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:** In-situ strain analysis of heterogeneous bulk metallic glasses: effect of interfaces	**Experiment number**: HC-2934
Beamline: ID11	**Date of experiment**: from: 05.05.2017 to: 09.05.2017	**Date of report**: 31.08.2017
Shifts: 12	**Local contact(s)**: Marta Majkut	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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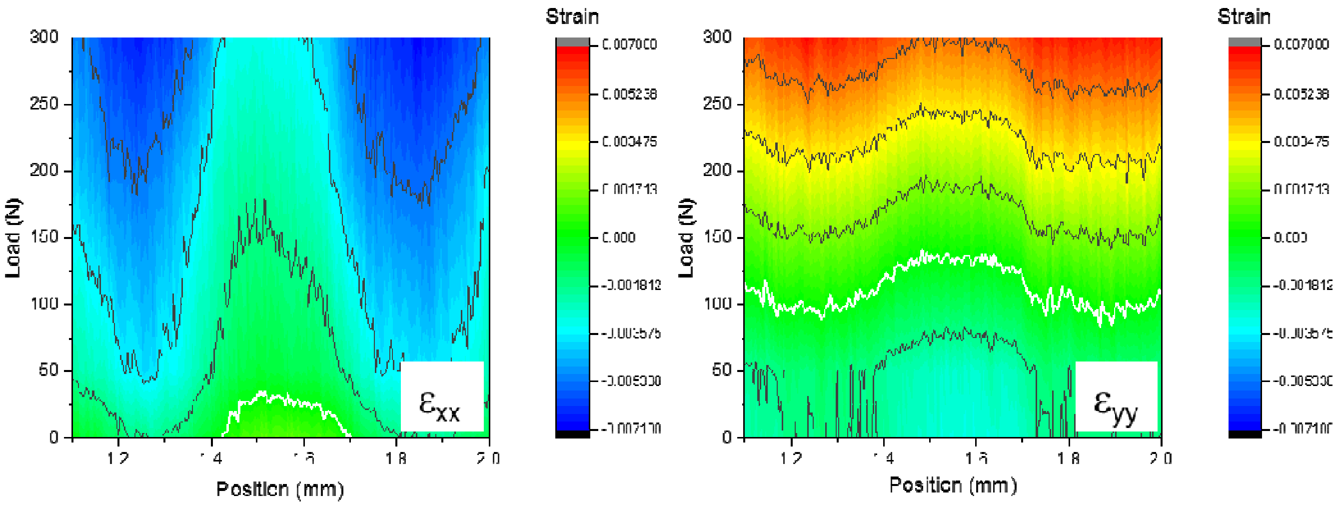
Report:

The aim of our experiments was to characterize the structure of heterogeneous bulk metallic glasses (BMGs) in order to understand their enhanced room-temperature tensile ductility compared to the corresponding homogeneous counterparts. To achieve this aim, we studied *in-situ* the strain fields created within and at the interfaces between the structurally heterogeneous regions during mechanical loading of an imprinted $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ BMG. Imprinting creates structural heterogeneities with controlled size and morphology, a prerequisite for accurately characterize the elasto-plastic behavior of heterogeneous BMGs.

The heterogeneous areas in the imprinted glass are expected to exhibit different elastic properties, which, as a result of the elastic mismatch arising at the interfaces between elastically-different materials (i.e. the heterogeneities), may generate a macroscopic non-uniform stress distribution during mechanical loading, triggering the formation of shear bands and improving the ductility. To clarify this aspect, we synthesized samples with different imprinting angles α = 0, 45 and 90° and analyzed the strain across the samples at increasing loads: 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, 200 and 300 N. An as-cast specimen was also investigated to compare the results with the parent homogeneous material. Mechanical deformation of the 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 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 3 µm along the transverse direction, covering a line of about 2 mm. In the present experiment, we used a beam size of 2×3 µm^2, small enough to extract structural information exclusively from the different heterogeneous areas, which have typical length scale of 200-300 µm, and more importantly from the interface between these regions.

The two-dimensional patterns were integrated in 10° azimuthal slices between 0 and 360° using the Fit2D program to give the XRD intensity distributions $I(Q,\eta_i)$ as a function of the scattering vector Q for the azimuthal angle η_j (j = 10…360°). In order to have a preliminary evaluation of the results, the strain ε induced in the samples 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 were determined according to the method described in Poulsen *et al.* [1] by fitting the angular variation of the peak shifts.

As a typical example of the data obtained in reciprocal space, the figure below shows the strain maps of the ε_{xx} and ε_{yy} components of the strain tensor as a function of the applied load evaluated from the variation of Q_1. The data refer to an imprinted specimen with α = 90°.



The results reveal that the strain is heterogeneously distributed in the different regions. The strain distribution becomes progressively more heterogeneous with increasing the load, with significant variation at the interfaces. The elastic constants also change dramatically: Young´s modulus and Poisson´s ratio increase from E = 92 GPa and ν = 0.30 for the as-cast material to E = 115 GPa and ν = 0.35 for the heterogeneous imprinted glass.

These results are necessarily preliminary and work is ongoing to obtain a complete picture of the strain distribution in real space along with the elastic constants for the different atomic shells, which we expect 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 effect of structural heterogeneities on the mechanical behavior of BMGs.

All in all, we consider these measurements to have been a large success and we have, to a great degree, accomplished our goals outlined in the proposal. No experimental difficulties were encountered and the scientific support from the local contact was outstanding.

[1] H.F. Poulsen *et al.*, *Nature Mater.* 4, 33 (2005).