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A synchrotron X-ray diffraction study of in situ biaxial deformation

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Abstract—The biaxial deformation of a ferritic sheet steel has been examined using high energy in situ X-ray diffraction. A purpose built biaxial loading mechanism was constructed to enable deformation across a wide range of strain ratios. Three nominal deformation conditions were compared: (1) uniaxial loading, $\epsilon_{TD}/\epsilon_{RD} = -\nu$, (2) biaxial deformation where $\epsilon_{TD}/\epsilon_{RD} = 0.4$, and (3) approximately balanced biaxial deformation, with $\epsilon_{TD}/\epsilon_{RD} = 1.5$. This novel setup allowed the full Debye–Scherrer diffraction rings to be acquired during arbitrary selected strain-paths, permitting lattice strains and reflection intensities to be measured across an unrivalled grain orientation range for such deformation conditions. This experiment reveals that the accumulation of lattice strain during deformation, as a function of azimuthal angle, is highly sensitive to strain path. For the $\epsilon_{TD}/\epsilon_{RD} = 1.5$ strain path, whilst lattice strain accumulates most rapidly in the ϵ_{TD} direction during early stages of plastic deformation, the lattice strain is shown to distribute almost perfectly isotropically for the observed orientations when plastic strain is high. This was found to be in contrast to strain paths where $\epsilon_{TD}/\epsilon_{RD} \ll 1.5$, demonstrating that lattice strain magnitudes remain highest in the direction parallel to the tensile axis with the highest applied load. Furthermore, the technique provides the capability to observe the evolution of texture fibres via changes in reflection intensity during different applied strain ratios.

Keywords: Synchrotron radiation; X-ray diffraction (XRD); Biaxial deformation; Texture; Lattice strain

1. Introduction

In a metallic material, deformation cannot be assumed to be accumulated homogeneously at a crystal level. For a single phase metallic material, this inhomogeneity will be influenced by a vast array of factors including crystal structure, grain size, morphology, orientation and distribution, which together influence the yield, plastic flow and failure of the material. The material behaviour is further complicated for a multiphase material where the partitioning and/or localisation of deformation will be influenced by the independent properties of the constituent phases. This inhomogeneous patterning of deformation results in a distribution of stress both at a macroscale (type I) and on a microscale (type II & III) [1]. The mechanical response of a material will be influenced by the superposition of these stress contributions, which if quantified, will help the understanding of deformation phenomena on a crystal scale.

Experimentally, only a limited number of techniques are capable of measuring the deformation at a crystal level. Electron backscatter diffraction (EBSD) [2], X-ray diffraction [3], and neutron diffraction [1] methods can provide suitable measurements of localised deformation. Each method has advantages and drawbacks, and hence a suitable selection will be dependent on the microstructural and/or micromechanical feature of interest, in conjunction with its length scale. EBSD may be used to describe local elastic strains to high sensitivities [4], though these measurements can practically only be made across a limited number of grains. Neutron diffraction has been successfully used to measure the evolution of lattice strains (e.g. [5]) through thick samples. Although in situ measurements are possible, neutron diffraction data collection times are typically too long for dynamic experiments. Testing via high energy synchrotron X-ray diffraction offers rapid data acquisition times with the ability to measure the micromechanical response [6]. However, the sample thickness may be restricted due to energy dependent attenuation.

The micromechanical behaviour of a material when subjected to deformation can be revealed when tested in a uniaxial manner, however, this understanding is unlikely to be satisfactory to explain the response to more complex, multiaxial stresses experienced during fabrication or service. For example, sheet forming operations such as drawing, stamping or stretch forming may subject material to a wide range of biaxial strain paths. Due to the extensive use of such processes for components used in the transport sector, any performance or cost saving benefit from optimisation of sheet forming methods are highly desirable. Improvements may only be exploited when the micromechanical responses are

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well understood. Furthermore, understanding residual stresses left after biaxial forming is important due to their potential influence upon component performance in service. By replicating biaxial forming processes experimentally, the knowledge gained can lead to more intelligent component design and manufacture. These measurements may also assist the calibration or validation of models when compared to simulated results [7].

Experimentally, the most obvious way of replicating the biaxial strain paths observed in real components is via the testing of cruciform specimens. However, the number of variables in the geometry is high, making optimisation of the specimen design difficult [8]. In spite of this, examples of successfully testing cruciform specimens do exist (i.e. [9]). Furthermore, experiments that acquire diffraction data from cruciform-type specimens have also been reported. For example, a small biaxial stage designed for thin metallic films of the order of 150 nm thick on polymeric substrates has been installed on the DIFFABS-SOLEIL synchrotron X-ray beamline [10]. Also, a multi-axial test rig has been recently installed onto the neutron time-of-flight diffractometer POLDI allowing deformation of metallic cruciform specimens, as demonstrated with stainless steel [11].

Whilst it is desirable to measure the micromechanical response of biaxial deformation, in practice testing in this manner is far from trivial, as evident from the limited number of biaxial deformation/diffraction experiments reported. Marin et al. [12] used a method of axial loading of a pressurised austenitic steel tube to control the hoop and axial stresses in the material in conjunction with neutron diffraction measurements to obtain lattice strain. An axial strain up to 3–4% was possible. This method has the advantage that the directions of the principal stresses remain constant for different strain ratios, though has a disadvantage that the radial and circumferential stresses vary through the thickness of the tube. A biaxial stress can also be achieved with the application of simultaneous tension and torsion on thin walled tubes in combination with X-ray diffraction (ex situ), [13], though the maximum shear strain was limited to 2% to prevent buckling of the tube. Other examples of X-ray diffraction to measure biaxial deformation include the measurement of an aluminium alloy during deep drawing (reflection diffraction at different orientations) [14], and the use of a Marciniak flat bottom ram [15] to perform balanced biaxial tests [16].

In this study, a purpose built biaxial loading mechanism has been used to enable, for the first time, in situ biaxial deformation measurements from synchrotron X-ray diffraction of metallic sheets. The experiment uniquely enables a rapid rate of data acquisition, recording the full Debye–Scherrer diffraction geometry. This describes the material response across a wide grain orientation range necessary to capture the micromechanical behaviour of a biaxially deforming specimen. This experimental method is shown to be valuable in assessing the deformation response of ferritic sheet steel through a selection of strain paths.

2. Experimental

The deformation of a single phase low carbon ferritic steel, denoted as DX54, has been studied. The nominal composition of this material is given in Table 1. As received, the material had been cold rolled and galvanised with a thickness of approximately 1 mm. Prior to all characterisation in this study, the galvanised zinc coating was removed by abrasive media.

Table 1. Chemical composition of the ferritic steel, DX54 [17].

Element	Fe	C	P	S	Mn
wt.%	Balance	$\leqslant$0.06	$\leqslant$0.025	$\leqslant$0.025	$\leqslant$0.35

The as-received condition of the material was examined using electron backscatter diffraction (EBSD) to characterise the texture, grain size and grain morphology. This was performed using a JEOL-6500F scanning electron microscope, operating at a beam current of 14 nA with an accelerating voltage of 20 keV, and equipped with a TSL/EDAX OIM v6 system. Data were collected with a 1 μm step size over an area of 900 μm × 900 μm and an acquisition time of 0.05 s per pattern. Each diffraction pattern was collected with a 1000 × 1000 pixel camera with 4 × 4 camera binning.

High energy in situ X-ray diffraction experiments were performed on the I12 beamline at the Diamond Light Source. A Shimadzu AGS-X 10 kN load frame was placed on the beamline sample stage orthogonal to the incident X-ray beam. Fitted within this load frame was a bespoke biaxial loading mechanism, based upon a design described by Brieu et al. [18], purpose built for use in this experiment. The rig itself is uniquely capable of deforming a cruciform shaped specimen, simultaneously pulling on all 4 arms to provide a biaxial stress state in the central region of the specimen. To illustrate the movement of the mechanism, computer aided design (CAD) drawings are shown in Fig. 1; in (a) the starting configuration and (b) the final configuration following displacement of the topmost crosshead. In this example, the rig has been configured to provide an equal displacement in the vertical and horizontal directions. However, the diagonal rods, set to 45° in the illustrations shown can be adjusted to change the ratio between the vertical and horizontal displacements, thus changing the strain ratio. A photograph of the biaxial mechanism is shown in Fig. 1(c). During the development of the biaxial loading mechanism, finite element modelling was used, as illustrated in Fig. 2 for example, to assist the mechanism design whilst ensuring the samples were subjected to the desired loading path(s). The design was adapted to ensure that load required to deform the sample would not cause plastic deformation in the rig whilst remaining close the desired strain ratio.

Prior to the experiment, the sample geometry was refined to ensure the sample would deform and fail in the desired region, and that the largest deformation was accommodated in the region where the X-ray beam would pass. The adopted geometry including selected dimensions is shown in Fig. 3(a). The features of the cruciform design can be seen in (a), including broad specimen tabs to fix into grips with pin holes to restrict sample sliding. The combination of waisted specimen arms, a thinned cross-shaped region (on each face of the cruciform) and thinned central disc-shape within the cross permits measurable plastic deformation in the centre of the specimen [19]. In the sections that have been thinned, the corners were chamfered to reduce stress concentrations, mitigating premature failure in these locations. During the design of the specimen, finite element modelling was used to ensure the specimen failure was in the desired location. An example map of Von Mises stress is shown in Fig. 3(b), showing the highest stress will be