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Thermo-mechanical processing in a synchrotron beam☆

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ABSTRACT

Well collimated, high energy X-rays of 90 keV from synchrotron sources have been used to study metals undergoing plastic deformation in situ, in real time and in the bulk of the materials. The spottiness of the Debye–Scherrer rings, showing reflections from individual crystallites, is analyzed to obtain grain statistics, mosaic spread and grain orientation. Upon cold deformation, coarse grained materials show fingerprints of subgrain formation, grain rotation, grain refinement and the evolution from a single grain into the asymptotic texture. Lattice strain, its partition and anisotropy can be simultaneously revealed. Heating of metals under continuous load drives the observation through the regimes of phase transformation and grain relationships therein, grain coarsening, dynamic recovery and dynamic recrystallization. Examples on copper, magnesium, twinning induced plasticity steel, zirconium alloy and titanium aluminium intermetallics are shown.

1. Introduction

The mechanical properties of a metallic product depend strongly on both phase composition and microstructure of the polycrystalline material. For example, copper work-hardens under deformation while it becomes soft under thermal treatment, related to grain and subgrain refinement and coarsening, respectively. These effects play a crucial role in applications such as airplane turbine blades or weldings in nuclear reactor vessels. Preferred crystallographic orientation, texture, influences the isotropic behavior of a material and may reflect in enormous cost and waste savings, as in the drink can production by mastering the deep-drawing process or improve the electric current density in high temperature superconductors. Therefore, most development efforts for the design of materials with novel or improved physical properties, like physical strength, ductility, formability deal with the control of the microstructure. Besides the basic physical parameters, the art lies in finding the right thermo-mechanical processing, which for highly designed materials, can be very sophisticated. After production, the lifetime of a metallic product depends on thermo-mechanic load, inducing non-desired effects, such as creep, which may alter the microstructure and thus the mechanical properties. Therefore, physical thermo-mechanic simulation is widely used in industry and research laboratories.

Conventionally, a metal is processed under realistic or model conditions at high temperature, with a defined time and stress profile and then quenched to freeze the structural state from that particular point in parameter space. To access its bulk properties, this specimen is then cut, polished and examined by microscopy or conventional X-ray diffraction in a surface-near region. In order to obtain the materials behavior along all time steps of the thermo-mechanical process, a second, third and many further specimens are prepared from slightly different temperature or stress states and subsequently analyzed in the same way. Obviously, this method is extremely time consuming and it is most doubtful, that the microstructure during the processing is still the same after quenching, particularly when inevitable phase transformations are involved.

Modern neutron and synchrotron X-ray sources are bright enough to study thermo-mechanical processes in situ, in real time, and from the bulk of the material. This elucidates the material behavior during heat treatment, or after heat treatment during cooling, and allows to define the best process temperatures for a specific material. Particular advantages of the neutron diffraction method are the extraordinary penetration, a good average over large volumes in grain statistics, and different atomic contrasts allowing to study order–disorder transitions.

Like neutrons, high energy X-rays from synchrotrons such as configured at the European Synchrotron Radiation Facility (ESRF) in France or the Advanced Photon Source (APS) in the USA show competitive deep penetration of centimeters into the bulk of the material [1]. In contrast, the synchrotron beam is sharply bundled and may reflect only from a small number of grains. With the

☆ Probing Strains and Dislocation Gradients with Diffraction.

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only recently available, time resolving large area detectors, high quality X-ray movies can be taken while the specimen undergoes thermo-mechanic simulation. Minute understanding of the X-ray diffraction process, the underlying crystallography and software development allow to extract novel and multi-dimensional information from the recorded patterns [2–5].

2. Experimental

The third generation synchrotrons APS and ESRF have been used for the present studies. The high electron-energy rings of 7 GeV and 6 GeV provide undulator radiation in the high energy X-ray regime into the beamlines 1-ID and ID15B, respectively. Silicon monochromators are used to define the incident beam at about energy $E = 90$ keV with wavenumber $k = 45.6\,\text{Å}^{-1}$ and wavelength $\lambda = 2\pi/k = 0.138$ Å feeding an experimental hutch containing a typical setup as given in Fig. 1. The $\varnothing \sim 100\,\mu$m beam impinges onto the polycrystalline sample of typical 4 mm thickness in transmission mode and is diffracted into Debye–Scherrer cones, which are recorded in about 1.0–1.5 m distance by a flat panel detector, oriented strictly perpendicular to the incident beam. The small diffraction angles of high energy X-rays allow to record simultaneously full diffraction rings up to typically $10\,\text{Å}^{-1}$. At APS we used the amorphous silicon pixel detector of General Electrics. It consists of 2048×2048 pixels of $200\,\mu\text{m} \times 200\,\mu$m size and the electronic hardware was set up to record continuously up to 300 images, here up to 5 Hz frame rate. The ESRF detector was a Pixium 4700 flat panel amorphous silicon detector [6] with $1920\,(\text{h}) \times 2640\,(\text{v})$ pixels measuring $154\,\mu\text{m} \times 154\,\mu$m, continuously streaming with 2.5 Hz including a dead time of a few 100 ms.

Instrument calibration occurs through a known standard material. The sample to detector distance is obtained by two exposures with known translation of the sample towards the detector measuring the change of size of the diffraction rings and evaluation through the rule of proportion. Then, the accurate beam energy can be calibrated by evaluation of the powder pattern of the standard.

Different sample environments can be set up according to availability and needs.

At APS, we used a stand-alone hydraulic load frame based on a MTS 858 with ±15 kN maximal force. Optionally, an infrared furnace was installed for external heating of the specimen up to 1100 °C. ESRF supplies a compact, hydraulic 5 kN frame with large rotational access of 170° and operation at room temperature or alternatively, a screw driven Instron electro-thermomechanical tester (ETMT) for simulating thermo-mechanical processing. In the present configuration loads up to 3000 N can be applied while the specimen is heated resistively with a maximum power angle of 8 V and 450 A. We have reached melting temperatures of iron and titanium aluminium intermetallics which lie around 1500 °C. Cylindrical and flat sheet bone shaped specimens have been prepared by wire cutting for usage in compression and tensile testing, respectively. Typically, a test starts by mounting the sample into the load frame followed by translational transmission scans to locate the sample center in the beam. Different temperature and stress profiles can be run in both constant load and constant displacement mode. Both facilities allow to record the thermo-mechanical load parameters in their beamline control software based on SPEC [7]. A regulation script was implemented into the control software to

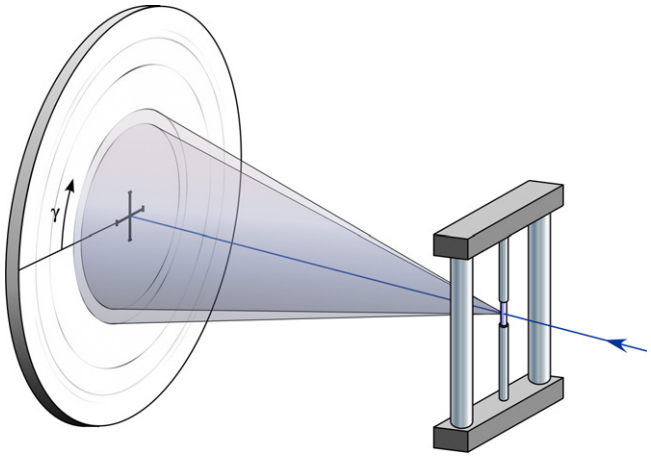

Fig. 1. Experimental setup. A fine synchrotron beam impinges from the right to the polycrystalline specimen and diffracts into Debye–Scherrer cones recorded on a 2D detector.

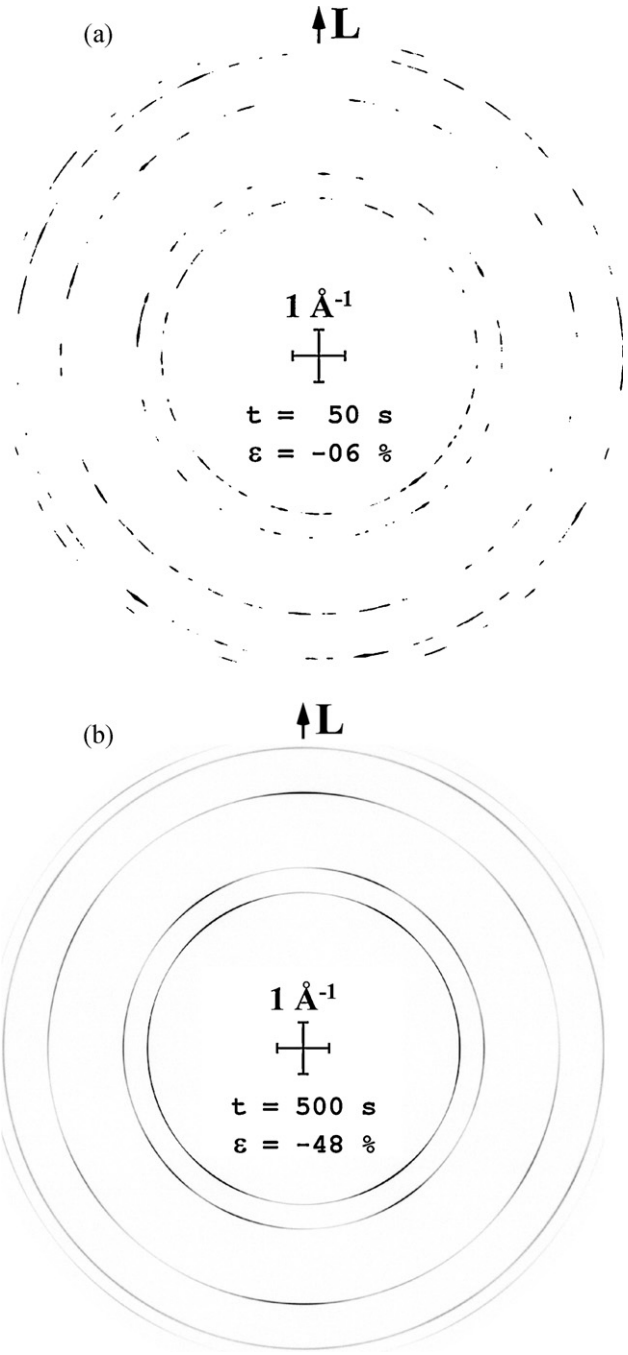

Fig. 2. Two-dimensional diffraction rings from polycrystalline copper recorded towards the beginning (a) and the end (b) of the deformation process.