Nanocrystallization of Al-rare earth metallic glasses studied by time-resolved simultaneous small- and wide-angle X-ray scattering

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Nanocrystallization process in binary aluminium-rare earth ($Al_{91}Gd_9$ and $Al_{92}Sm_8$) metallic glasses was investigated *in-situ* by means of time-resolved simultaneous small and wide-angle X-ray scattering (SAXS/WAXS) method. The SAXS/WAXS data were acquired every 60 seconds during isothermal annealing at temperatures close to the crystallization onset point. The resulting SAXS spectra exhibit a characteristic maximum indicating the presence of compositional fluctuations with average size of about 10 nm appearing in the initially homogeneous amorphous phase. The SAXS data are reminiscent of phase separation occurring via spinodal mechanism. Simultaneously taken WAXS spectra prove that the observed compositional fluctuations have amorphous character.

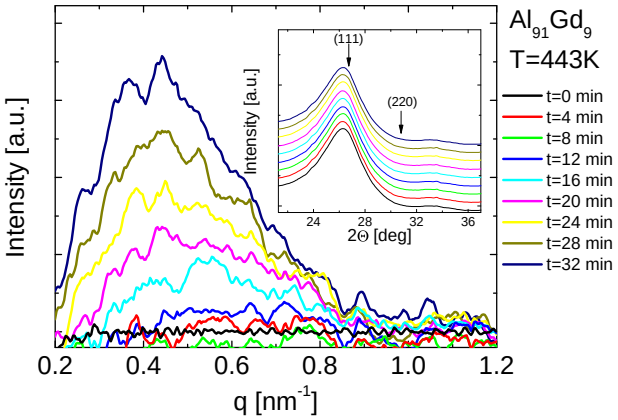

Figure 1. SAXS spectra evolution for $Al_{91}Gd_9$ metallic glass annealed isothermally at 443K. The inset shows simultaneously taken WAXS spectra indicating fully amorphous character of the sample.

On further annealing the Bragg peaks of nanocrystalline fcc-Al phase appear on the amorphous "halo" pattern in the wide-angle regime. The crystalline volume fraction was derived from WAXS spectra using method based on total scattered intensities originating from amorphous and crystalline phase [1]. The results are in good agreement with the differential scanning calorimetry (DSC) data. The average nanocrystal size was calculated from the Bragg peak broadening using Scherrer formula and was found to be nearly constant in course of the transformation.

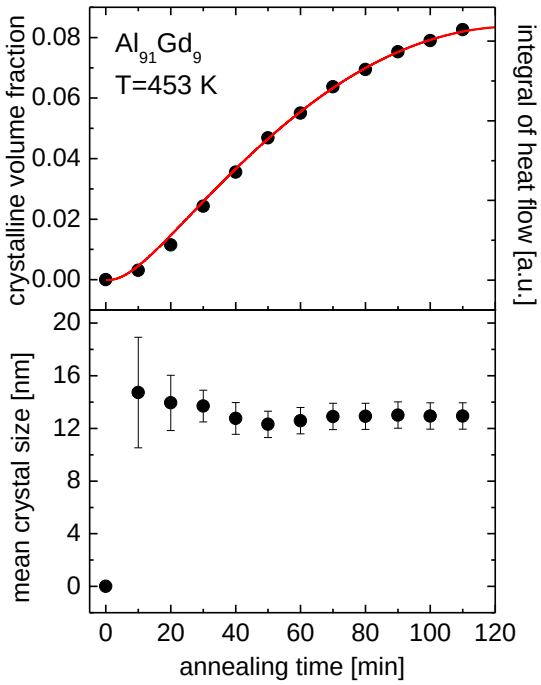

Figure 2. Crystalline volume fraction (top) and mean crystal size (bottom) obtained from WAXS data taken at 453K. The integral of heat flow from isothermal DSC experiment is included in the crystalline volume fraction plot.

The SAXS/WAXS results indicate that the nanocrystallization process in Al-rare earth metallic glasses is triggered by decomposition of the glassy phase into Al-rich and Al-poor amorphous phases. Subsequent crystallization process is initiated by nucleation of fcc-Al phase in the Al-rich phase regions and the crystal size is constrained by a nanometer-scale size of the regions [2,3]. The Al-poor phase acts as a residual amorphous matrix with higher thermal stability against crystallization. The above mechanism leads to formation of a nanocrystalline microstructure consisting of nanocrystals embedded in an amorphous matrix.

References

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