In-situ structural investigations of kinetically strong bulk metallic glass-forming liquids in containerless environment

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Metallic glasses—also known as amorphous alloys— are a relatively new class of materials. Their unique combination of properties makes them promising candidates for many fields of applications. Due to the lack of a long-range ordered lattice structure, metallic glasses show unique properties, such as interesting combinations of mechanical properties (e.g. high yield strengths combined with high elastic limits), the capability for thermoplastic forming similar to the processing of polymers, as well as net-shape forming. Metallic glasses were first discovered in 1959 by Paul Duwez’s group at the California Institute of Technology. Early alloy compositions were characterized by high critical cooling rates needed to freeze-in the liquid’s structure and therefore could only be produced up to thicknesses in the µm-range; nowadays alloys can be processed with much slower cooling rates (< 100 K s⁻¹) and therefore can be produced up to several cm in thickness. In general, bulk metallic glasses are formed by quenching the liquid fast enough to avoid crystallization. As a result, the supercooled liquid state is frozen-in and preserved at room temperature. It has been suggested that short-range and/or medium-range order may be present. However, investigations of the structure of the supercooled liquid state are difficult because of the interference of crystallization. Nevertheless, recent studies on levitated droplets have been performed and show evidence for transformations within the liquid state, which might be related to the high glass forming ability of these alloys [1].

With the aim to investigate structural changes within the liquid state, several bulk-glass forming alloys were investigated using in-situ high-energy synchrotron X-ray scattering at the PETRA III-P07 beamline. In order to avoid heterogeneous nucleation, experiments were performed in an electrostatic levitator (ESL) under high-vacuum conditions (10⁻⁷ mbar) [2]. Sample temperature was controlled using infrared lasers and captured with pyrometers.

Figure 1: Electrostatic levitation (ESL) setup used for in-situ structural investigations of glass forming liquids in a containerless environment.

Credit: Institute of Materials Physics in Space at the German Aerospace Centre (DLR) in Cologne, Germany

Diffraction studies were performed in transmission mode using a Perkin Elmer 1621 AN/CN digital x-ray detector at a wavelength of 0.124 Å—corresponding to a photon energy of 100 keV. Intensity patterns were obtained by integrating the measured two-dimensional XRD-patterns using Fit2D software [3]. Subsequent data analysis was performed using PDFgetX2 [4] software. The integrated measured intensity patterns I(Q) were corrected by background subtraction and correction terms for sample absorption, fluorescence and inelastic (Compton) scattering. The total
structure factors S(Q) were obtained from integrated intensities I(Q) after the correction of Laue diffuse scattering and normalization to the atomic X-ray form factor. Finally, the reduced pair distribution functions G(r) were calculated from the total structure factors S(Q).

A variety of metallic glass forming alloys was successfully investigated during heating and cooling and the corresponding structural changes were determined from the measured intensity data. In case of the Zr_{58.5}Cu_{15.6}Ni_{12.8}Al_{10.3}Nb_{2.8} bulk metallic glass forming alloy (Vit.106a) we were able to undercool the liquid fast enough to avoid crystallisation which enabled a complete in-situ study of structural changes within the liquid state from more than 300°C above the liquidus temperature down to the glassy state. Figure 2 shows exemplarily the evolution of the reduced pair distribution function G(r) of Vit.106a as a function of temperature from 1200°C to 400°C (left side). From these patterns, it is possible to draw conclusions about the structural evolution of the liquid state and related physical properties during undercooling, e.g., the thermal expansion of the second coordination shell as deduced from the shift of the 2nd peak position (right side). Other studied alloys including Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5} (Vit.1) and Zr_{46.75}Ti_{8.25}Cu_{7.5}Ni_{10}Be_{27.5} (Vit.4) have been measured and the results will be shown elsewhere.

In summary, high energy x-ray diffraction measurements were successfully conducted on a variety of kinetically strong metallic glass-forming alloys. The obtained data will help explore possible transformations in the liquid state, which may be related to the glass forming ability.

References