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HIPPO/CRATES - In-situ deformation strain and texture studies using neutron time-of-flight diffraction

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Abstract

In situ *deformation studies by diffraction allow studying of deformation mechanisms and provide valuable data to validate and improve deformation models. In particular, deformation studies using time-of-flight neutrons provide averages over large numbers of grains and allow to probing the response of lattice planes parallel and perpendicular to the applied load simultaneously. In this paper we describe the load-frame CRATES, designed for the HIPPO neutron time-of-flight diffractometer at LANSCE. The HIPPO/CRATES combination allows probing up to 20 diffraction vectors simultaneously and provides rotation of the sample in the beam while under load. With this, deformation texture, i.e. the change of grain orientation due to plastic deformation, or strain pole figures may be measured. We report initial results of a validation experiment, comparing deformation of a Zircaloy specimen measured using the NPD neutron diffractometer with results obtained for the same material using HIPPO/CRATES.*

1. Introduction

An urgent need of methods for measurements of elastic and plastic strains on a microscopic scale exists. Such measurements enable a better understanding of micromechanical mechanisms occurring during the elastic and plastic deformation of polycrystalline materials. Especially in materials with a high technical potential and susceptibility for premature failure caused by internal stresses, the importance of such measurements becomes evident. Metals with a hexagonal crystal structure, like Zirconium, Beryllium or Magnesium, are examples of such materials. Due to its applicability as light structural material, Magnesium is of great commercial interest, for instance in the automotive industry.

During *in-situ* deformation studies a specimen is deformed while irradiated by neutron or synchrotron radiation. Such deformation studies utilizing diffraction techniques to probe lattice strains and texture changes provide unique insight into active deformation mechanisms during plastic deformation of polycrystalline solids. Data gathered during such *in-situ* deformation is valuable to calibrate, validate and improve model calculations describing deformation behavior like finite-element or elasto-plastic and visco-plastic self-consistent models describing deformation. Successful modeling with constraints from both macroscopic data, such as the flow curve, and microscopic data, such as lattice plane dependent flow curves and the texture evolution, provides quantitative insight into the active deformation mechanisms [1]. Since diffraction is phase sensitive, multi-phase systems may be studied and the deformation of each constituting phase can be separated. Simultaneous macroscopic flow-curves can be measured using load-cells and extensometers.

The approach of *in-situ* deformation studies using neutron or synchrotron radiation is nowadays a well-established technique. Instrument such as SMARTS at LANSCE [2], ENGIN-X at ISIS [3] or EPSILON at IBR-2 in Dubna [4] are equipped with load-frames and utilize time-of-flight neutrons to measure lattice strains during deformation. The probed sample volume at such instruments is typically of the order of cubic centimeters, hence providing sufficient grain statistics even for coarse-grained materials (~100 μm grain size). However, none of these instruments provides sufficient detector coverage to measure also the texture changes during plastic deformation. Deformation studies at synchrotron facilities typically utilize the much smaller beam spot size to study deformation of thin films [5], nano-materials [6] or local damage

evolution in composites [7]. The so-called three-dimensional X-ray structural microscope technique allows measurement of strains spatially resolved in individual grains [8] or to follow the re-orientation of individual grains during plastic deformation [9,10]. The latter two techniques, however, currently do not allow measurement of large enough number of grains to allow comparison with models averaging over large numbers of grains with different initial grain orientations and orientations of adjacent grains. The local environment of each grain influences the observed deformation of individual grains. In-situ deformation using TEM also has its strength in studying individual grains and cannot provide sufficient grain statistics for modeling approaches of deformation of polycrystalline solids.

In this paper we describe the newly developed HIPPO/CRATES instrument. It allows texture measurements by utilizing the HIPPO (*Hi*gh *P*ressure – *P*referred *O*rientation) diffractometer [11,12] with its large detector coverage and the possibility to rotate the sample in the beam during application of stress in the CRATES apparatus. Similar to the above-mentioned neutron diffractometers it allows measurement of lattice strains averaged over sample volumes of the order of cubic centimeters, but provides information along many more sample directions. Neutrons, with their weak attenuation by most materials, allow measuring these quantities averaged over the bulk of the sample - a great advantage over X-Rays, which probe only the surface or small sample volumes in the case of Synchrotron Radiation. Only residual strains from the deformation applied in CRATES can be measured, i.e. relative to the initial deformation state. Any residual strain pre-existing in the initial sample cannot be reliably characterized since this requires extremely accurate alignment of the sample [13].

2. Design criteria

2.1 Overview

The limitations of the stress rig are given by a maximum load of 100 kN, converting for instance to a uni-axial stress of 1.2 GPa for a sample of 10mm in diameter, and a cross head velocity ranging from 10^{-4} mm/min to 5 mm/min. Various setups allow investigation of samples with cross-sections between 40 and 120 mm^2. For alignment, CRATES allows to move the sample in the stress-rig parallel to the loading direction and both horizontally and vertically perpendicular to the beam direction. Furthermore, rotation of the stress-rig increases the number of sample directions probed with the neutron beam. A schematic of CRATES in the HIPPO sample chamber is shown in Figure 1. Typical experiments with HIPPO/CRATES can be automated such that a complete loading/unloading cycle with interruptions at programmed load or strain levels for neutron data acquisition runs without user interaction.

The geometry of the stress-rig and the resulting eccentricity of the sample position in combination with the design of the HIPPO sample chamber require motion of the stress-rig into the experimental position after CRATES is loaded into HIPPO. The weight and the eccentricity caused a huge hinge moment of the positioning system, requiring correspondingly large dimensioning of components for the positioning system.

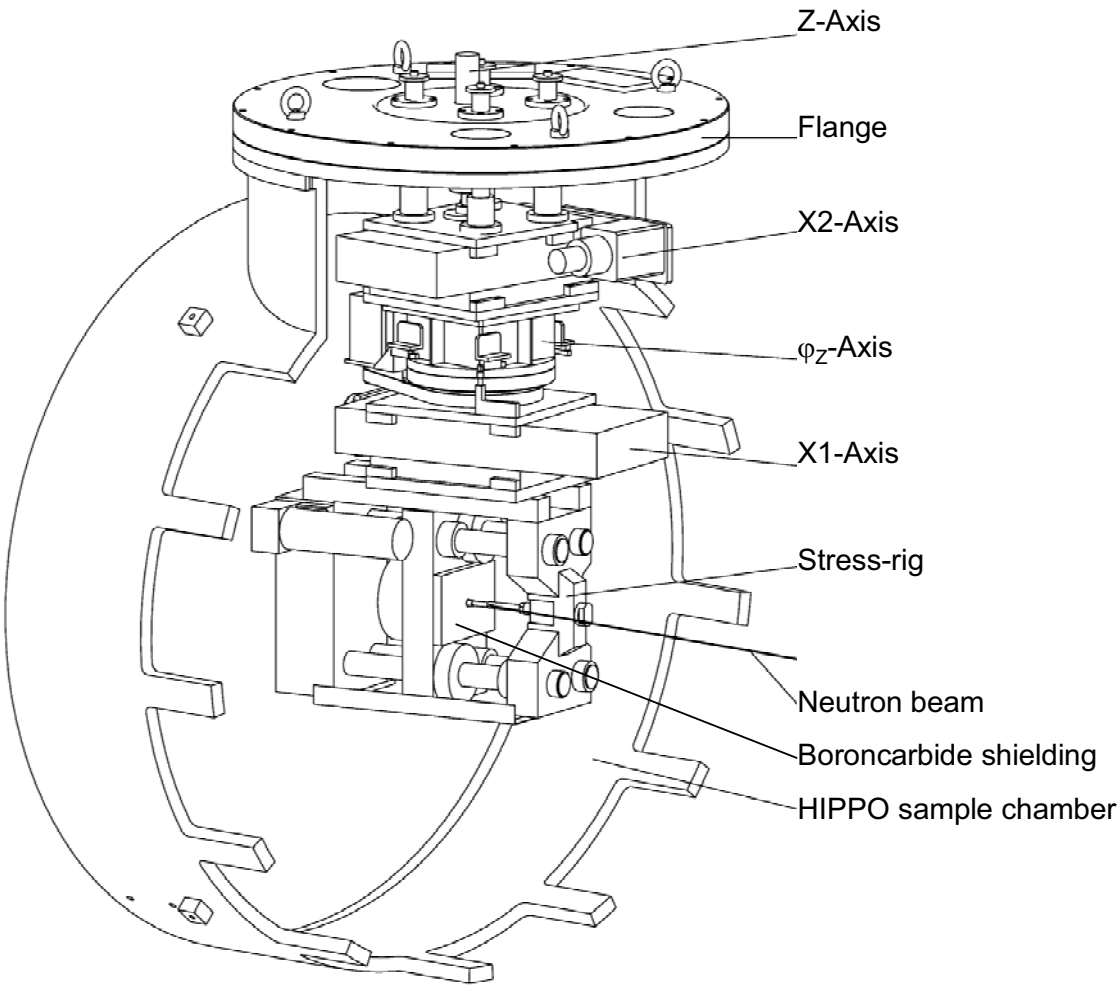

Figure 1 Schematic of CRATES in the HIPPO sample chamber.

2.2 Loading apparatus

TIRA Maschinenbau GmbH in Schalkau, Germany, designed the loading apparatus for CRATES. The stress-rig consists of two rigid crossbeams, connected by two shafts, and a driving crossbeam. Two threaded spindles move the driving crossbeam. The interchangeable sample holders are mounted in the driving crossbeam and the head crossbeam. They are furnished with threads into which the specimen is screwed. Figure 2 shows drawings specifying the geometry of CRATES specimen.

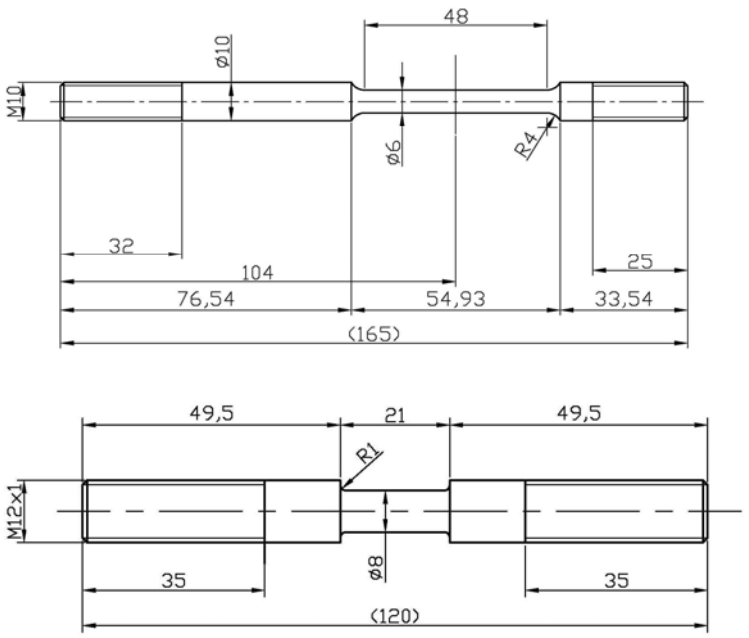

Figure 2 Drawing of the two specimen geometries for tensile samples (top) and tension/compression samples (bottom). All dimensions in millimeter. Compression only samples can be cylinders up to 12 mm diameter.

During the experiment, an Epsilon extensometer with either 10 or 20 mm gage lengths is attached to the sample using rubber bands. The rubber bands do not result in diffraction lines as would conventionally used steel springs or clamps. The blades of the extensometer are shielded with a small Cadmium plate mounted in front of the extensomter. Figure 3 shows a picture of a sample mounted in CRATES.

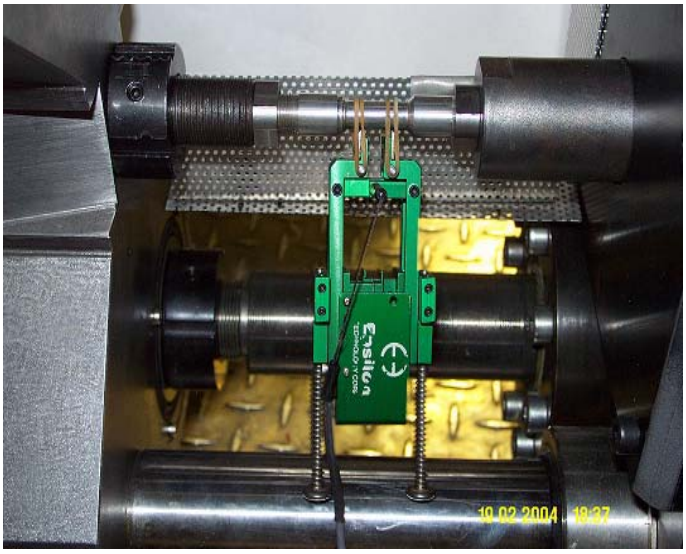

Figure 3: Photograph of a sample mounted into the CRATES load frame with the extensometer in place and the Cadmium shield aligned in front of the extensometer (beam is coming from the back towards the user).

The front sample holder can be removed with the sample through the head crossbeam, eliminating the need to touch a potentially activated sample. Due to the threaded spindles with a with a pitch of 1 mm, a belt drive and a motor with upstream planetary gear a small drive power of 100 W provided by a direct current motor is sufficient. This allows for the compact design required to minimize shadowing of detectors by the apparatus. Parts of the driving crossbeam are covered with boron carbide to prevent scattering from steel. The stress-rig has dimensions of 400×565×340 mm^3 and a weight of 131 kg. Possible crosshead velocities range from 10^{-4} to 5 mm/min (strain rate of 10^{-3} to 10^{-7} s^{-1} for sample with 20 mm gage length). The manufacturer specifies the stiffness of the apparatus with 1.3×10^{-5} mm/N. A machine-specific controller (EDC, external digital controller), provided by TIRA, controls the stress-rig. The EDC communicates via RS232 with the control PC, running software TIRATest developed by TIRA. This software allows crosshead motion and readout of applied force and extensometer readout. TIRATest does not provide any interface to the neutron data acquisition system, for instance to hold the load constant at a given level to count neutrons. TIRATest was automated using the software Macro Scheduler from MJT Net Ltd, London, UK, which sends out pre-programmed keystrokes and mouse motion. A LabView program, running on the same machine as TIRATest, receives new load levels and crosshead speeds from the main data acquisition PC via the EPICS interface [14] and sends the appropriate user input to TIRATest using Macro Scheduler. Similarly, it obtains the current load-level and crosshead velocity from TIRATest and transfers them back to the main data acquisition PC to control the experiment.

2.3 Motion

Main component of the X1 (parallel to the loading axis) and X2 (horizontally perpendicular to the beam direction) motion axes are heavy-duty linear tables SLT-15 provided by Neff Antriebstechnik, Waldenbuch, Germany. Each table consists of two parallel mounted linear guides and a ball screw drive. The motion ranges of the X1 and X2 axes are 160 and 100 mm, respectively, and are monitored by encoders mounted to the stepper motors. The vertical motion axis, *Z*, consists of a worm gear screw Jack *Muli 2*, provided by Neff Antriebstechnik, and four shaft guides. A spindle nut is rotated via a worm gear and lifts or lowers trapeze spindle from which the other axes and the stress-rig are suspended. A stepper motor with encoder provide drive and path measurement. The total stroke of the vertical motion axis is 40 mm. Franke GmbH, Aalen, Germany, constructed the vertical rotation axis, φ_Z, as a welded structure with a pivot bearing *LDV 200*. A drive belt allows mounting the stepper motor sideways for a compact design. A rotation by 180° is possible. A stepper motor with encoder provides drive and path measurement. All four axes are equipped with inductive limit and home switches allowing safe operation and reliable homing. All electrical lines are fed through the top plate. Motion controllers and drivers are mounted on top of CRATES, reducing the cables required to operate the device to a TCP/IP network cable for the motion control, a serial cable for the stress-rig control and a power cable. These three cables can be easily fed through small conduits in the shielding, allowing to mount and align a sample outside the beam without the need to interrupt power or network for loading of CRATES into HIPPO, which would require homing and reset of deformation parameters (cross-head position and extension are reset upon power cycling). The motion part is controlled by a LabView program, which can communicate via EPICS with the main data acquisition PC for experiment automation.

3. Benchmark experiment

To validate the performance of CRATES a Zircaloy-2 compression specimen of 20 mm length and 8 mm diameter was deformed in HIPPO/CRATES. The same material was tested on the NPD instrument, providing a dataset for comparison. Figure 4 shows the macroscopic flow curves and one lattice strain response measured on the two instruments. The macroscopic flow curves, measured by force-cell and extensometer attached to the sample, agree well with each other for the yield stress and the elastic modulus. The ragged appearance of the flow curves in the plastic region is due to the holding at constant cross-head position for the neutron measurements during which the material relaxes.

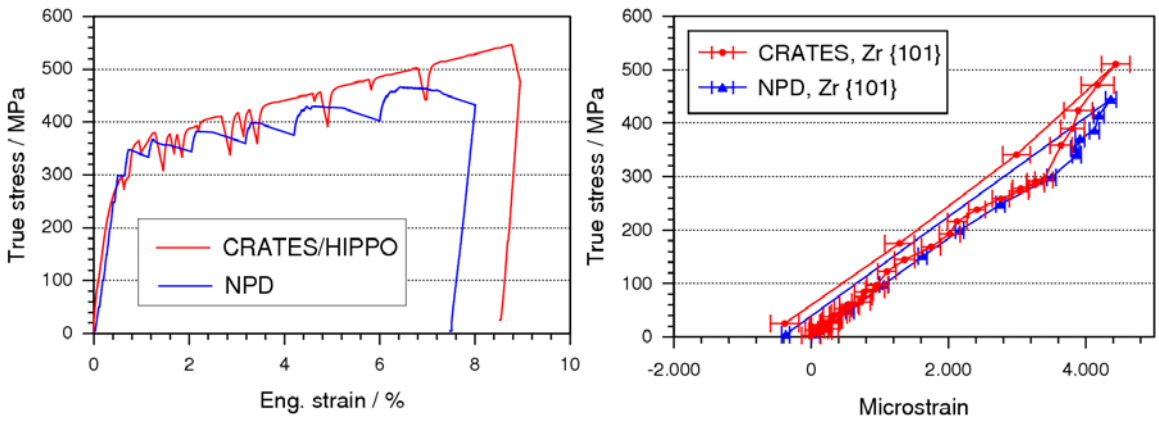

Figure 4: Flow curve of a compression test of a Zircaloy-2 specimen (left) and lattice strains of $\{10\bar{1}1\}$ *lattice planes in grains with their* $\{10\bar{1}1\}$ *lattice plane normal parallel to the loading direction. The same experiment was conducted on NPD and with HIPPO/CRATES.*

Lattice strains are derived from peak shifts which are analyzed using the single peak fitting feature of the GSAS program RAWPLOT [15]. The lattice strain responses measured on the two instruments are identical within error bars for the elastic region up to ~300 MPa. For higher stresses the differences in the load profiles, namely the different count times of 1 hour on NPD and 20 minutes on HIPPO/CRATES resulting in different amounts of relaxation, disallow comparison of the strains in that region. Due to its much larger detector coverage, HIPPO/CRATES probes about 20 sample directions simultaneously whereas NPD or its predecessor SMARTS only probe 2 sample directions. In each case lattice planes with d-spacings from ~0.4 Å are available. Figure 5 shows a comparison of the resolution of each detector bank for a CaF_2 calibration powder. Data on the same sample obtained on the high resolution SMARTS instrument are also shown.

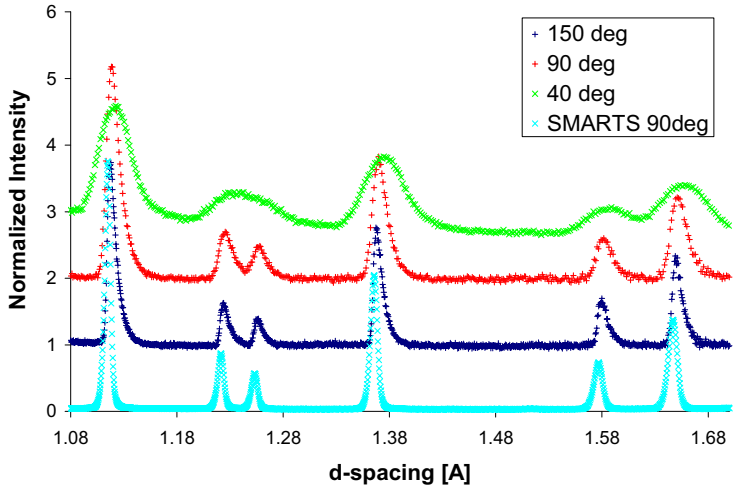

Figure 5: Comparison of the resolution of the three HIPPO detector rings with the high-resolution engineering diffractometer SMARTS. The increase in peak width (decreasing resolution $\Delta d/d$*) with decreasing diffraction angle* 2ϑ *can be explained by geometrical factors. The sample used for this comparison was* CaF_2 *powder.*

Initial results on a deformation study of Fe-Cu and the magnesium alloy MgAZ31 are reported elsewhere [16].

4. Summary

The load-frame CRATES for the neutron time-of-flight diffractometer HIPPO provides unique opportunities to study deformation *in situ*. The large detector coverage of HIPPO allows probing 20 sample directions simultaneously, providing data for strain and texture analysis which can be used to validate various deformation models and provide required material parameters. In an initial benchmark experiment result for a Zircaloy-2 sample agreed very well with data measured on the established NPD instrument.

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