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Experiment Report Form

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Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

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Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Strain distributions around an individual shear band analysed by nanobeam X-ray diffraction	**Experiment number**: HC-2030
Beamline: ID11	**Date of experiment**: from: 03.02.2016 to: 09.02.2016	**Date of report**: 01.03.2016
Shifts: 18	**Local contact(s)**: Diadkin Vadim	*Received at ESRF:*

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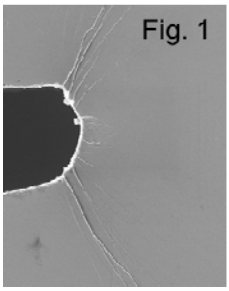
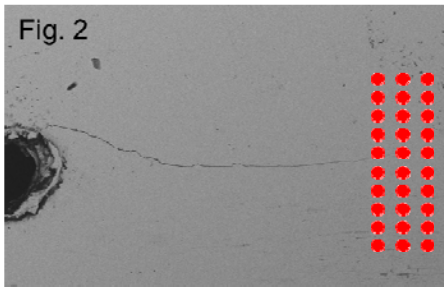
Report:

For bulk metallic glasses (BMGs), the distinction between elastic and plastic strain is a very challenging task because plastic deformation in these materials occur through the formation of highly localized shear bands with thickness of about 10-100 nm [1]. The size of conventional high-energy X-ray beams (5-50 μm) does not permit to make such a distinction as structural information comes from both shear bands and surrounding elastic matrix. The situation is further complicated by the fact that plastic deformation in BMGs under conventional loading conditions, such as compression, is highly heterogeneous, with unpredictable location of the shear bands. Therefore, the decoupling of the elastic and plastic contributions to the strain requires the use of an X-ray beam of size comparable with the shear bands thickness combined with the exact knowledge about the position of *any* shear band forming and propagating in the sample. The aim of this experiment was to overcome this limitation and investigate for the first time the atomic rearrangements occurring within and around a nascent shear band during the early stages of its formation.

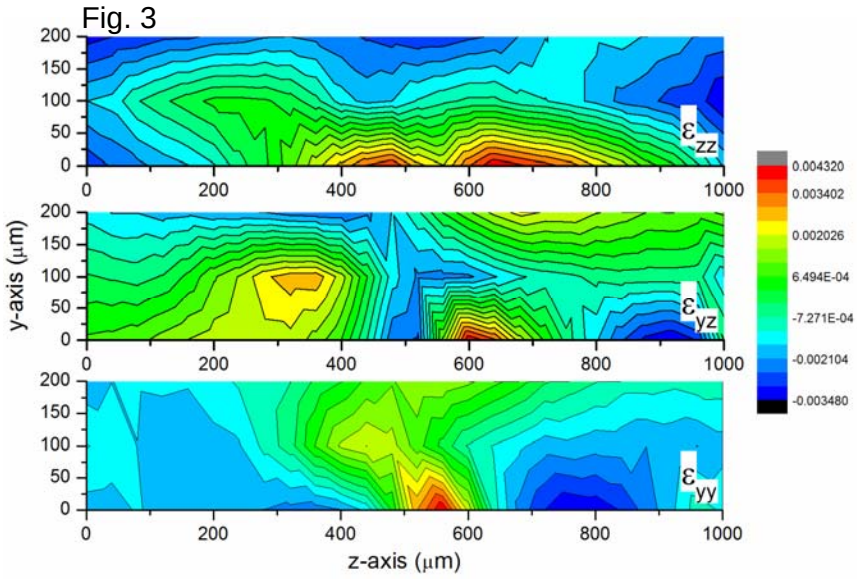
To achieve this aim, in our experiment we used the smallest high-energy beam size existing to date (0.15×0.15 μm^2), which is only available at the beam line ID11. The precise control over the location of the shear band formation was obtained by deforming notched-only BMG specimens, where shear bands are formed at two characteristic positions (see Fig. 1), and fatigue pre-cracked samples (see Fig. 2), which gives enhanced control over the shear band morphology. In order to increase the chances of getting the shear band parallel to the beam, sample thickness was reduced to about 100 μm. Mechanical deformation of the BMG samples was carried out at room temperature under tensile loading using the tensile rig





available at ID11 and operating at a strain rate of 1×10^{-4}. The samples were firstly investigated at zero load, to have the initial value for the strain calculation, and subsequently in-situ during loading at 50, 100, 120 and 150 N. The structure after unloading was studied as well in order to extract information on the structural changes without the effect of the elastic contribution. For each loading step, diffraction patterns were collected at every 0.5 μm along the loading z-direction in a sequence of three parallel lines with spacing of 100 μm (see red dots in Fig. 2). This way, the variation of the strain at different distances from the notch or pre-crack was investigated carefully keeping the measurement within a reasonable time (20-30 h), given the long acquisition time (15-30 s per pattern) needed for acquiring good intensity for the present combination of small beam size and reduced samples thickness. The first line was collected at about 100 μm from the notch or pre-crack.

The two-dimensional patterns were integrated in 10° azimuthal slices between 0 and 360° using the Fit2D program [2] to give the XRD intensity distributions $I(Q,\eta_i)$ as a function of the scattering vector $Q = 4\pi\sin\theta/\lambda$ (where λ is the wavelength and θ is half of the scattering angle) for the azimuthal angle η_j (j = 10…360°). In order to have a preliminary evaluation of the results, the strain ε induced ahead of the notch/pre-crack during loading was determined in reciprocal space through the shift of the main diffraction peak (Q_1) as $\varepsilon = (Q_0 - Q_{load})/Q_{load}$, where Q_{load} is the peak position of the mechanically-loaded material and Q_0 of the initial zero-load condition. The three components of the strain tensor (tangential ε_{yy}, axial ε_{zz} and in-plane shear ε_{yz}) for each point scanned on the y-z plane were determined according to the method described in Poulsen *et al.* [3] by fitting the angular variation of the peak shifts.



As a typical example of the data obtained in reciprocal space, Fig. 3 shows the strain maps of the ε_{yy}, ε_{yz} and ε_{zz} components of the strain tensor evaluated from the variation of Q_1. The data refer to a pre-cracked sample loaded to 120 N. The pre-crack is parallel to the y-axis and its tip is located at about (-100, 550) on the y-z plane.

The results reveal that plastic deformation ahead of the pre-crack creates a spatially heterogeneous atomic arrangement, consisting of compressive and tensile strain fields. The shear strain ε_{yz} is rather large; indicating that, ahead of the pre-crack, a significant fraction of the atomic displacement is not accommodated along the y and z directions. This is associated to the plastic deformation localized within the shear bands, which form an angle of about 45° with the loading direction. The shear strain changes sign along the z-axis, in accordance with

the atomistic simulation of shear bands in Cu-Zr BMGs [4] showing a similar behaviour on the opposite sides of the bands. Furthermore, the shape of the shear strain distribution is strikingly similar to the average particle displacements across an elementary shear band simulated by Tanguy *et al.* [5], who observed an inversion from negative to positive values in the displacement of the particles across the band. The comparison of the simulated data with the present shear strain profile reveals that, although the length scales of the datasets are necessarily different, the experimental results are in excellent agreement with the simulated values.

For amorphous materials there is no one-to-one correspondence between the position of a scattering maximum and a particular atomic spacing in the material, as in crystalline materials. Instead, information about the real-space atomic structure is distributed throughout reciprocal space. Consequently, the results reported here are necessarily preliminary and work is ongoing to obtain a complete picture of the strain distribution in real space, as we have already done for ex-situ measurements of plastically-deformed BMGs [6].

In conclusion, the unique experimental setup of this experiment consisting of a small high-energy beam size combined with the precise control over the location of the shear band formation allowed us to investigate for the first time the atomic rearrangements occurring within and around a nascent shear band during the early stages of its formation. The results in reciprocal space have already revealed significant structural features, which are in good agreement with previous simulations. The real space results are expected to lead to a significant leap forward in the understanding of the intrinsic properties of metallic glasses and to generate a comprehensive picture of the glassy structure including local changes at different length scales caused by shear banding.

References

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