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Radial and longitudinal variations in the Young's modulus of a $Zr_{55}Al_{10}Ni_5Cu_{30}$ bulk metallic glass rod

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ABSTRACT

Radial and longitudinal variations in the Young's modulus of a $Zr_{55}Al_{10}Ni_5Cu_{30}$ metallic glass cylindrical rod were examined by means of nanoindentation. The metallic glass rod, 2.4 mm in diameter, was prepared using a novel mold-casting method designed to suppress crystallization resulting from the heterogeneous nucleation originating from slag covering a molten alloy. In this study, Young's modulus was measured using a multiple partial unloading technique with a spherical indenter. In general, it is assumed that the cooling rate of a metallic glass rod depends on the radius of its cross section. However, no significant difference was found in the measured Young's modulus.

1. Introduction

Amorphous alloys have isotropic properties that originate from their noncrystalline random atomic arrangement. They exhibit distinctive mechanical properties, such as high strength, low Young's modulus, and large elastic strain, compared with conventional crystalline metallic materials. However, amorphous alloys have not been widely used because they do not form bulky materials. Recently, some amorphous alloys, which are called metallic glasses, have been found exhibit glass transition. A number of molten alloys have been reported to form bulk metallic glasses by casting, because a supercooled liquid state of metallic glasses is more stable than that of conventional amorphous alloys. In particular, Zr- and Pd-based metallic glasses can form bulk specimens greater than 10 mm in diameter, even if they are cooled slowly [1–3]. The distinctive isotropic and mechanical properties of bulk metallic glasses are promising. These properties act as standard specimens for calibrating a wide range of testing equipment, in particular, for testing the mechanical properties of materials used in micro-electro-mechanical system (MEMS) devices.

However, the mechanical properties of a bulk metallic glass specimen are expected to vary from place to place because the cooling rate during the casting of a specimen is not uniform. In addition, variations in properties may influence calibration if the specimen is used as a standard. To use bulk metallic glasses as standard specimens for calibrating equipment for MEMS devices, their properties need to be ascertained by the equipment to confirm whether the variations in the properties of bulk metallic glasses are detectable.

In this study, radial and longitudinal variations in the Young's modulus, which is one of the mechanical properties of a Zr-based bulk metallic glass cylindrical rod, were investigated by using nanoindentation equipment.

2. Experimental procedures

Ingots of $Zr_{55}Al_{10}Ni_5Cu_{30}$ composition were prepared by Ar arc-melting. The ingots were crushed into pieces and subjected to modified mold-casting using a Cu horizontal mold in an Ar atmosphere. The pieces were placed in a silica nozzle and melted using high-frequency induction heating. Then, the molten alloy was injected into the Cu horizontal mold using pressurized Ar gas. The cross section of the Cu mold used in this study is shown in Fig. 1. The injected molten alloy was first stored in a reservoir. At this point, the molten alloy was covered with slag consisting of oxides and contamination. When the reservoir became full, the slag was ripped apart by the pressurized Ar gas, and the molten alloy stored in the reservoir began to flow into the horizontal mold. The molten alloy was rapidly quenched as it flowed into the horizontal mold, and a shiny cylindrical rod of 2.4 mm in diameter was formed. It is suspected that the ripped slag remained trapped at the edges of the opening between the reservoir and the horizontal mold and that crystallization by heterogeneous nucleation was suppressed. The flow rate between the bottom surface and the top surface of the molten alloy in the horizontal mold was almost the same because the tip of the rod obtained was spherical.

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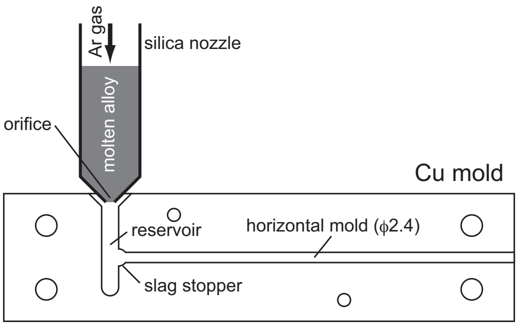

Fig. 1. Cross section of a Cu horizontal mold used for casting a metallic glass rod.

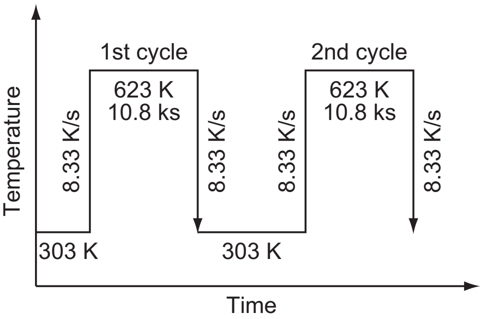

Fig. 3. Temperature history programmed for isothermal DSC measurement.

In this study, the length of the cylindrical rod prepared by this method was approximately 80 mm. The rod was then sliced into 1-mm thick disks, as shown in Fig. 2. The disks were then polished using colloidal silica powder. The cross sections of the disks were examined by X-ray diffraction (XRD) using Cu Kα radiation. The crystallization temperature T_x and the glass transition temperature T_g of one of the disks sliced at position B (see Fig. 2), 10 mm from the tip of the rod, were measured using a power-compensation-type differential scanning calorimeter (DSC) (Diamond DSC, Perkin-Elmer, MA, United States) at a heating rate of 0.67 K/s.

The depth profiles of the distribution of several elements – Cu, C, N, and O – in the polished surface layer of another disk at position B were examined at every 1 nm in depth by means of Auger electron spectroscopy (AES) with Ar sputtering to ascertain the effect of polishing on the Young's modulus. The intensity of Auger electrons of $\mathrm{Cu_{LMM}}$ at 920 eV, $\mathrm{C_{KLL}}$ at 272 eV, $\mathrm{N_{KLL}}$ at 381 eV, and $\mathrm{O_{KLL}}$ at 510 eV was monitored. The Auger electrons of Zr, Al, Ni, Si were not monitored because of overlapping of the Auger electron energy with other elements or because they were very low in intensity for analysis.

In this study, six disks sliced at positions F, G, and H (30, 35, and 40 mm from the tip of the rod, respectively) were subjected to isothermal DSC measurement or nanoindentation. The endothermic heat flow into the disks was measured, using isothermal DSC measurement, for the disks neighboring those subjected to nanoindentation. The temperature history programmed for the isothermal DSC measurement is schematically shown in Fig. 3. The specimens were rapidly heated at a rate of 8.33 K/s and then maintained at 623 K for 10.8 ks, as programmed. However, the cooling rate was slower than intended. Isothermal DSC measurements were performed twice for each specimen, with the endothermic heat flow for the second measurement being used to provide a baseline.

The reduced Young's modulus E_r of the specimens was measured by means of a multiple partial unloading technique with a spherical indenter proposed by Field and Swain [4–6], using a nanoindentation measurement device (UMIS-2000, CSIRO, Lindfield, Australia). In this study, the radius of the spherical indenter used was 5 μm, and the maximum load applied was 0.8 mN. The frame compliance of 0.3 nm/mN was used to analyze the indentation results. The equipment was calibrated using fused silica as a standard specimen, whose E_r and Poisson's ratio ν were 69.6 GPa and 0.17, respectively. The indents made by the measurement were observed by using a scanning electron microscope (SEM).

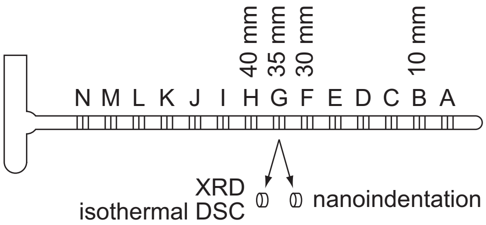

Fig. 2. Disks sliced from the metallic glass rod. Disk B was used to measure T_x and T_g. Disks F, G, and H were subjected to XRD, isothermal DSC and nanoindentation measurements.

3. Results

3.1. Specimen characterization

Fig. 4 shows the XRD pattern of the cross sections of disks and the DSC trace of one of the disks sliced at position B. The halo peaks in the XRD pattern show that the disk has a random atomic arrangement. In the DSC trace, endothermic heat was detected prior to crystallization, indicating that the disk exhibited glass transition. Thus, the disks were confirmed to be glassy. T_x and T_g are 773 K and 682 K, respectively. Fig. 5 shows the depth profiles of Cu, C, N, and O elements in the polished surface layer of another disk B. The top surface within 20 nm includes more light elements, which resulted from polishing compounds or contamination, than the deeper part of the specimen.

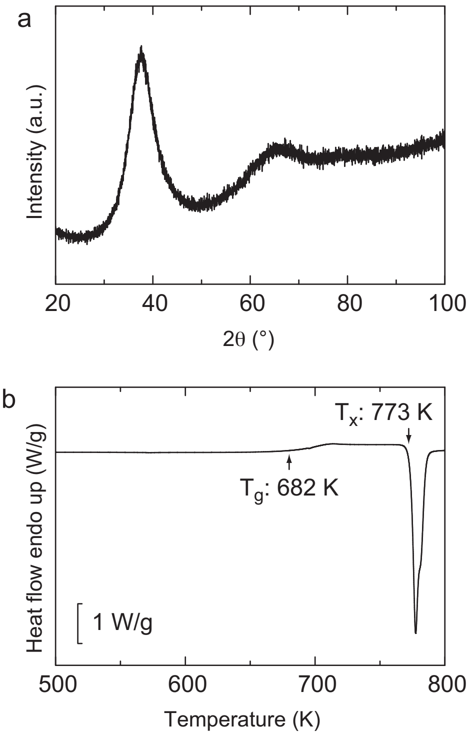

Fig. 4. (a) XRD pattern of disks subjected to mechanical testing, and (b) DSC trace of another disk.