ESRF	**Experiment title:** Probing nano-scale heterogeneities in metallic glasses	**Experiment number**: HC-4889
Beamline: ID16A	**Date of experiment**: from: 07/04/2022 to: 11/04/2022	**Date of report**:
Shifts: 12	**Local contact(s)**: Federico Monaco (federico.monaco@cea.fr)	*Received at ESRF:*

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Report:

The aim of the experiment is to investigate whether sub-micrometer density fluctuations exist in metallic glasses. This is motivated by the results reported in Ref. [1] where, using nano-tomography, the authors observe density fluctuations in various metallic glasses. The length scale of the fluctuations range from around 200 nm to more than 1 μm [1]. Our proposal aims to test whether these phenomena can be found in metallic glasses with other compositions, and if so, whether these fluctuations depend on how the samples are prepared.

During this beam time, we have performed nano-holotomography on different metallic glass samples at ID16A. The high stability and high resolution (down to 10 nm voxel size) are ideal for the purpose of our study. While analysis is still underway, we have obtained some preliminary results that may point to interesting behaviors in the samples.

In particular, we show in Figure 1 the cross-sections of two $Pd_{77.5}Cu_6Si_{16.5}$ metallic glass samples, one made with melt-spinning, and the other with vapor-deposition at high substrate temperatures. It can be seen that, while the melt-spun sample indeed appears to contain density fluctuations, the vapor-deposited sample looks rather homogeneous. This indicates that, indeed, the degree of density fluctuations depends on the sample preparation method. Moreover, it has previously been observed that metallic glasses that are vapor-deposited at elevated substrate temperatures and controlled rates exhibit higher mechanical homogeneity [2]. Thus, our results may provide a structural explanation behind this observation.

However, further work is needed to exclude the possibility that our observations are artifacts arising from the measurement and/or the analysis. For example, the images shown in Figure 1 contain fringes which are particularly clear close to the surface, and they appear to be influenced by the shape and position of the samples. We are working to minimize these artifacts and investigate whether they may influence the results.

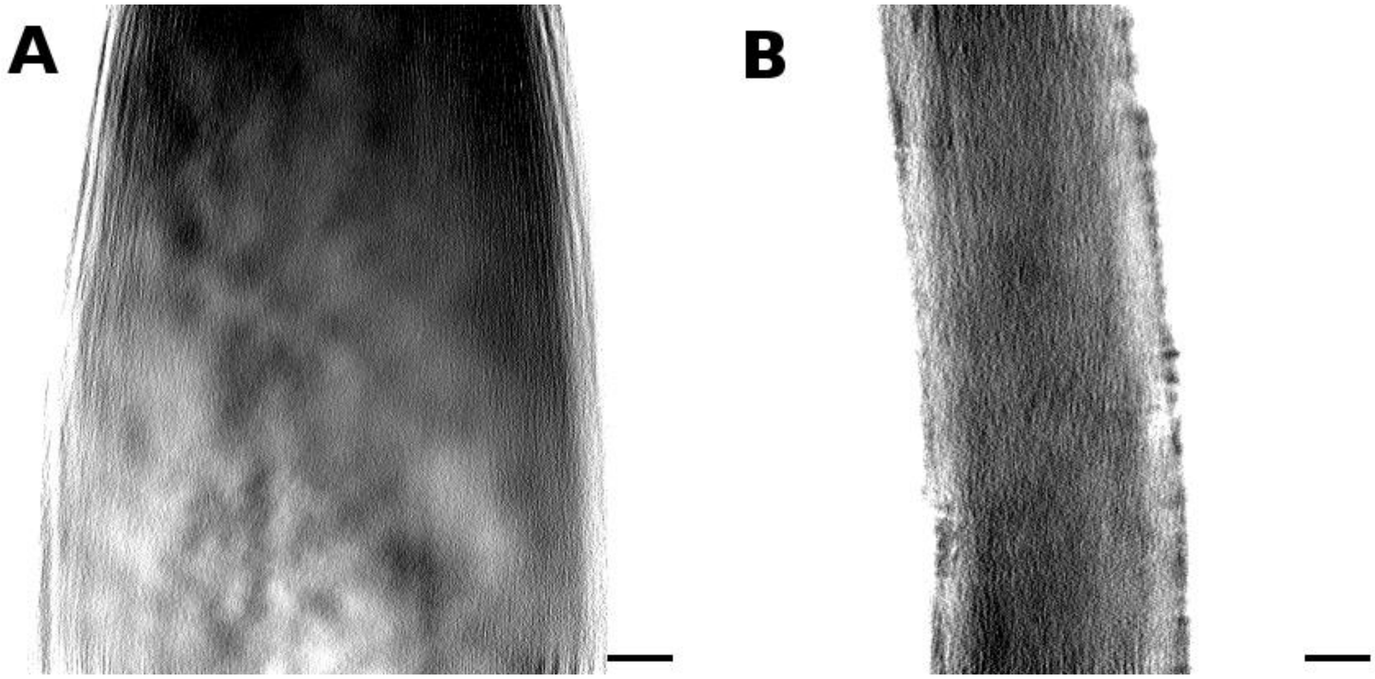

Figure 1: (A) Cross-section of a $Pd_{77.5}Cu_6Si_{16.5}$ metallic glass sample produced by melt-spinning. (B) Cross-section of a sample with the same composition, but made with vapor-deposition at elevated substrate temperatures. Scale bar: 2 µm.

An ideal way to test whether these fluctuations are real or artificial would be to measure the same sample before and after some treatments to the sample, for example annealing. This could be accomplished in a future beam time.

References

[1] Huang, B., et al. (2018). Density fluctuations with fractal order in metallic glasses detected by synchrotron X-ray nano-computed tomography. *Acta Materialia*, *155*, 69–79.
[2] Dziuba, T., Luo, Y., & Samwer, K. (2020). Local mechanical properties of an ultrastable metallic glass. *Journal of Physics: Condensed Matter*, *32*(34), 345101.