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In-situ neutron diffraction during biaxial deformation

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ABSTRACT

A change in strain path may have a significant effect on the mechanical response of metals. In order to understand or even predict the macroscopic behaviour under such conditions a detailed knowledge on the microstructural evolution is crucial. Yet relatively little work has been done to quantify and understand how the inter- and intragranular strains are affected during a change in strain path. In this work we present a new multiaxial deformation rig that allows performing in situ proportional and non-proportional loading under neutron diffraction. We demonstrate the capabilities of this new setup for the case of a 316 L stainless steel. We show that the nature and magnitude of intergranular strain strongly depends on the applied stress state and demonstrate that micro yielding and internal strain recovery are responsible for the observed transient softening during a 90° strain path change. We anticipate that this new characterization method will provide previously inaccessible microstructural data that can serve as input for benchmarking current state-of-the-art crystal plasticity models.

1. Introduction

Engineering materials often experience complex strain paths during synthesis or under service conditions. A prime example is metal forming, which typically involves multiple operations including strain path changes during which transient and permanent changes in hardening rate are observed (see, for instance [1], and references therein). Many metals exhibit a lower yield stress (Bauschinger effect [2]) when the sign of the load is reversed after plastic deformation, in spite the hardening observed during the forward straining path. Some materials however exhibit a larger yield stress after the loading direction is changed, a phenomenon called cross-effect [3]. Furthermore, softening/hardening upon changing strain path can be a transient phenomenon or permanent where differences in hardening rate between the first and the second loading path maintain. These phenomena play an important role in manufacturing and have to be taken into account when setting forming limits.

The origin of such strain path dependence has to be found at the inter- and intra-granular level. Polycrystalline metals yield heterogeneously and this leads to inter-granular stresses, which remain after unloading and will have to be added to the stress applied in the next strain path. Dislocation slip, an inherent anisotropic mechanism, will develop anisotropic dislocation substructures within the individual grains, generating long range intragranular stresses that will have to be considered simultaneously with the intergranular stresses [4]. Incorporation of the above effects on the correct physical length scale through multiscale modelling can offer significant improvements in computer-aided engineering. Modelling non-monotonous and/or non-proportional deformation requires improved constitutive equations [1,5,6]. Different models have been put forward to incorporate for instance the Bauschinger effect [7]. These computational models necessarily need to take into account the evolution of the microstructure and in particular the development of internal stresses.

In the last decade in-situ x-ray and neutron diffraction has been extensively used to investigate the evolution of intergranular and intragranular stresses during deformation. The information has been directly compared with various crystal plasticity models [8–12]. Most studies are however restricted to uniaxial tension or compression tests. Some in-situ strain path change experiments

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have been performed, in particular tension-compression Bauschinger tests [13–15]. In 2013 Repper et al. [16] have performed the first in-situ neutron diffraction study on cruciform shaped specimen. They have demonstrated that during equibiaxial loading the evolution of lattice strain is quite different from that during uniaxial straining. Foecke et al. have implemented various devices inside a large laboratory x-ray diffractometer in order to measure multiaxial stress–strain curves and corresponding yield loci [17–19]. Here diffraction is used to obtain the macroscopic stress state in the sample using the $\sin^2\Psi$ method. Imbeni et al. [20] have performed in situ tension and torsion experiments on thin tubular samples of Nitinol during X-ray diffraction. Mehta and coworkers [21] used X-ray microdiffraction to map out multiaxial strain fields in Nitinol.

Recently it was shown that in case of a 90° strain path change the commonly used models fail to predict both the magnitude and nature of intergranular stresses that arise as a consequence of elastic and plastic anisotropy [22]. In order to further optimize crystal plasticity models there is a clear need for adequate in-situ devices allowing studies of engineering materials subjected to complex strain paths. Collins and co-workers [23] have developed a new setup to perform in-situ x-ray diffraction during biaxial deformation of sheets. They have studied the evolution of lattice strain and texture during uniaxial and biaxial tensile deformation of a cold rolled low carbon ferritic steel. It was shown that the distribution of lattice strain with respect to azimuthal angle is highly dependent on the applied biaxial strain ratio. Furthermore it was demonstrated that for strain ratios close to balanced biaxial the lattice strain initially accumulates more rapidly in the direction of the tensile axis with highest load, whereas at larger plastic strain the distribution becomes more uniform.

High-resolution reciprocal space mapping with high energy x-rays is a powerful method to investigate in detail the microstructural evolution during a strain path change. This has been demonstrated extensively for the case of Cu [4,24–26]. Wejdemann et al. have [4] demonstrated that during an orthogonal strain path change two different regimes can be distinguished: a microplastic regime during which the elastic stresses are significantly altered and only subgrain plasticity occurs, and a macroplastic regime where a new microstructure is formed. Unfortunately such measurements are rather time-consuming.

Some x-ray and neutron beam lines are now equipped with load frames that allow in-situ mechanical testing under multiaxial stress state and/or to perform strain path changes. At the DIFFABS beam line (SOLEIL, France) a biaxial deformation rig has been installed, which allows for in situ characterisation of thin polycrystalline films deposited on a compliant substrate [27,28]. At the engineering neutron diffractometer VULCAN (SNS, USA) a large load frame has been installed which allows for combined tensile/compression/torsion experiments [29,30].

In this work we present a unique biaxial deformation rig that can be used for in-situ biaxial proportional and non-proportional testing during neutron diffraction at the beamline POLDI of the Swiss Neutron Spallation Source (SINQ). The rig exhibits two independent axes and a torsional unit, which allows to apply various multiaxial stress states and to perform complex strain path changes. We apply this method to study the evolution of the inter/intra granular stresses in a 316 L stainless steel known to exhibit transient softening when changing the strain path [22]. We show that the nature and magnitude of intergranular strain strongly depends on the applied stress state and demonstrate that micro yielding and internal strain recovery are responsible for the observed transient softening during a 90° strain path change. We anticipate that this new characterization method will provide previously inaccessible microstructural data that can serve as input for benchmarking current state-of-the-art crystal plasticity models.

2. Material and methods

2.1. Multi-axial deformation rig

The in situ multi-axial deformation rig has been developed in collaboration with Zwick/Roell (Ulm, Germany) and is based on an innovative modular approach, which allows for testing under various complex deformation modes. Fig. 1 (in web version) displays a picture of the rig mounted on the 3D translation and rotation stage of the POLDI beam line. It consists of a 100 kN standard load frame that is equipped with a 200 Nm torsion unit, which allows for both regular tension/compression tests and proportional and non-proportional biaxial tension/torsion tests. Additionally a 50 kN axis can be mounted perpendicular to the main axis. With this setup in-plane proportional and non-proportional deformation tests on cruciform-shaped specimen can be performed. With these various possibilities the rig allows covering large part of the stress space. In what follows we will focus on the in-plane biaxial setup. The machine is controlled with testXpert, the standard control software from Zwick/Roell. In order to control the machine remotely, an interface between testXpert and SICS (SINQ Control System) has been written. This allows for full control of the deformation rig within the standard POLDI software environment.

2.2. Samples

The machine is designed for mounting planar cruciform shaped samples. Optimizing the shape of such samples is not a trivial task and has been topic of many studies (see, for instance, Refs [30–34]). Fig. 2 (left) displays a picture of the type of samples used in this work. The most important feature is the thickness reduction at the centre of the sample (from 10 mm in the arms to 3 mm in the gauge volume). The shape has been optimized with the aid of ABAQUS/Standard finite element modelling (FEM) [35]. Fig. 2 (right) shows the Von Mises stress distribution generated during equi-biaxial deformation. As expected stress concentrations occur at the notches, which results in local fracture at large levels of deformation. Therefore the value for the thickness reduction is a result of a trade-off between optimizing the volume for neutron diffraction and the possibility to reach at least 20% plastic strain in the centre, prior to fracture as the notches. For comparison regular flat dogbone shaped specimen have been prepared. The gauge sections of these dogbones exhibit the same thickness as compared to the cruciform shaped samples. This ensures that the neutrons sample the same volume taken from the same section of the original material.

The material under investigation is a warm rolled 316 L stainless steel with composition 17.25Cr–12.81Ni–2.73Mo–0.86Mn–0.53 Si–0.02C (wt%) purchased from ThyssenKrupp in a sheet thickness of 10 mm. The outer shape was cut using waterjet cutting whereas the thickness reduction is achieved by mechanical grinding. To learn more about the microstructure of the material corresponding with the region where the neutron diffraction data were collected, EBSD (electron back scatter diffraction) measurements were carried out at the center of the sheet plate (after etching away a 5 mm thick layer). Fig. 3 (left) shows a crystal orientation map of a representative area. The rolling and normal directions are indicated. The transvers rolling direction is perpendicular to the plane. As can be observed, the grains are equiaxed with an average grain size of 7 μm. On the right hand side of Fig. 3 the inverse pole figures for the three principal directions are shown. These show that there is no strong texture in the plane of the sheet. There are however more grains having a <001> or a <111> direction along the rolling