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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

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

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

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:** Spatially-resolved local atomic fluctuations in bulk metallic glasses	**Experiment number**: HC 2319
Beamline: ID 11	**Date of experiment**: from: 22 June 2016 to: 28 June 2016	**Date of report**: October 2016
Shifts: 18	**Local contact(s)**: Andrea BERNASCONI	*Received at ESRF:*

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Report:

The proposed experiment aimed to resolve, for the first time, the local atomic fluctuation in bulk metallic glasses (BMGs). The key focus is the influence of atomic contributions on the intrinsic plasticity, hence detailed analysis of the corresponding structural variations in the medium and short range order was planned. Despite the fact that monolithic BMGs are intrinsically brittle, some of them undergoes large compressive plastic deformation and this is favored by local fluctuation of the atomic arrangements. High deformations of BMGs can be attained only if numerous shear bands are formed. The such-named shear transformation zones (STZs) serve as nucleation sites for the shear bands. The ability of a region to undergo a shear transformation depends on the local microstructure. The STZs occur preferentially in soft regions in BMGs and evolve into shear bands upon loading. Further, the shear-band propagation is impeded by the hard regions. This impedance alters the propagating directions and assists shear-band multiplication, postponing the catastrophic failure and enhancing the ductility. Therefore, to detect such local fluctuations and determine their nature is the crucial aspect to understand and to control the plastic deformability of BMGs.

The collected X-ray diffraction (XRD) data was taken in computer tomography (CT) configuration and allow the calculation of pair distribution function (PDF). The XRD-PDF-CT approach was used for the first time on the BMG samples, with the purpose to map their structure at nanoscale.

The experiment was designed for the following glassy alloy samples:

$Zr_{64.13}Cu_{15.75}Ni_{10.12}Al_{10}$ [1]

$Pt_{57.5}Cu_{14.7}Ni_{5.3}P_{22.5}$ [2]

These BMGs show a relative large plastic deformation, which it is supposed to arise from a particular structure, consisting of an alternation of hard and soft zones, as shown in Fig. 1 [1]. However, the discussion

remains at the level of speculation and no real differences were detected in the TEM because the observations are strongly affected by the thinning rate of the sample, as well as the beam intensity, which do not allow for accurate measurements.

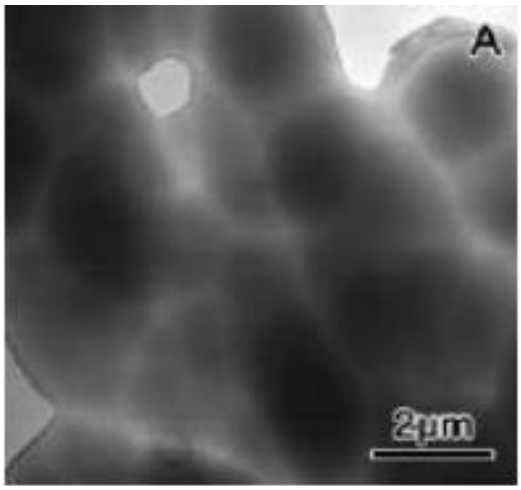

Fig. 1 Dark regions surrounded by bright regions (hard and soft regions, respectively) [1].

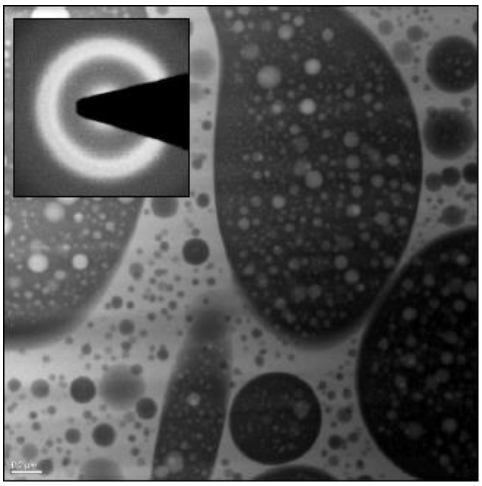

Fig. 2 Amorphous phase-separated $Gd_{27.5}Hf_{27.5}Co_{25}Al_{20}$ sample. White: Hf-rich phase, black-Gd-rich phase

A main problem of the XRD-PDF-CT is that the diffraction data are prone to several measurement artefacts, which should be carefully analyzed and ruled-out. Therefore, during the allocated time, we performed several investigation, as detailed in the following.

- Measurement algorithm: a line scan consisting of 51 equally distanced (i.e. at every 500 nm) points for every $\Delta\omega$ rotation angle (CT approach). The rotation angle was set for $\Delta\omega = 3°$, so a total of 61 projections were taken.
- The samples were placed in the rotation center of the hexapod sample holder, which was fixed on the piezoactivated table.
- Defined a procedure to check if after every line scan and $\Delta\omega$ rotation the samples comes back in the rotation center.
- In order to prove the reliability of the method, a phase separated amorphous $Gd_{27.5}Hf_{27.5}Co_{25}Al_{20}$ sample, consisting of two amorphous phases (Hf rich and Gd rich, respectively, see Fig. 2) was used as phantom sample.
- In order to check the reliability of the method, every sample was line scanned as well for a $\Delta\omega = 10°$, in this way 19 projections were obtained.
- The diffraction patterns were obtained upon radial and azimuthal integration of the 2D images recorded by a Frelon 4M pixels detector. The patterns must be corrected for the high-Q camera nonlinearity (the absorption of the X-ray beam in the phosphor screen may be less than 100% ("thin phosphor regime"), with a consequent detector response that depends on the incident angle and the effective thickness of the phosphor) as described in [3]. Further, the corrected patterns are analyzed using the PDF-getX3 software.

[1] Y.H. Liu, G. Wang, R.J. Wang, D.Q. Zhao, M.X. Pan, W.H.Wang, Science 315 (2007).
[2] J. Schroers, W.L. Johnson, Phys. Rev. Lett. 93 (2004).
[3] A. Bernasconi, J. Wright, N. Harker, Powder Diffr. 30 (2014).