ESRF	**Experiment title: In-situ X-ray diffraction and tomography investigation of preheated bulk metallic glass**	**Experiment number**: MA-720
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Shifts: 12	**Local contact(s)**: Thomas Buslaps, Marco DiMichiel	*Received at ESRF:*

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Report:

The mechanical properties of bulk metallic glasses (BMG) are different from crystalline materials. They show high strength on the expense of brittleness with almost no plastic deformation ability at room temperature. Heat treatment in the temperature range between Tg (Tg-glas transition temperature) and Tx (Tx-crystallization temperature) changes the microstructure of BMG by forming nano-crystallites and also a crystalline volume fraction, which lead to an improved plasticity of BMG. Prior to the here performed experiment samples of the BMG $Cu_{46.25}Zr_{44.25}Al_{7.5}Er_2$ with Tg=698K and Tx=762K were annealed at T=743K for 20 minutes.

The in-situ tensile creep experiments were carried out at a fixed temperature (T=743K) and two fixed loads (σ_1=150MPa, σ_2=110MPa) in order to observe the further evolution of the volume fraction of the nano-crystallites and the crystalline phases. Combined high energy dispersive diffraction with monochromatic beam and tomography with white beam were used to follow the forming, and evolution of the crystalline phases including their identification and the forming and evolution of pores and cracks, which finally cause the fracture of the material.

Miniature creep samples of the BMG $Cu_{46.25}Zr_{44.25}Al_{7.5}Er_2$ fitting into a miniature creep device exhibiting a gauge volume of appr. 1mm³ were used for the tensile creep experiments. For the diffraction analyses a 2D detector, model Pixium 4700, with a pixel size of 154µm and an area of 381.9mm horizontal and 294.1mm vertical corresponding to an image size of 2480pixel*1910 pixel was used. The sample to detector distance was 1620mm, which results in a range of 2θ=5° vertical and 2θ=7° horizontal of the transmitted and diffracted beam on the 2D detector image plate. The beam size for the XRD measurements was

0.25mm*0.25mm. For the tomography a DALSA 1M60 CCD-camera with a chip-size of 1024*1024 pixels was used. The pixelsize of 1.2µm leads to a field of view of 1.2*1.2mm² of the sample and a resolution of 1.6µm/ pixel in the tomograms. The distance from sample to detector was 200mm. The measurements were carried out sequentially: At first the tomography was done using white beam, followed by the diffraction measurements with monochromatic beam at E=88keV. A translational movement of the whole sample station of appr. 57mm perpendicular to the beam was done, for switching from the tomography to the diffraction position. To protect the 2D diffraction detector during the tomography measurements a lead plate was inserted. To protect the tomography camera during the diffraction measurements the camera was moved from the beam position. One run with tomography and diffraction requires 7 minutes (tomogram appr. 4 minutes, diffraction pattern appr. 2 minutes). The sample was each rotated around 180° for taking the tomogram as well as for the diffraction pattern. Three samples were measured: two samples at T=743K and σ=150MPa. Both samples fractured during beamtime. A further sample was measured at T=743K and σ=107MPa. This sample did not fracture during the beamtime.

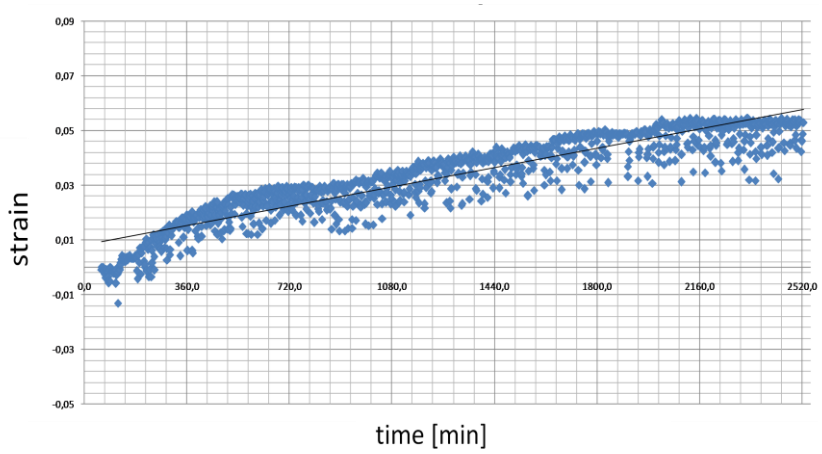

Fig.1 Macroscopical strain in tensile load direction; T=743K, σ=107MPa

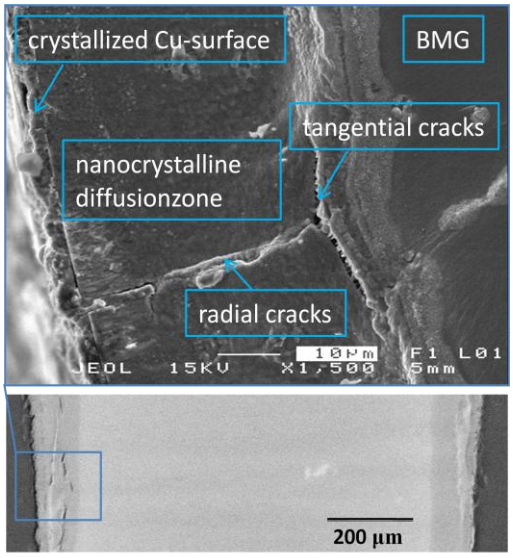

Fig.3 Microstructural details (SEM) of the near surface layer, T=743K, σ=107MPa, t≈2500min

Fig. 1 displays the macroscopical strain as a function of time for the sample exposed to T=743K and σ=107MPa. After the initial loading a nearly linear increase in strain is observed. In contrary to "usual" creep curves, the increase in strain is slowing down beyond appr. 2000min. This may be due to the ongoing growth of the surface layer, shown in Fig. 2. The surface layer shows a different density from the bulk material and the ongoing growth of the surface layer consequently leads to the formation of radial as well as tangential cracks (Fig. 2a, Fig. 3). Fig. 4 shows the time dependent formation of tangential and radial cracks. The density difference between the bulk and the surface layer of the sample is due to the formation of a nano-crystalline zone and a crystallized zone at the outer surface of the sample due to the high temperature and stress exposure of the originally glassy material. The increase in the crystalline volume content is demonstrated by the increase in reflection intensities in the diffractogram (Fig. 5). Further manifold evaluations of the experimental data are still under way.

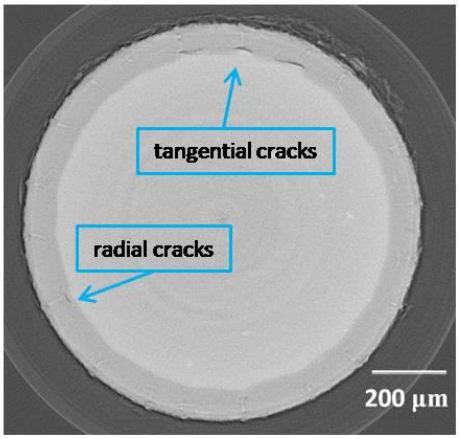

Fig. 2a Tomographic slice perpendicular to the load axis

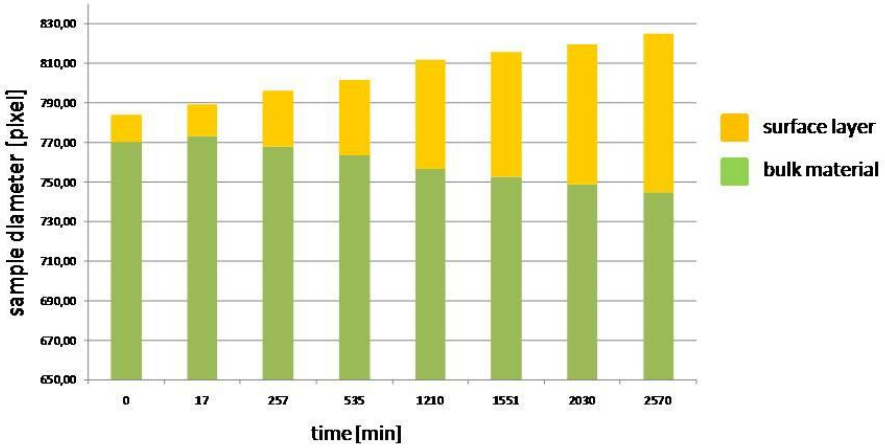

Fig. 2b sample diameter (green: bulk material; yellow: surface layer)

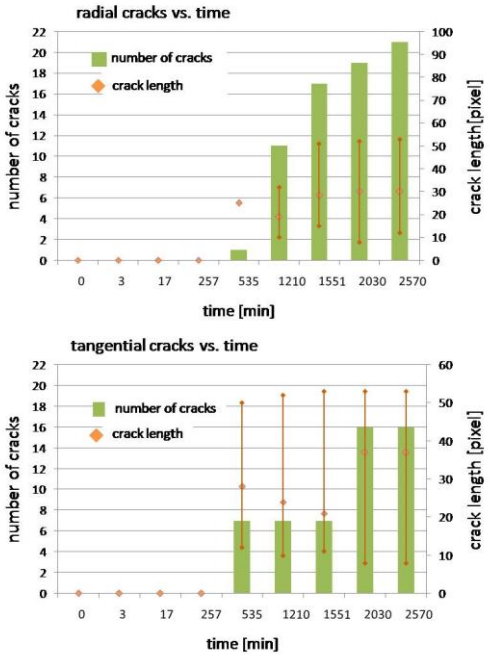

Fig. 4 tangential and radial cracks

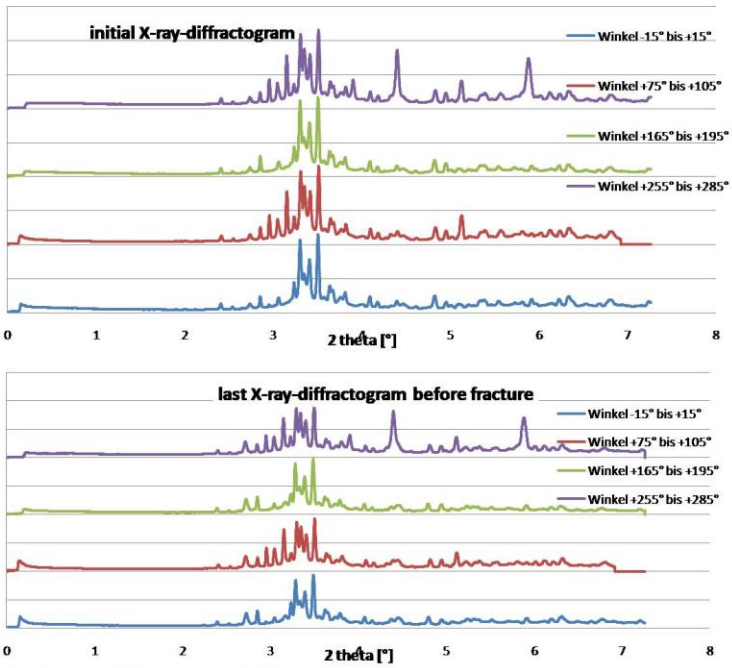

Fig. 5 In-situ diffractograms, T=743K, σ=107MPa