ESRF

EUROPEAN SYNCHROTRON RADIATION FACILITY

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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

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for <u>each project</u> or series of measurements.
- type your report in English.
- include the experiment number 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:** Study of Orientational Order in Bulk Metallic Glasses	**Experiment number**: HC-3723
Beamline:	**Date of experiment**: from: 21 June 2018 to: 26 June 2018	**Date of report**:
Shifts:	**Local contact(s)**: Jussi-Petteri Suuronen	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Peihao Sun, SLAC National Accelerator Laboratory

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Report:

We received great support during the beamtime and the nano-focusing was realized with a beam size of <53nm×62nm at the focus. However, the original goal of the experiment was not achieved because of several difficulties which arose during the beam time.

Firstly, the K-B focusing mirrors had mechanical problems which limited the X-ray energy to 18.5 keV and above. This is above the Zr K-edge (18.0 keV) and makes it impractical to measure our main sample—Zr-based bulk metallic glasses. Therefore, we resorted to the backup sample—a PdCuSi metallic glass.

Secondly, there appeared to be a large background from air scattering, partly due to the fact that >18 keV X-rays could easily penetrate the plastic sample mount. We tried to minimize this background using lead tape. This reduced the background significantly, but some still remained. Because this background changed with sample position, it significantly complicated the data analysis.

Thirdly, we found significant background from the fiber optics of the FReLoN detector as well as image distortions from the CCD camera of the detector. Because our study focuses on subtle effects in the diffraction images corresponding to intensity fluctuations on the order of a few percent or lower, the detector background and distortions made it impossible to reliably extract the desired information. Neither we nor the beamline staff were aware of these problems before the beamtime, because the detector had normally been used for simple XRD characterization with nano-focusing which was not sensitive to these detector issues. Since the FReLoN camera was the only 2D detector available for the proposed experiment, the original goal to extract orientational order could not be achieved.

Nonetheless, we discovered some intriguing orientational properties of the sample. Shown in Fig. 1 are four diffraction patterns corresponding to different positions on the same sample, each divided by the average over the entire scan (7749 patterns). Large anisotropies around the azimuthal angle can be clearly observed in the diffraction ring, and the exact pattern varies with the position on the sample. We have corrected for some of the background and distortion mentioned above, and the remaining effects should be well below the ±10% anisotropy level observed in Fig. 1, so we believe these effects originate from the sample. Notably, no change is observed in the position of the structure factor maximum as a function of the azimuthal angle, so the anisotropy cannot be explained by strain effects. Thus, the results suggest the existence of structural ordering beyond the pair distribution function.

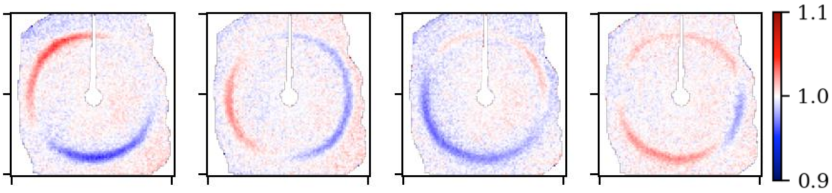

Figure 1: Anisotropy in vapor-deposited PdCuSi glass measured at ID16B. Each image is divided by the average of the entire scan (7749 images, each at a different sample position). Anisotropies on the ±10% level can be seen around the first diffraction ring.

Such high levels of anisotropy were quite unexpected. Because the sample was ~5μm thick, the illuminated volume for each pattern was on the order of 50nm × 50nm × 5μm. If there is only short-range (< 2nm) structural ordering in the metallic glass as commonly believed, there will be approximately 1.6×10^6 correlated volumes that are illuminated, which means the anisotropy (arising from number fluctuations) should be on the order of $(1.6\times10^6)^{-1/2} = 8\times10^{-4}$, much less than the 10% we observe. Therefore, there must be medium- to long-range structural ordering present in the amorphous sample, presenting a challenge to generally accepted theories on the structure of glasses.

However, because of the aforementioned limit on the X-ray energy, we were only able to examine this particular sample, so we could not determine whether these effects were sample-specific or universal among these glasses. Therefore, another experiment is necessary to evaluate the universality of the phenomenon described above and its scientific value.