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In-situ biaxial mechanical testing at the neutron time-of-flight diffractometer POLDI

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Abstract. Complex strain paths are often applied to materials during production processes. This paper shows the first successful in-situ biaxial mechanical tests during neutron diffraction performed on a cruciform steel sample and reports on the differences compared to uniaxial deformation. Digital image correlation is demonstrated to be an appropriate tool to monitor spatially resolved the straining of the sample caused by the applied force. Additionally we present the new multiaxial mechanical test rig of POLDI. Beside uniaxial in-situ testing the modular test machine set-up will allow in-situ tension/compression testing with neutrons on cruciform samples. The uniaxial set-up is equipped with a torque actuator.

Introduction

Uniaxial deformation is the most widely used mechanical test procedure for metals. Because applications of metals usually involve more complex strain paths there is a need to move away from simple uniaxial testing towards more multiaxial loading conditions [1, 2, 3]. Multiaxial testing allows to simulate even complex strain paths, which are often applied to materials in the production route of industrial components. Diffraction methods provide non-destructively and phase-sensitively information about the sample. When performed with neutrons possessing a high penetration depth, they deliver information from deep inside the material [4]. Neutron diffraction experiments combined with mechanical tests provide information on the evolution of the microstructure during deformation. These so-called in-situ experiments are frequently used to understand and explain the thermo-mechanical behavior of bulk materials [5, 6, 7]. Also at large facilities one has recognized the need for extending mechanical test facilities beyond the uniaxial regime. Thus, many beamlines are equipped with devices for simultaneous tension/compression and torsion experiments [8, 9, 10]. The only machine allowing to individually activating two in-plane strain axes is at DIFFABS (Soleil) but designed only for thin films on stretchable polymers [10]. For the moment there are no in-plane biaxial test rigs available at neutron beamlines allowing to independently activate two principal axes of strain. Space restrictions and the demand of a free flight path for neutrons limit the possibilities to install commercially available devices at existing beam-lines.

The current paper presents first results of a biaxial mechanical in-situ test performed during neutron diffraction on cruciform stainless steel samples with a biaxial test machine that was temporarily installed at the neutron time-of-flight diffractometer POLDI. Whereas lattice strains were determined by neutron diffraction, the straining of the sample caused by the applied force were measured spatially resolved using a digital image correlation system. In addition, the paper introduces the mechanical biaxial test machine, that will be installed in summer 2013 at POLDI. The device will also be suitable for uniaxial tension/compression/torsion experiments.

Neutron diffractometer POLDI

The measurements presented in this paper were performed at the **P**ulsed **O**ver**l**ap **Di**ffractometer POLDI, which is a neutron time-of-flight (TOF) instrument located in the target hall of the SINQ spallation neutron source of the Paul Scherrer Institute in Switzerland. POLDI consists of a chopper system, a neutron guide including a focussing neutron mirror, and a time and angle resolved ^{3}He wire chamber detector. The centre of the detector is located at approx. $2\theta = 91^\circ$ and covers a 2θ range of $\pm 15^\circ$. POLDI works with a thermal wavelength distribution of 1 to 6 Å. The TOF character of POLDI allows recording simultaneously all Bragg reflections which are accessible with this wavelength range at the same time. This is particularly advantageous for the study of the development of intergranular and/or interphase stresses during in-situ testing, where it could be necessary to record all $\{h\,k\,l\}$ diffraction peaks at the same time to obtain information for the same stress/strain state and the same microstructure, e.g. for samples highly affected by creep. A detailed description of the instrument concept is given by Stuhr [11, 12].

Sample

A cruciform 316L stainless steel sample (17.25Cr-12.81Ni-2.73Mo-0.86Mn-0.53Si-0.02C (w%)) was used for the biaxial in-situ mechanical test. The sample shape was defined by finite element simulations and cut by wire electrical discharge machining (EDM). Each arm of the cruciform sample has a total length of 250 mm. The thickness of the sample close to the grips is 5 mm. In the centre region of the sample the thickness amounts 2 mm. This geometry ensures plastic deformation in the centre of the sample, where the neutron beam defines the gauge volume (figure 1).

Experimental details

A biaxial test machine designed by the department for nuclear energy research (NES) of the Paul Scherrer Institute was used for the in-situ experiments at POLDI. The machine is usually operated in

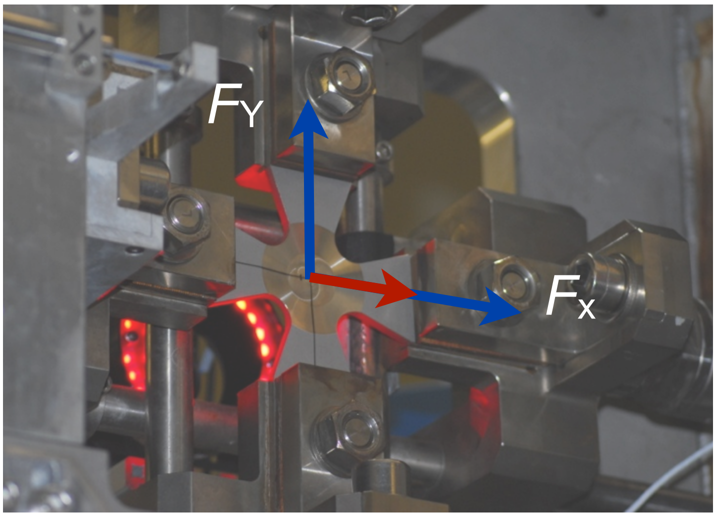

Fig. 1: Cruciform steel sample clamped in the biaxial test machine, which is mounted on POLDI. The red lights in the background belong to the camera system, which monitors the macroscopic straining of the sample. The measurement direction of the neutrons (short arrow) was in-plane and pointed in the direction of $\mathbf{F}_x$.

horizontal mode and was not dedicated to neutron diffraction measurements. To access the in-plane strains of the sample by diffraction it was necessary to position the entire biaxial machine vertically. No horizontal movement of the biaxial test machine was possible once mounted on the POLDI sample table. As assumed due to the symmetric sample geometry and proofed by FEM simulations, the strain state through the thickness of the sample was constant, assuming a homogenous materials microstructure. Thus, a GV of 3.8 x 10 x 3.8 mm^3 was chosen in order to obtain good counting statistics in a reasonable time by averaging over the thickness (2 mm) of the sample. The measurement gauge volume was positioned 5 mm out of the centre of the sample, necessary to avoid beam shielding by test machine components. Load ratio in the arms (x and y directions) was set to 1:1. The measurement direction was in-plane, parallel to the load F_X applied in direction of one of the arms (figure 1). Neutron diffraction measurements were carried out at 8 different loading steps. Additional measurements were carried out at unloads after 20 and 28 kN and after a reload to 20 kN. The load was kept constant during measurements. The measurement time for each point was approx. 20 min. To measure the straining of the sample caused by the applied force digital image correlation (DIC) was used. For this reason a speckle pattern was applied to one side of the sample. The speckle pattern was observed by a digital camera installed ca. 12 cm away from the sample. This set up allowed observing the surface straining of the full central region of the sample (figure 2). Due to the assumed uniformity of the strain state through the thickness of the sample, the measured surface strains represent the straining of the sample caused by the applied force. Analyzing these strains only for the area, where the neutron diffraction gauge volume was positioned, enables drawing the relation between straining of the sample due to the applied force and lattice strains measured by neutron diffraction.

Results and Discussion

Figure 2 shows the results of the digital image correlation analysis. The figures compare the values of the macroscopic straining of the sample in x and y directions respectively for a maximum load of 28 kN. The maximum of the strain distribution is situated close to the arms and is similar for the x and y directions. The strain distribution in the central region shows however slight differences between the x and y directions. These differences may result from slightly different performances of the two actuators, which might be related to the vertical orientation of the machine. These slight inhomogeneities in the strain distribution play a minor role, as the neutron gauge volume averages over a large material

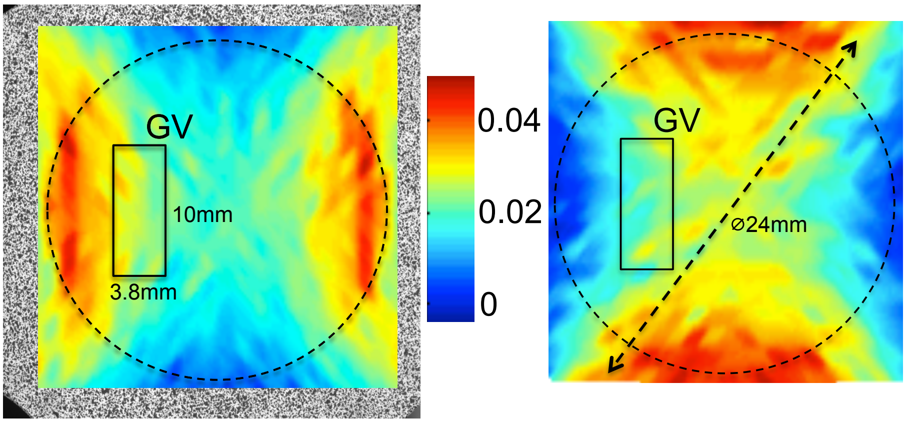

Fig. 2: Raw strain in x (left) and y (right) in-plane directions of the cruciform sample analyzed by digital image correlation (DIC). The software analyzes the movement of chosen points in the raw data image (background left picture) and plots them as raw strain values. The position of the gauge volume (GV) is sketched as well as the disc-shaped area in the center of the sample with a constant thickness of 2 mm.

area. Thus, DIC allows contact-free monitoring the homogeneity of the applied strain and provides information on the strain distribution in the sample, which is caused by the applied force. This is crucial for such an experiment as there is no other possibility to directly access the strains in such a cruciform sample.

Figure 3 shows the lattice strain evolution for the lattice planes {111}, {200}, {311} and {222} as function of the x component of the macroscopic straining of the sample (figure 2 left). The graph shows very similar behavior for the {111} and {222} planes, as expected from the crystal symmetry (fcc). Load sharing between the {111}, the {200} and the {311} planes was observed: After unloading the {111} and {222} planes remain in tension, whereas the {311} and {200} planes are in compression. This is in contrast to results reported in literature, gained from uniaxial load experiments at samples consisting of similar stainless steel (18.25Cr-13.42Ni-3.66Mo-1.48Mn-0.44Si-0.02C (w%)) [7]. In uniaxial test experiments the {200} planes perpendicular to the loading direction usually accumulate large tensile intergranular strains. For those {200} planes, which are aligned parallel to the loading direction almost no intergranular strains are measured.

Most likely this change from tension (in uniaxial tests) to compression (in biaxial tests) is a direct result from the biaxial stress state, considering that there is only one free direction (out-of-plane, z direction) allowing compensation of the tensile load in biaxial testing, whereas there are two such directions in an uniaxial test.

New test machine for in-situ testing

A new modular multiaxial test rig set up will be installed at the time-of-flight neutron diffractometer POLDI. The modular set up consists of two frames and is schematically pictured in figure 4. The first frame may be used as uniaxial test rig for tension/compression and low cycle fatigue tests with a maximum load of 100 kN. Here, the behavior of lattice planes may be studied with planes aligned both parallel and perpendicular to the loading direction. The influence of shear strains can be investigated in combined tension/compression/torsion tests due to an additionally installed torque actuator with a maximum angular moment of 200 Nm. The second frame completes the biaxial test set-up. While the

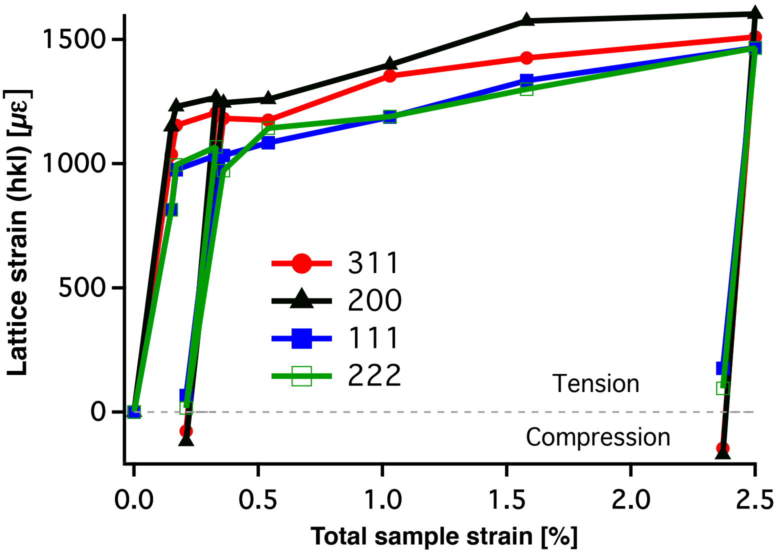

Fig. 3: Lattice strain measured by neutron diffraction against total sample strain caused by applied force obtained by digital image correlation for the different crystallographic directions {111}, {200}, {311} and {222}.

first frame has a capacity of 100 kN the second frame / axis is limited to 50 kN. Similar to uniaxial experiments lattice strains parallel and perpendicular to the load directions are accessible in biaxial operation. Tension/Compression and low cycle fatigue tests can be performed in biaxial operation mode. The electro-mechanic actuators will allow low cycle fatigue testing with a max. frequency of 1 Hz and a max. amplitude of ca. 5 mm. The set up with four actuators holds the sample in the centre of the test rig. In that way the neutron beam sees always the same sample area and thus the strains are measured for all lattice planes at the same microstructure. Different load ratios and changing strain paths will be applicable on cruciform samples.

A specially designed sample table (not shown) will allow performing line scans along the arms of the cruciform specimen as well as normal to its surface. The sample table will include also an in-plane (θ) rotation. The machine will be equipped with a digital image correlation system to measure straining of the sample caused by the applied force. The DIC system can be run without problems during the neutron diffraction measurements. This new mechanical test machine, built by the German company Zwick, will be installed on POLDI in summer 2013 and used for in-situ and ex-situ experiments.

Conclusion

The experiment presented demonstrates that biaxial in-situ testing during neutron diffraction using a cruciform test specimen is feasible. The observed strain evolutions showed significant differences from uniaxial experiments on similar materials. It is planned to perform uniaxial in-situ testing on samples consisting of the identical stainless steel as used for the biaxial test. More experiments including texture studies as well modeling efforts are needed to understand the particular behavior. It was shown, that digital image correlation is an appropriate method to monitor spatially resolved the total straining of the sample and to evaluate the homogeneity of the strain distribution. Finite element simulations are necessary to optimize the sample shape and evaluate the stress level in the sample. A new test rig concept dedicated for biaxial and uniaxial in-situ and ex-situ testing will be installed at the neutron time-of-flight beam line POLDI in summer 2013. This new test rig will allow performing

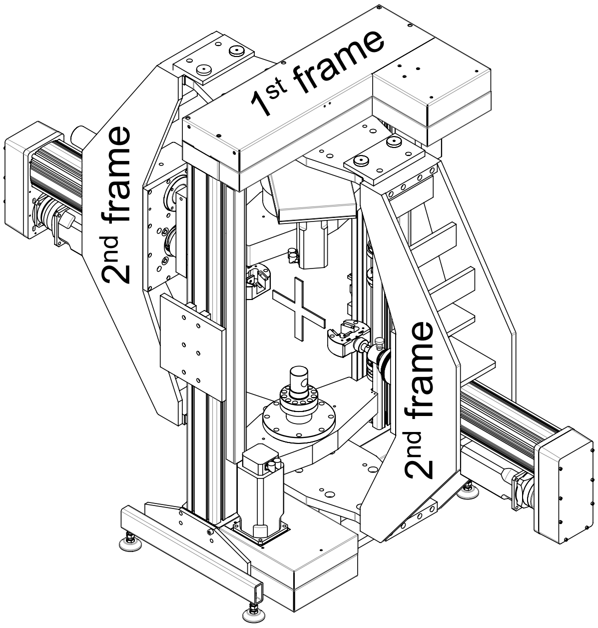

Fig. 4: Schematically picture of the new POLDI test rig from Zwick, Germany. The torsion actuator is equipped at the first frame.

experiments including changing strain paths, different load ratios and/or different pre straining of the sample. During mechanical testing the whole test machine will be moveable in order to scan the gauge area by neutron diffraction.

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