Time-Resolved Studies of $Ti_{34-x}Cu_{47}Zr_{11}Ni_8Si_x$ Metallic Glass Devitrification Using High-Temperature X-ray Powder Diffraction

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Introduction

Time-resolved devitrification studies of metallkic glasses Ti_{34}-x$Cu_{47}Zr_{11}Ni_8Si_x$ were performed using a recently developed high-temperature furnace in a Debeye-Scherrer geometry. Samples included powders produced by high-pressure gas atomization (HPGA). Synchrotron radiation at the Advanced Photon Source (APS) at Argonne National Laboratory was used to follow the devitrification of samples during heating at 40 K min^{-1} between 623 and 1073 K. The crystallization behavior observed with structural diffraction data compare well with results from thermal analysis using differential scanning calorimetery (DSC). At 1073K, these amorphous alloys evolve to a four-phase microstructure that includes phases that appear to be closely related to $Cu_{51}Zr_{14}$, CuTi, and Cu_2TiZr.

Methods and Materials

Two similar metallic glass alloys were used: $Ti_{34}Cu_{47}Zr_{11}Ni_8$ and $Ti_{33}Cu_{47}Zr_{11}Ni_8Si_1$. Samples of each composition were prepared as powders using HPGA. As discussed by Choi-Yin *et al.*,[1] the addition of Si to Ti-Cu-Zr-Ni metallic glasses increases the onset of primary crystallization (Tx), and extends the supercooled liquid region. The time-resolved studies of the powders were performed with the 6ID-B beamline at the APS in collaboration with the Midwest Universities Collaborative Access Team (MU-CAT). The data presented in this study were obtained with an incident energy of 38.33 keV. The atomized powders were loaded into 2-mm internal diameter, thin-walled amorphous silica tubes. Prior to sealing, the tubes were evacuated to a pressure $< 10^{-2}$ mbar and then backfilled with Ar. A Debeye-Scherrer geometry (with an area detector) for collection of diffracted x-rays was employed. The detector was a 200 x 250 mm Fuji image plate with pixel dimensions of 100 x 100 µm. Intensity correction was determined using NBS intensity standard 674 $A_{12}O_3$.

Results and Discussion

At lower temperatures (e.g., 773K), the time-resolved data do not show the primary crystallization peak that developed during heating for 24 h at the same temperature. At intermediate temperatures of 973 K, the continuous heating diffraction data is comparable to the data from the sample annealed for 24 h, but further heating to 1073 K again shows that the continuously heated sample did not fully devitrify. Despite the likely higher diffusion rate at 1073 K, other factors (e.g., diffusion distances due to prior crystallization) must be retarding final crystallization during continuous heating.

Time-resolved x-ray diffraction detects the formation of non-equilibrium phases that may not be observable with traditional heat and quench techniques.[2] Here we demonstrated that the thermal events observed in the Ti metallic glasses heated at 40K min^{-1} in the DSC do not necessarily correspond to equilibrium phases. The current experimental set-up will allow for extended dwell times at temperature to track phase changes over time.

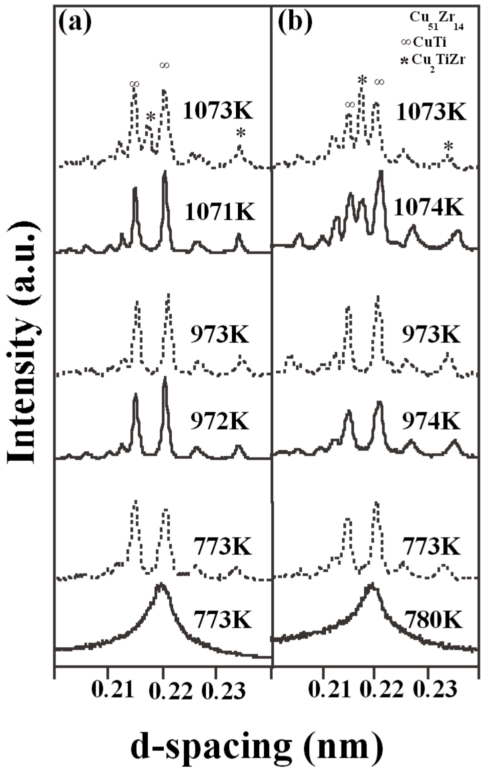

FIG. 1. (a) $Ti_{34}Cu47Zr_{11}Ni_8$ and (b) $Ti_{33}Cu_{47}Zr_{11}Ni_8Si_1$ atomized powder obtained during heating at 40 K min^{-1} between 623 and 1073 K (solid lines) and obtained at room temperature following annealing for 24 h at indicated temperatures (dotted lines).

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