Anomalous Small-Angle X-ray Scattering Studies of Phase Separation in Zr-Based Bulk Amorphous Alloys

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Introduction

Several Zr-based bulk glass-forming alloys undergo phase separation upon annealing, prior to the crystallization of the final stable intermetallic crystalline phases.[1-4] Not all bulk metallic glasses based on zirconium, however, undergo phase separation prior to crystallization; $Zr_{46.8}Ti_{8.2}Cu_{7.5}Ni_{10}Be_{27.5}$, for example, does not, which may explain why its glass-forming ability is not as good as some of the other Zr-based alloys.[5]

We have performed small-angle x-ray scattering (SAXS) and anomalous small-angle x-ray scattering (ASAXS) on samples of amorphous $Zr_{52.5}Ti_5Cu_{17.9}Ni_{14.6}Al_{10}$ annealed at temperatures near the glass transition. We observe the development of phase separation on the nanometer scale. ASAXS observations performed near the Zr-*K* absorption edge reveal that the distribution of Zr is homogeneous in the as-cast samples but becomes increasingly inhomogeneous with increasing annealing time.

Methods and Materials

Samples of the amorphous alloy were produced by casting of master alloy ingots into a copper mold. We performed the SAXS observations on beamline 12-ID of the Advanced Photon Source; the design of the SAXS instrument is described in detail elsewhere.[6] Scattering experiments were conducted at several x-ray energies near the Zr-*K* absorption edge (17998 eV). After normalization to absolute units (using a polyethylene standard), SAXS patterns at two different energies were subtracted to produce differential anomalous scattering patterns.

Results

Differential ASAXS patterns from samples annealed for various times at 705~K (in the supercooled liquid region) are shown in Fig. 1. The development of a pronounced scattering maximum clearly indicates that phase separation involving Zr takes place at this temperature. The earlier times correspond to the onset of phase separation; at longer times, wide-angle scattering (not shown) reveals the presence of nanometer-scale crystalline precipitates of intermetallic phases.

Discussion

The data in Fig. 1 clearly indicate that phase separation involving Zr occurs in the early stages of crystallization of amorphous $Zr_{52.5}Ti_5Cu_{17.9}Ni_{14.6}Al_{10}$. We have also collected data at the Cu and Ni absorption edges; these data, which could reveal phase separation of these elements, are presently under analysis. At this point we do not have enough data to definitively identify the mechanism of phase separation. A leading candidate is spinodal decomposition, but conclusively identifying this mechanism will require data from additional samples annealed for short times. These experiments are ongoing.

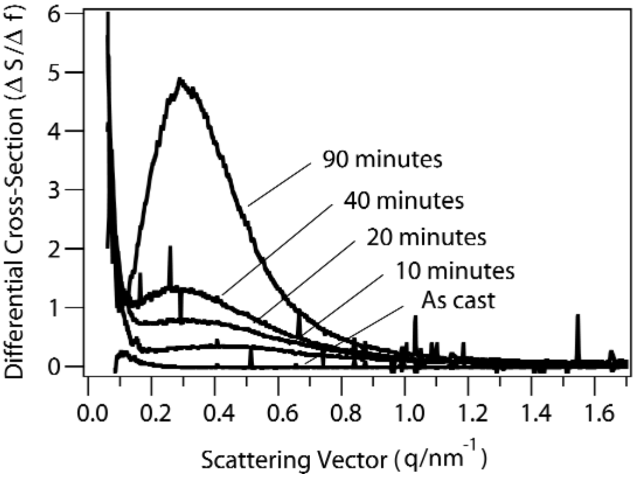

FIG. 1. Differential SAXS at the Zr-K absorption edge.

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