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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.

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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:** Cluster assembled metallic glasses	**Experiment number**: HC-620
Beamline: ID 03	**Date of experiment**: from: 24.04.2013 to:29.04.2013	**Date of report**:
Shifts: 15	**Local contact(s)**: Dr. Roberto Felici, Thomas Dufrane	*Received at ESRF:*

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Report:

The property-structure correlation in metallic glasses is a long standing challenge of the field. Unlike in crystaline metals, where the structure of the material can be divided into atomically distiguishable subunits, in metallic glasses (MG) the structure of the matter is solely described by the lack of such crystalline order. Recent experimental and theoretical studies on the structure of conventionally fabricated MGs suggest closely packed atomic clusters as possible building blocks for amorphous alloys [1, 2].

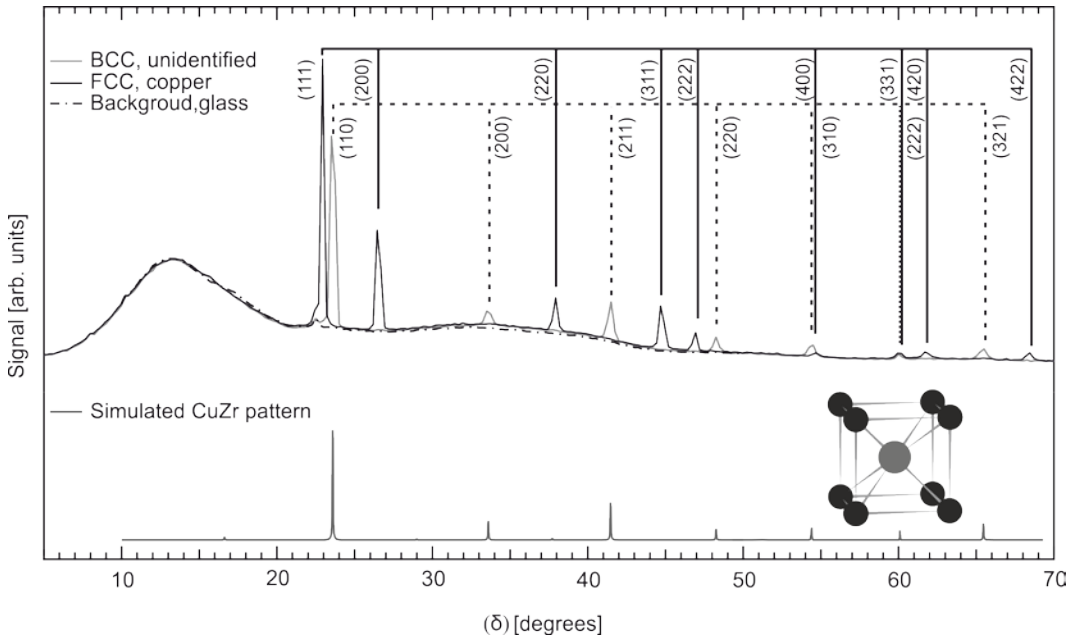
Recent advances in cluster beam sources [3], provide a great oppurtunity to examine the validity of the above-mentioned hypothesis. It has been suggestd that by producing a metallic film from gas phase metal clusters [4], the following questions could be answered : 1) is it possible to nano-fabricate an amorphous structure by deposition of gas phase metal clusters? if yes, 2) how are the structures and properties of such cluster-assembled metallic glasses (CAMG) related to conventionally produced MGs?

Cluster-assembled $Cu_xZr_{(1-x)}$ metallic films were produced by depositing a combination of clusters generated through a state-of-the-art laser vaporization cluster beeam source [5] on to borosilicate plates actively kept below room temperature. Samples were then transported under argon atmosphere and at room temperature to ID03 at ESRF for surface x-ray diffraction (SXRD) measurements. After initial difficulties with the quality of the samples

SXRD experiments were carried out with the x-ray wavelength tuned to 15keV to be well above Cu K-edge (~9keV) and below Zr K-edge (~18keV). Measurements were generally performed at room temperature, except in one case where the sample was first studied at room temperature and then heated up to 450 K.

For samples with cluster coverage lower than 10^{16} cm^{-1}, no crystalline structure is observed. Absence of any crystalline diffraction pattern may be interpreted as an indirect proof for presence of amorphous structures. Due to the diffraction signal originating from the amorphous glass substrate, however, the unambiguous observation of the expected halo structure from the metallic films is not trivial. For the thickest sample in our set (cluster coverage > $2x10^{16}$ cm^{-1}), however, two series of crystalline peaks are observed in the

Figure 1

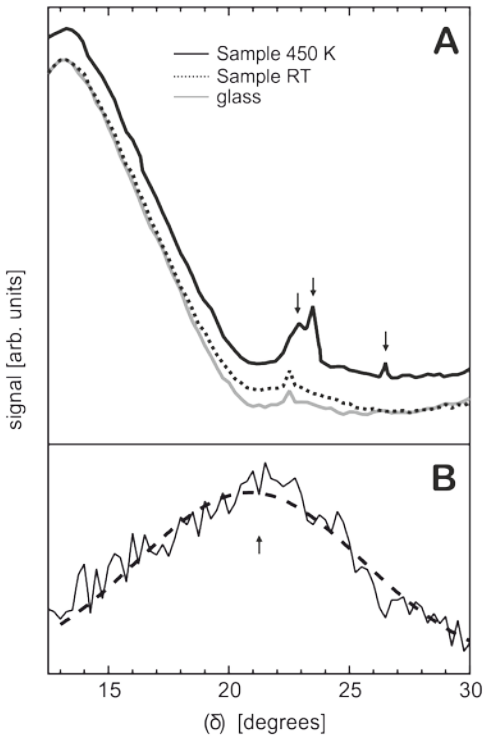


diffraction pattern as shown in figure 1, where the two independent peak series are labelled. One of the series can be confidently assigned to face centred cubic (FCC) crystals of Cu (indicated by vertical solid lines). The lattice constant of this phase is 0.36 nm, which is a perfect match for Cu. The other series (indicated by vertical dashed lines) belongs to a body centred cubic (BCC) structure with 0.286 nm lattice constant that cannot be attributed to any of previously reported phases in Cu-Zr phase diagram and relevant oxide phases. Our simulations (also included in the figure) suggest that the observed diffraction series belongs to a CuZr phase as shown in the figure inset. This phase could be generated by compressing the reported CuZr phase (lattice constatnt = 0.326 nm and stable only above 950K) and so stabilizing it at room temperature. Such compressed structures are common in thin films and have been reported in Zr-based films earlier [6, 7].

Figure 2A shows the diffraction pattern of a sample recorded at room temperature and after annealing at 450 K for 10 minutes. Also shown in figure 2A is the diffraction pattern of the

borosilicatre glass substrate used in this studies. It is clearly demonstrated that upon heating three peaks grow on top of the otherwise festureless diffraction pattern (indicated by arrows). These peaks are located at exact positions for the three most intese peaks observe in figure 1. Since the metastable noncrystalline phase could be transferred to the same crystalline structures observed in other sample with thicker film, we conclude that the longer deposition and thus further temperature raise during sample preparation is responsible for the presence of crystalline phases in thicker samples. Figure 2B presents the difference between the cluster-coated and uncoated silicaglass. The position of the observed halo corresponds to a scattering vector of 27.43 nm^{-1}, which is in a good agreement with previous studies [8].

Figure 2



We could demonstrate for the first time that non-crystalline metallic films could be produced by deposition of gas phase metal clusters. Although, a full study of composition dependence could not be realised due to the quality of our samples this time, such measuremnts are on the way.

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