-energy X-rays between 100 and 300 keV on beam line ID15A at the ESRF. This was achieved by using a multiple-peak Pawley-type refinement on the recorded spectra. An asymmetric peak profile was necessary in order to obtain a point-to-point strain uncertainty of 10^{-5}. The presented results have been validated with alternative methods, in this case FE model predictions. This technique promises to be a significant development in the in situ characterisation of strain fields around cracks in bulk engineering samples. The implication of slit size and grain size are discussed. This paper is a concise version of the work published in [A. Steuwer, J.R. Santisteban, M. Turski, P.J. Withers, T. Buslaps, J. Appl. Cryst. 37 (2004) 883].

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1. Introduction

We demonstrate the feasibility of undertaking strain mapping in bulk engineering components with high-spatial and strain resolution using energy dispersive synchrotron X-ray diffraction (EDXRD) and full-profile analysis. The aim was to improve the spatial and strain resolution achievable in bulk materials-engineering components in order to investigate fatigue and creep cracks under in situ loading, in particular the development of the residual strain fields around cracks tips, where there are potentially great benefits to be gained.

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We investigated a fatigue crack in a 25 mm thick austenitic stainless steel compact tension (CT) specimen. A spatial resolution of 0.4 mm was achieved, but this is not limited by the beam geometry but by the average size and hence number of diffracting grains in the sampling volume. The photon energy range used in the experiment reached 300 keV, which together with the high flux available at the ESRF provides penetration depths of several cm in most engineering materials. The use of high X-ray energies is normally accompanied by relatively small diffraction angles, in our case $2\theta = 3.5°$, which leads to considerable elongation of the gauge volume. This limits the applicability of this technique somewhat to essentially two-dimensional problems. The implication of slit size and grain size are discussed. This paper is a concise version of the work published in [1].

2. Background

Energy dispersive synchrotron X-ray diffraction is a versatile and powerful tool for materials research providing full X-ray spectra at a single diffraction angle. The combination of high-flux, spectral information and excellent beam definition offers the prospect of high- spatial resolution with short counting times, opening up a whole new range of possible applications ranging from basic materials engineering to dynamic in situ measurements. Energy dispersive strain measurements have tended to be focussed on near-surface regions, intergranular studies that do not require strain mapping or single-peak analysis for strain mapping of bulk components [2–10]. In many cases it has proven difficult to attain the benchmark 10^{-4} accuracy within reasonable data acquisition timescales considered optimal for engineering strain measurements. This has been interpreted as an inherent uncertainty attributed to the comparatively broad widths of the observed peaks which stems from the limited energy resolution of current detectors, as well as limitations in electronic data acquisition hardware [11]. Kuntz et al. argued correctly that the (instrumental) strain resolution depends critically on the position of the diffracting grains in the gauge volume [4].

When considering accurate peak location it should be borne in mind that energy dispersive profiles typically comprise asymmetric peaks due to geometric, hardware and other effects [11–15]. However, the appropriateness of the traditional Gaussian/Lorentzian/Pseudo-Voigtian profiles in full-pattern refinement has received little attention, an exception being e.g. [16]. Furthermore, there are potentially great benefits to be gained by undertaking full-pattern analysis of the type routinely used on pulsed source neutron diffraction instruments [17] in terms of shorter acquisition times, better grain sampling statistics and the averaging over many reflections to negate the influence of elastic and plastic anisotropy as well as texture, and the mentioned low instrumental resolution due to insufficient grain sampling. It is encouraging that uncertainties in determining the lattice parameter of similar magnitude to that required for strain measurements have been found by EDXRD during high-pressure experiments using a novel type of pattern fitting code [18].

3. Cracked austenitic stainless steel specimen

The sample material, austenitic steel of type 316H, was kindly provided by British Energy Plc., UK, and is a commonly used engineering stainless steel with a composition of mainly Fe, 17% Cr, and 11% Ni with minor additions of Mo and Mb. This alloy has a relatively low yield (0.2% proof) stress of around 200 MPa but relatively high-ultimate tensile stress of around 500 MPa. Several nominally identical specimens (labelled CT1 to CT5) were cut to compact-tension (CT) geometry. A schematic is shown in Fig. 1 (right). A 25 mm sample thickness was chosen in order to generate a triaxial stress state within the bulk. It is reasonable to assume that the residual strain field at mid-thickness is approximately *plane strain*, which allows the use of an elongated gauge volume (inevitable at high energy). A residual stress field was generated in four samples by preloading by compressing each sample beyond yield at the points indicated, and then releasing the applied force. This plastically deforms the material locally around the notch. Upon removal of the ap-

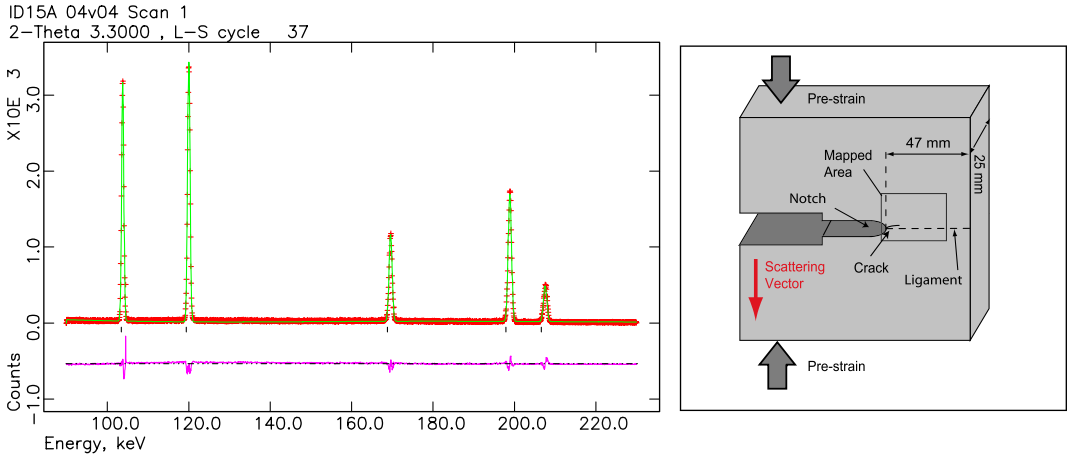

Fig. 1. (Left) A typical refined EDXRD spectrum of the CT steel specimen in the energy domain. Only the part of the spectrum visible in the figure (the five lowest reflections) has been used in the refinement. The difference curve indicates the use of an inappropriate Gaussian profile function. (Right) A schematic of the CT sample, illustrating the location of the crack near the notch, and the position where the pre-strain was applied (see also Fig. 2).

plied force, the plastically deformed notch regions become residually tensively stressed in the direction of the applied preload. In one sample (labelled CT1) a 0.5 mm wide and 1 mm long pre-crack was electro-discharged machined at the notch, from which a fatigue crack of 2 mm length was introduced (see Fig. 2). The results presented have been obtained from this sample. One sample was left undeformed in order to have an unstressed reference specimen. The typical grain size in this material is between 50 and 100 μm. The sample was mounted on a translation stage and placed in the calibrated rotation centre of the experimental set-up. An area of $14 \times 10\ \text{mm}^2$ has been mapped with a gauge volume defined by $0.4 \times 0.4\ \text{mm}^2$ incident beam geometry at $2\theta = 3.5°$, with the scattering vector (the direction of strain measured) parallel to the initial loading direction. The length of the resulting gauge volume was approximately 15 mm. Counting times were 50 s per point. The total map consisted of around 800 measurements points, which is equivalent to around 20 h of counting time. Fig. 1 (right) shows a schematic of the CT sample, the location of the crack and the scanned area.

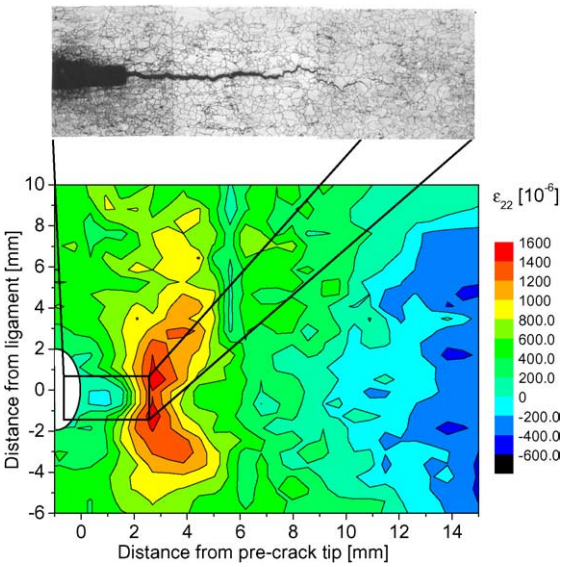

Fig. 2. The elastic strain ε_{22} measured in the vicinity of the crack tip for specimen CT1. The apparent vertical feature in the centre of the sample (at 6 mm) is due to missed data points when spectra were lost during a beam refill. Also shown is the location of the crack (image above) with respect to the measured strain field.

4. Data pre-processing and analysis

The results presented in this paper were prepared using a Pawley [19] refinement approach, using the software package GSAS [20] in intensity extraction mode. Although GSAS is intrinsically

able to perform refinement on energy dispersive data, it only provides a Gaussian peak profile for its analysis. However, it is well known that the electron-hole pair creation process in the detector and the beam divergence as well as slit settings can lead to asymmetric peak profiles the peak profile in EDXRD spectra are of asymmetric nature [12–15]. In our approach the collected EDXRD spectra were transformed to an artificial, reciprocal scale, essentially equivalent to that of time-of-flight, and then stored in GSAS RALF format using logarithmic re-binning, which then allows the use of the asymmetric peak profile (No. 3) developed for TOF neutron diffraction in GSAS. A similar approach, but using a transformation to the angular dispersive domain has been adopted and reported by [16]. Any Compton scattering contribution is assumed to be included in the refinement of general background.

Our approach was driven by the observation that although the individual peak fits, as well as Pawley refinements of the data in the energy domain using symmetric profiles achieved reasonable fits with very good lattice parameter uncertainties (of the order 10^{-6}) but the resulting strain profiles were relatively poor with very large point-to-point scatter of the order 10^{-3}. Fig. 1 (left) shows the typical diffraction spectrum obtained on ID15, the fit, and the difference curve.

5. Results and discussion

Fig. 2 shows the longitudinal strain map obtained by EDXRD, compared with finite element prediction in Fig. 3, which are in very good agreement. Additional validation by neutron diffraction of the presented results exist [1], but have been omitted for sake of brevity. We have demonstrated the ability to use full-pattern fitting technique on energy dispersive synchrotron X-ray diffraction data for strain scanning on relatively thick engineering components. It was found that for the given settings an asymmetric peak profile significantly improves the strain profile characterisations and uncertainty. A (point-to-point) lattice strain resolution of the order 10^{-5} was routinely achieved. The spatial resolution of 0.4 mm was limited by the sample microstructure. This method has the potential to significantly enhance strain measurements on engineering alloys even in relatively thick sections with high-spatial resolution, particularly in situations where the stress field varies sharply in two-dimensions, such as around cracks. The asymmetry can to a large extend be attributed to the large slits opening gaps. Reducing the slit gap and working with fine-grained materials should therefore increase the resolution significantly.

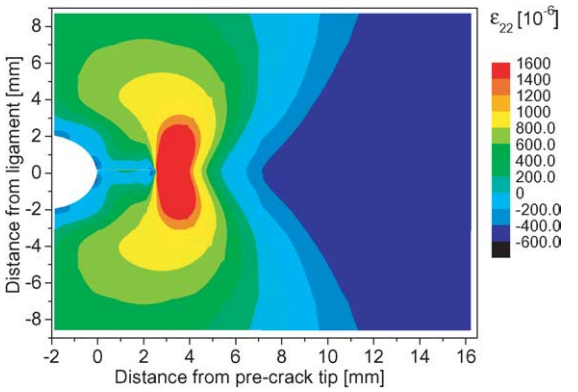

Fig. 3. The elastic opening strain ε_{22} as predicted by the 3D finite element analysis for a cracked specimen (CT1). Compare with Fig. 2.

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