



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://www.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.



	Experiment title: Investigation of anisotropy induced by post-processive mechanical treatments in a Zr-based bulk metallic glass (BMG)	Experiment number: HC-1178
Beamline: ID11	Date of experiment: from: 13.06.2014 8:00 to: 17.06.2014 8:00	Date of report: 28.02.2015
Shifts: 12	Local contact(s): Jonathan Wright (wright@esrf.fr)	<i>Received at ESRF:</i>
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Report:

The properties of bulk metallic glasses (BMGs), such as high strength and a large elastic limit, are closely related to their structure [1-3]. Within this project we have investigated changes in the structure of amorphous alloys induced by the application of different kinds of elastic as well as plastic pre-deformation. In doing so, we have compare three different glass-forming alloys with varying tendency to vitrify to see whether the stability of the glass reflects in the way the structure changes during pre-deformation.

The three different compositions with decreasing glass-forming ability (GFA) used for the experiments are: $\text{Cu}_{45}\text{Zr}_{45}\text{Al}_5\text{Ag}_5$, $\text{Cu}_{47.5}\text{Zr}_{47.5}\text{Al}_5$ and $\text{Cu}_{47.5}\text{Zr}_{47.3}\text{Al}_5\text{Sc}_{0.2}$. The addition of Ag to Cu-Zr-Al system is known to enhance the stability of the liquid phase whereas Sc tends to stabilise the B2 CuZr phase and this deteriorates the GFA. The following deformation treatments were applied to these rapidly quenched alloys with various geometries: (i) static compression (72 h at 85% of the yield strength) (ii) dynamic compression (10^2 , 10^3 , 10^4 and 10^5 cycles, 10 Hz, sinusoidal, 0 MPa to 85% of the yield strength) (iii) cold-rolling (40%, 80% and 250%) and 4. High pressure torsion (HPT) (4 GPa and 8 GPa, 80 turns). The rather extensive amount of data recorded at the ESRF is still being analysed in depth and will be prepared for publication.

Within this report we merely concentrate on the results obtained for the HPT samples. There are publications on the analysis of the structure of metallic glasses, which were subjected to severe plastic deformation by means of HPT, using high-energy X-rays [4-5]. These authors performed linescans across the sample diameters and found that the position of the first scattering maximum, Q1, correlates with the shear strain, which is a function of the distance to the sample centre. The shear strain can be described as $\gamma(r) = 2\pi Nr/l$, with r being the radius of the disk, N being the number of turns and l being the sample thickness. We have used a different approach and placed a rectangular grid with lateral and vertical spacings of 0.125 mm across one half of each HPT sample. In total about 2000 diffraction patterns were recorded with an exposure time of 5 s. These measurements allow a much more detailed view on the behaviour of the material. Aspects like homogeneity of the structure and deformation as well as possible artifacts caused by a slight misalignment can be discussed. Figure 1 shows the positions of the first broad maximum (Q1) in the intensity curve (fitted with a pseudo-Voigt function with linear background) over the scanned area. The upper two images display the HPT samples of $\text{Cu}_{45}\text{Zr}_{45}\text{Al}_5\text{Ag}_5$ deformed under pressures of 4 GPa and 8 GPa (80 turns), respectively. The lower two figures depict the $\text{Cu}_{47.5}\text{Zr}_{47.5}\text{Al}_5$ HPT samples subjected to pressures of 4 GPa and 8 GPa. The colour scaling is identical for all figures.

The radial symmetry of all the samples is obvious and the central regions show a minimum in Q1 for all the samples. Towards the sample edges the value of Q1 increases and reaches a maximum at intermediate radii and then decreases again. This is due to the fact that the thickness of the samples near the edges is slightly reduced during the HPT process. This lowers the friction with the plunger and thus the degree of deformation. Changes in Q1 are commonly attributed to structural changes in the medium-range order (MRO) arising from elastic strains [6]. The shifts of Q1 in the present case can be interpreted as compressive strains residing from plastic deformation.

Additional aspects such as calculating the local strain based on a comparison with the peak position of the first maximum in the as-cast state [6], analysis of the behaviour of additional scattering maxima other than the first, analysis of the behaviour in real space or the detailed analysis of the peaks widths will be also addressed in the future using the present data. Nevertheless, two findings can be immediately discussed from the present results: The change of the structure depends on the composition of the glass and on the pressure applied during the HPT treatment. If the pressure is 4 GPa, the first scattering maximum of $\text{Cu}_{45}\text{Zr}_{45}\text{Al}_5\text{Ag}_5$ varies much more strongly than that of $\text{Cu}_{47.5}\text{Zr}_{47.5}\text{Al}_5$. It seems that the better glass former retains more of the strain introduced during deformation. Additionally, crystalline regions can be detected, which might have formed during the severe plastic deformation. These cannot be found in the more stable $\text{Cu}_{45}\text{Zr}_{45}\text{Al}_5\text{Ag}_5$ glass. All as-cast plates were analysed by reflection XRD and DSC prior to the HPT experiments and the results indicated the samples to be fully amorphous. At the lower pressure, ordering processes might have occurred in the glass, which tends to crystallise more easily.

The first diffraction maximum of both alloys decreases when the samples are deformed at 8 GPa and simultaneously, the structure of the material seems to become more homogeneous. Under appropriate conditions HPT can either amorphise crystalline material or cause ordering in glassy systems. This threshold pressure seems to be between 4 GPa and 8 GPa. The processing thus has a pronounced influence on the structure of the two metallic glasses and it can be traced back by the present approach.

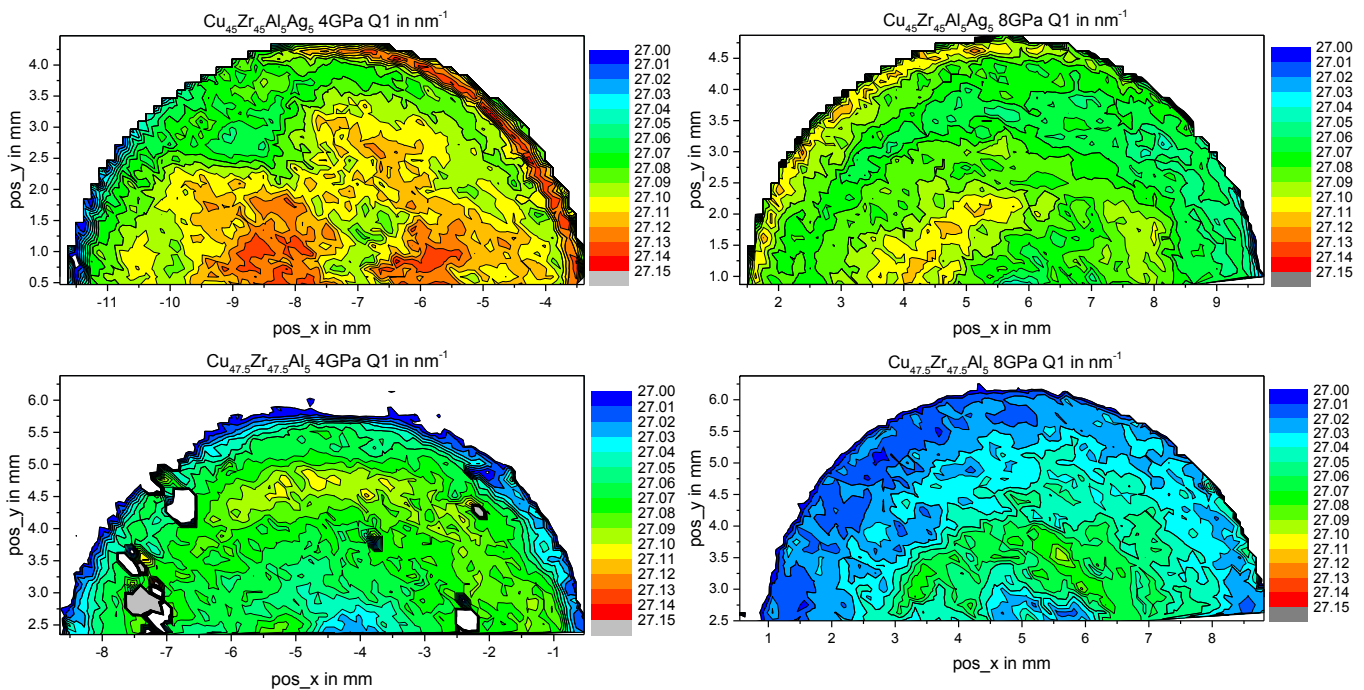


Figure 1: Position of the first scattering maximum (reciprocal space) in one half of a glassy $\text{Cu}_{45}\text{Zr}_{45}\text{Al}_5\text{Ag}_5$ and $\text{Cu}_{47.5}\text{Zr}_{47.5}\text{Al}_5$ disk deformed by HPT (80 turns and at pressures of 4 GPa and 8 GPa). The white and grey spots indicate partially crystalline regions.

References

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3. Schuh, C. A. et al., *Acta Mater.* **2007**, 55 (12), 4067-4109.
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5. Révész, Á. et al., *Appl. Phys. Lett.* **2008**, 92 (1), 011910.
6. Poulsen, H. F. et al., *Nature Mater.* **2005**, 4 (1), 33-36.