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3DAP and TEM analysis of crystallization in Zr-Ti-Cu-Ni-Be metallic glass

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The Zr-Ti-Cu-Ni-Be metallic glass developed by Peker and Johnson [1] exhibits a wide supercooled liquid region and an excellent glass forming ability. The crystallization process of this metallic glasses has been extensively investigated in order to understand the thermal stability of this alloy [2-4], and it was reported that nanocrystalline microstructure developed after the phase decomposition of the supercooled liquid [2-4]. However, the detailed mechanism of the nanocrystalline microstructure evolution from the Zr-Ti-Cu-Ni-Be metallic glass is not completely understood. In this work, we have investigated the pre-crystallization and early crystallization stages of Zr-Ti-Cu-Ni-Be metallic glass by X-ray diffraction (XRD), three-dimensional atom probe (3DAP), transmission electron microscopy (TEM/HREM) and energy filtered electron diffraction to establish whether or not a phase separation or a structural change can be detected prior to the onset of the crystallization.

Amorphous $Zr_{41.8}Ti_{14.1}Cu_{12.5}Ni_{10.4}Be_{21.2}$ ribbon was prepared by single roller melt spinning. The thermal stability of the ribbon was studied by differential scanning calorimetry (DSC). The ribbon samples were isothermaly heat treated at 400°C for 5, 10, 30 and 60 min in a helium atmosphere. The glass transition temperature was chosen as the isothermal annealing temperature. The microstructures were characterized by JEOL JEM-2010F, Carl Zeiss LEO-922 OMEGA and a locally built energy compensated 3DAP equipped with the CAMEMA optical tomographic atom probe detection system.

Figure 1 shows bright field TEM micrographs and the selected area electron diffraction (SAED) of the specimens annealed for (a) 5, (b) 10, (c) 30 and (d) 60 min. The specimens annealed less than 30 min showed structurally uniform amorphous phase. The nanocrystalline particles were observed in the specimens annealed for 30 and 60 min. The structure of the initial crystalline phase was identified as quasi-crystal by XRD and SAED. Also, Ti rich phase in the precipitations was examined by energy dispersive X-ray spectroscopy. Figure 2 shows 3DAP analysis result of the specimen annealed for 60 min. Ti-rich and Be-poor regions were found and other elements were distributed uniformly. Thus, it is understood that the decomposition of Ti and Be closely related to the nano-quasi-crystallization. The crystallization process of this alloy will be discussed in details based 3DAP, TEM, HREM and energy filtered electron diffraction results. [5]

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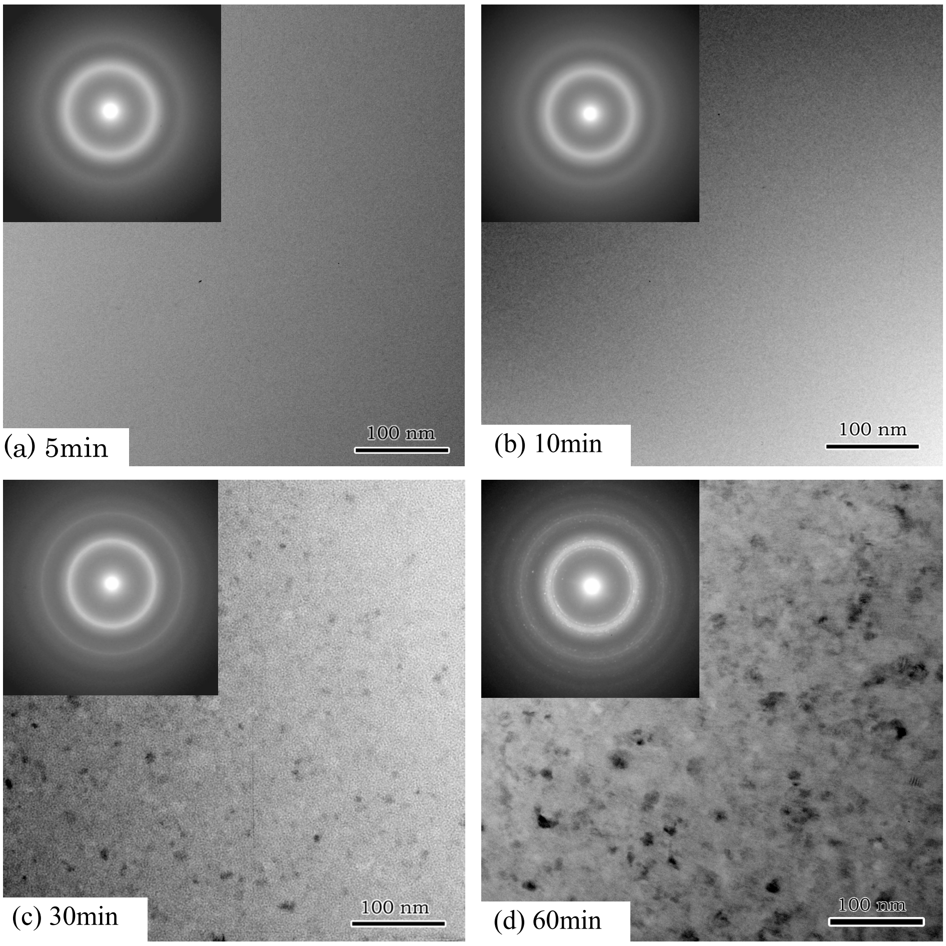

Figure 1. Bright field TEM micrographs and the SAED of the specimens annealed for (a) 5, (b) 10, (c) 30 and (d) 60 min.

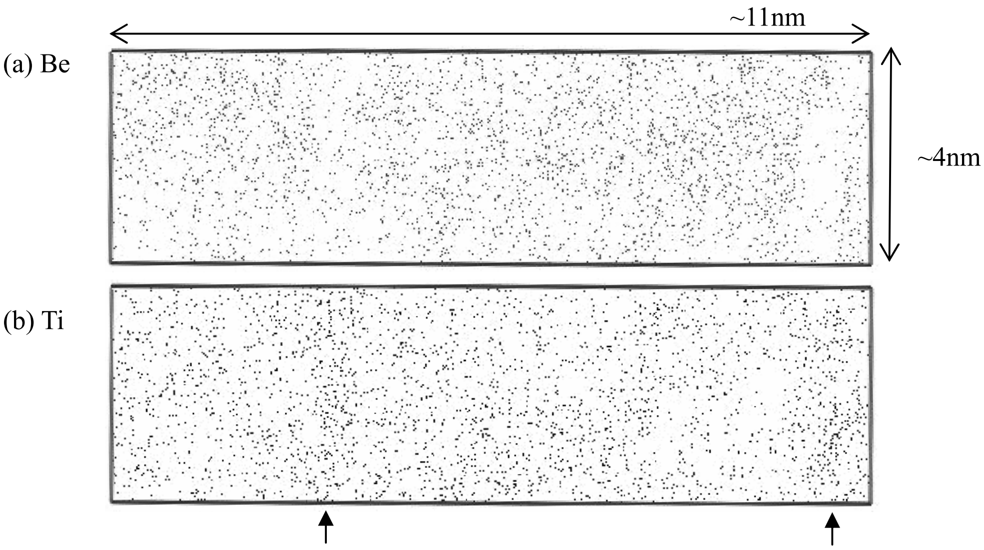

Figure 2. 3DAP analysis result of the specimen annealed for 60 min. Elemental mapping of (a) Be and (b) Ti. Arrows are showing Ti-rich and Be-poor region.