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# White beam microdiffraction experiments for the determination of the local plastic behaviour of polycrystals

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**Abstract.** The overall plastic behavior of polycrystalline materials strongly depends on the microstructure and on the local rheology of individual grains. The characterization of the strain and stress heterogeneities within the specimen, which result from the intergranular mechanical interactions, is of particular interest since they largely control the microstructure evolutions such as texture development, work-hardening, damage, recrystallization, etc. The influence of microstructure on the effective behavior can be addressed by physical-based predictive models (homogenization schemes) based either on full-field or on mean-field approaches. But these models require the knowledge of the grain behavior, which in turn must be determined on the real specimen under investigation. The microextensometry technique allows the determination of the surface *total* (i.e. plastic + elastic) strain field with a micrometric spatial resolution. On the other hand, the white beam X-ray microdiffraction technique developed recently at the Advanced Light Source enables the determination of the *elastic* strain with the same spatial resolution. For polycrystalline materials with grain size of about 10 micrometers, a complete intragranular mechanical characterization can thus be performed by coupling these two techniques. The very first results obtained on plastically deformed copper and zirconium specimens are presented.

## Introduction

Polycrystalline materials are made of a compact assembly of grains of different crystallographic orientations, with a typical grain size of a few micrometers for those of industrial interest. Since the mechanical behavior of grains is generally anisotropic, strongly heterogeneous stress and strain fields develop inside polycrystals even upon macroscopic homogeneous loading. In the last decades, several mechanical models [1-3] have been developed towards understanding and predicting the overall behavior of these materials. These models aim at describing the scale transition from the grains to the polycrystal. In this framework, addressing the overall plastic response is a particularly difficult task since the collective movement of lattice defects (dislocations) leads to a highly non-linear behavior with extremely strong strain heterogeneities arising at the submicron scale. The local constitutive relation at the scale of the grain or even smaller scale is necessary for a proper scale transition, but there are two main difficulties for its determination. First, one needs measurements with a micrometric spatial resolution. Second, both the stress and the strain fields are required. Since such measurements were not possible up to now at such small scale, the local constitutive relation is classically estimated either (i) on large single crystals, but these crystals have generally a

different chemical composition than the polycrystal of interest, and it is well known that they behave differently than single grains owing to the classical Hall-Petch size effect, or (ii) by identification on the overall behavior of the polycrystal, by the use of scale transition models. But it is now admitted in the community that for a proper validation of such a method, local measurements at the grain scale are necessary [4].

This problem is particularly difficult for Zr based alloys widely used in the nuclear industry [5]. Indeed, this material exhibits a very strong plastic anisotropy which induces very high levels of stress and strain heterogeneities [6,7] as already shown by a monochromatic microdiffraction experiment [8]. Large single crystals are extremely difficult to grow. At present, the behavior of the secondary slip systems (harder slip on basal and pyramidal planes) is not clearly identified. A microextensometry technique has been developed for the analysis of the strain field at the surface of plastically deformed polycrystals [9-11]. Local strain fields with a typical resolution of  $10^{-3}$  and a spatial resolution of only a few micrometers can then be measured over areas millimetric in size, statistically representative of the polycrystalline microstructures. Classically, very large strain heterogeneities are observed, and strain is localized within bands which length is typically 3 to 10 grain size [11], depending on the considered material. Besides, the recent development of the White Beam (WB) microdiffraction technique at the beamline 7.3.3 of the Advanced Light Source (LBNL) allows completely new scanning of polycrystals [12]. In particular, a real stress map can be obtained with a micrometric spatial resolution, i.e. similar to that for microextensometry. Significant progresses are expected from the simultaneous application of these complementary techniques.

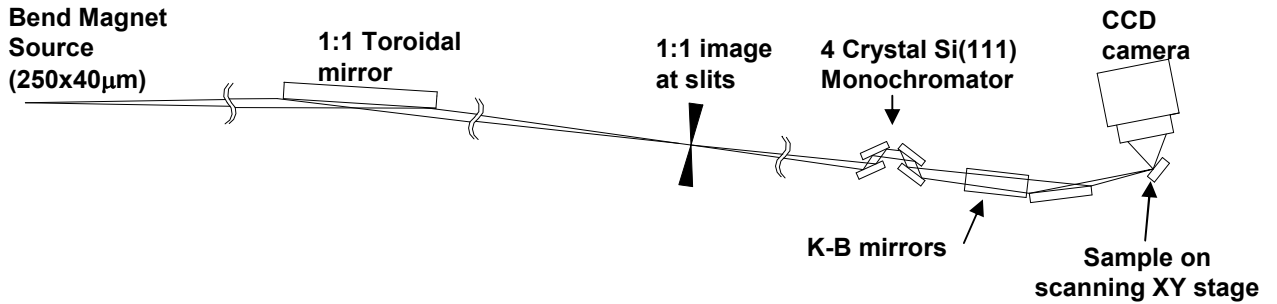
In this paper, we present the very first attempt to couple microdiffraction and microextensometry techniques. It is worth noting that analyzing the deformation of grains lying at the specimen surface under synchrotron radiation is of high interest since it provides the unique possibility to combine the analysis with laboratory measurements, mostly limited to the specimen surface. Two different materials are considered, Cu and Zr, for reasons that will be explained in the sequel. Typical evolutions of Laue pattern, that could be acquired only very recently, will be presented. The capacity of WB microdiffraction for improving our understanding of active deformation mechanisms will be highlighted.

## Microdiffraction setup

X-ray diffraction is a powerful experimental technique that is used to investigate the structural properties of materials. At Beamline 7.3.3 of the Advanced Light Source (LBNL), the high flux white X-ray beam provided by the bent magnet source, with 5-14 keV energy range, is focused down to micrometric spot size by means of elliptically bent mirrors in Kirkpatrick-Baez (KB) configuration (figure 1). A 4-bounce Si(111) monochromator can be optionally inserted between the secondary X-ray source and the KB mirrors for switching between white and monochromatic radiation while illuminating the same area of the sample. A high precision XYZ positioning stage allows scanning of the sample under the microbeam and collecting diffraction data at each step (Scanning x-ray microdiffraction). A MAR CCD detector (16 bits, 1024×1024 pixels, 25 cm diameter), positioned about 6 cm away from the specimen at 90° from the incoming beam provides collection of diffraction data from a large solid angle.

Laue pattern recorded under White Beam (WB) mode contain a large amount of information concerning the state of the specimen. Indeed, the data can be seen as a special integration of the intensity over the reciprocal space, between the two Ewald spheres corresponding to the lowest and the largest wavelength of the beam. The local crystallographic lattice orientation can be assessed from the position of the Laue spots on the detector with high precision. The local misorientation within the gage volume, as well as the distribution of lattice spacing, can also be evaluated since these distortions broaden the Laue spots in specific directions. The displacement of the Laue spots with respect to those obtained for a perfect crystal enables the evaluation of the mean strain. The

deviator of the elastic strain tensor can be estimated at once as far more than 5 spots are measured simultaneously (there are generally much more than that). The accuracy of strain determination is of the order of  $10^{-4}$ . For materials with isotropic compressibility, as considered here, all components of the deviatoric stress can thus be mapped with a micrometric spatial resolution.



**Figure 1.** Schematic layout of the Beamline 7.3.3 at the Advanced Light Source (LBNL)

For the present experiment, the focused beam at the sample reached a size of  $1.2 \times 0.8 \mu\text{m}^2$  (H×V, FWHM), with a beam convergence of about 1 mrad. According to the energy range of the incoming beam, the attenuation length is smaller than  $30 \mu\text{m}$  for Cu while it can reach  $60 \mu\text{m}$  for Zr. It is thus worth noting that the gage volume takes the shape of a thin needle going deep into the material. For Zr particularly, the attenuation length being larger than the grain size, several grains may be analyzed simultaneously, even if the beam cross section is much smaller than the grain size. Practically, this means that several Laue pattern are generally superimposed at each acquisition step. This feature has been used in [13] for performing 3-D scanning by means of a dedicated triangulation technique which allows to determine precisely the depth corresponding to each Laue spot. In the present work, only the data coming from the surface grains are analyzed. Laue spots from these grains can be identified owing to their larger intensity. Besides, two independent cross-checks can be performed simultaneously to verify that only the surface grain is considered. First, the local orientation of the crystallographic lattice given by the Laue setup can be compared to that obtained by EBSD. Second, an indication concerning the depth at which the Laue pattern has been generated is provided by the data analysis software XMAS [14] thanks to a triangulation technique and thus grains below the sample surface can be disregarded.

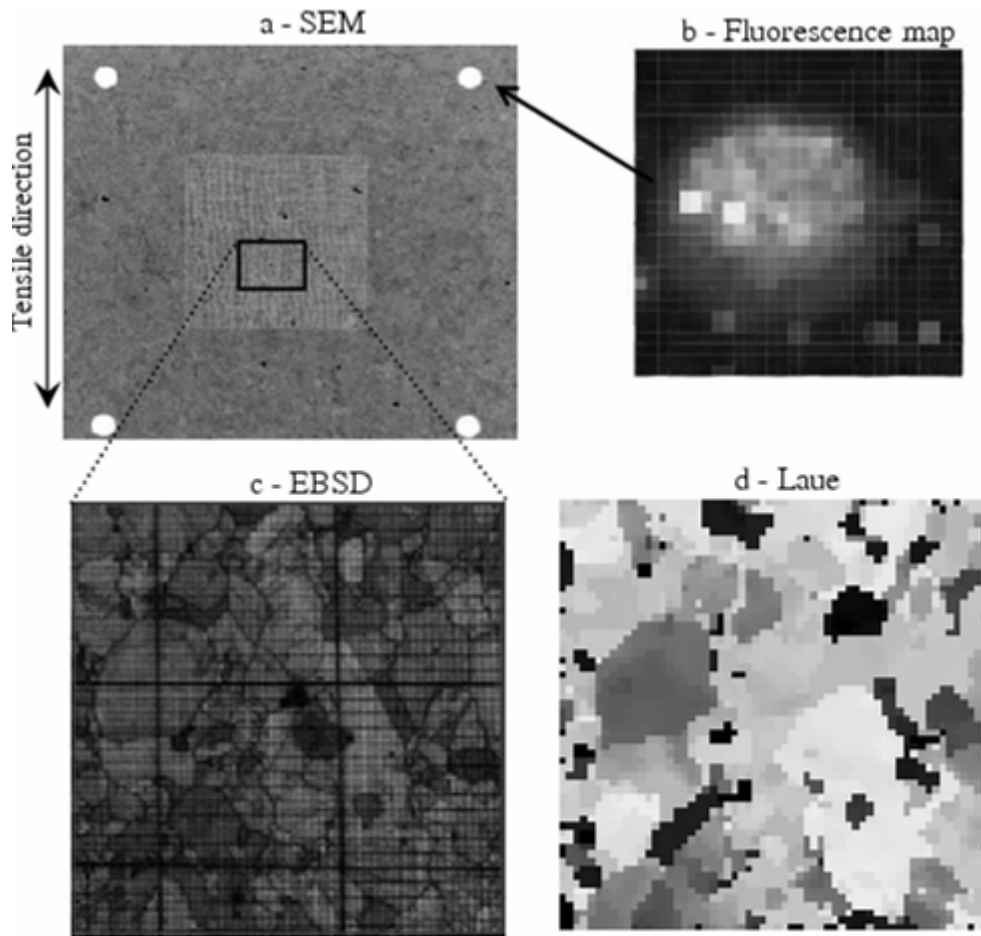
## Specimens

Specimens of ETP copper (Cu) and zirconium alloy (Zr702) were investigated. Both materials were initially recrystallized with a grain size of  $50 \mu\text{m}$  for Cu and  $10 \mu\text{m}$  for Zr. Zirconium grains are equiaxed while elongated recrystallization twins are mostly observed for Cu. The specimens have been tensile deformed for overall plastic axial strain ranging between 2% and 9%. On each specimen, a thin gold grid [9] has been deposited prior the mechanical tests (figure 2a). It enables the evaluation of the surface strain field in the Region of Interest (ROI), by means of image correlation techniques applied to high definition digital SEM images of the sample recorded before and after deformation [10]. The step of these grids,  $5 \mu\text{m}$  for Cu and  $1.5 \mu\text{m}$  for Zr, is smaller than the grain size to make it possible the estimation of *intragranular* heterogeneities of total strain. Besides, 4 gold spots ( $1.2 \mu\text{m}$  thickness,  $100 \mu\text{m}$  diameters) have been deposited around the ROI by ion beam sputtering. They are easily observed by a scanning fluorescence map under the focused WB (figure 2b) and thus allow determining precisely the position of the X-ray beam on the specimen surface. From this, a small region within the gold grid can be selected and scanned by microdiffraction. The accurate combination of SEM total strain measurements and microdiffraction elastic strains maps is made possible by an appropriate choice of reference marks in or near the ROI

that can be located precisely with both mapping techniques. Local grain orientations obtained from both EBSD and microdiffraction are in addition helpful for this combination.

## Results

It can be verify that a very good agreement is obtained with the orientation map obtained by EBSD (figure 2c), indicating that the positioning technique using the fluorescence of the “thick” gold spots is well adapted for our purpose, and that surface grains can be really extracted from the rough Laue pattern. An absolute spatial accuracy of about 50  $\mu\text{m}$  could be reached. The next step will be to compare these data to the deformation map obtained by microextensometry. This will be performed soon.

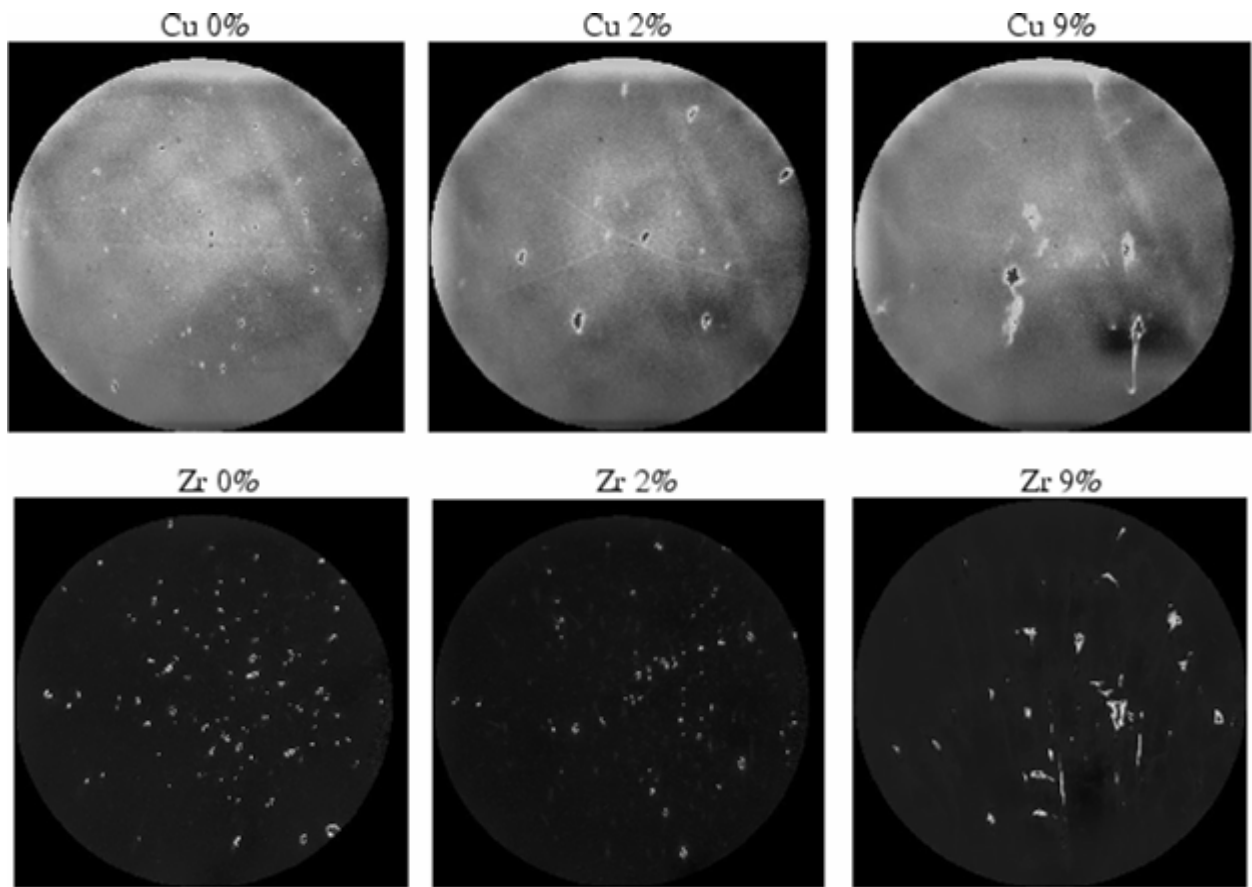


**Figure 2.** (a) SEM image of a Cu specimen with the  $1 \times 1 \text{ mm}^2$  gold grid and four thick gold spots. (b) Fluorescence map of a gold spot obtained with the focused WB. (c) EBSD image of the ROI superimposed with the gold grid. (d) Orientation map of the same ROI obtained by scanning WB microdiffraction.

This very first experiment was oriented towards a comparison between the mechanical responses of Cu and Zr. Copper polycrystals have been often investigated in the literature, owing to the relative simplicity of this material. Cu crystals exhibit a strong elastic anisotropy but an isotropic compressibility owing to its cubic structure. The slip systems for dislocation glide are well identified as for most FCC materials, and several models for work hardening have been identified on the basis of mechanical tests on single crystals [15]. The presence of the 12 equivalent  $\{111\}\langle 110 \rangle$  slip systems makes it possible for a grain to accommodate any plastic deformation. On the other hand, Zr crystals exhibit an almost isotropic elastic behaviour. However, owing to its HCP

structure, only few easy glide slip systems are available, and the  $\langle a \rangle$  prismatic system is by far the most active deformation mechanism [16]. It follows that axial deformation of Zr crystals along the  $c$  axis is rather hard as compared to deformation along the  $a$  axis. Consequently, a very high level of intra- and inter-granular residual stress is expected to build up during plastic deformations.

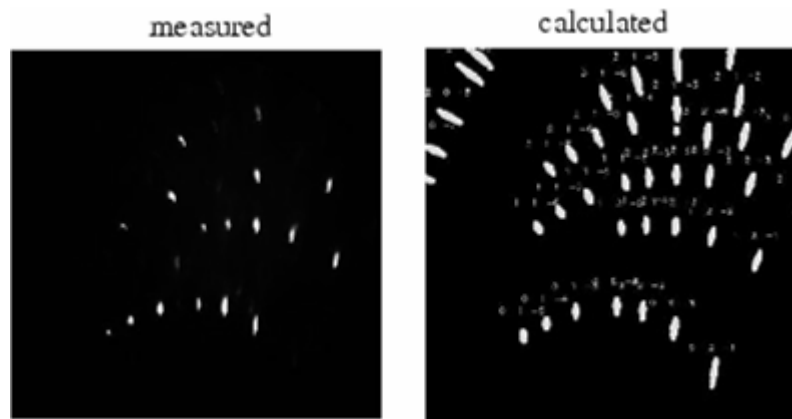
At the initial stage, Laue spots have almost Dirac shape, indicating a small intragranular misorientation in the recrystallized grains for both Cu and Zr (figure 3). With increasing plastic strain, a very high distortion of Laue diagram can be observed. Laue spots become significantly elongated, indicating misorientations often larger than 10 deg within the micrometric gage volume. The corresponding energy distribution within each Laue spot, as analysed by a monochromatic scan with continuously varying beam energy, has been found to have a range of more than 1 keV. The distortion of the pattern is such that the evaluation of local deviatoric stresses should become a challenging task for plastic strain larger than 10-15%. The different plastic response of Cu and Zr polycrystals is also clearly highlighted by WB microdiffraction. In average, Laue diagram for Zr have been found to be much more distorted than those for Cu, for similar overall plastic strain. This has to be attributed to the strong anisotropy of plastic behaviour of Zr crystals. Such features had already been observed by a Monochromatic Beam (MB) microdiffraction technique [8] however with a poorer spatial resolution than the WB technique used here. An intragranular stress deviation of about 100 MPa was observed in Zr alloys after 15% overall plastic strain, i.e. a stress level of the same order than the macroscopic yield stress.



**Figure 3.** Typical Laue pattern observed for Cu and Zr polycrystals, at 0% (recrystallized state), 2%, and 9% overall axial strain. Note the Kossel lines observed on Cu diagrams.

Finally, it is clearly observed that the distortion of Laue diagram is not random. Indeed, the local misorientation within the gage volume has to be attributed to the presence of geometrically

necessary dislocations. Since each dislocation type produces a different lattice rotation, the shape of Laue pattern can be used to identify the type and the density of dislocations within the gage volume. Figure 4 shows a Laue diagram measured on the Zr specimen after 15% tensile strain. The modelling of the corresponding pattern by the technique described in [17], when assuming that only  $\langle a \rangle$  prismatic dislocations are present, lies in perfect agreement with the measurement. This is fully consistent with deformation mechanisms since  $\langle a \rangle$  prismatic slip is the dominant slip in Zr alloys.



**Figure 4.** Laue diagram measured on the deformed Zr specimen (left) and simulated assuming  $\langle a \rangle$  prismatic dislocations.

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