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Evaluation of Micro-Sized BMG Tensile Specimen Fabricated by Electrolytic Polishing Technique

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An electrolytic polishing technique was used to fabricate micro-sized tensile specimens from two $Zr_{55}Al_{10}Ni_5Cu_{30}$ bulk metallic glass samples. One sample had a fully amorphous phase, while the other had dendrites with an average size of 33 µm within the amorphous matrix. A scanning electron microscope showed that specimens fabricated by electrolytic polishing had smooth surfaces. The specimens were then subjected to tensile tests at room temperature. The fully amorphous specimen had a fracture strength of 1,640 MPa. This specimen also showed slight plastic deformation after yielding, as well as vein patterns and smooth surface on the fracture surface. Multiple thin shear bands generated from a shear band were observed on the side surface of fractured specimen. The specimen containing dendrites was quite low in strength compared to the fully amorphous specimen, and had yield and fracture strengths of 460 and 600 MPa, respectively. The specimen containing dendrites also showed plastic deformation and work hardening after yielding, as well as vein patterns and a brittle fracture region on the fracture surface. [doi:10.2320/matertrans.MJ200755]

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1. Introduction

Metallic glasses exhibit exceptional high strength compared to crystalline materials.[1)] Specimens having a fully amorphous phase under uniaxial loads fail catastrophically along one dominant shear band and show little global plasticity. The plasticity of amorphous materials under uniaxial loads can be improved by homogeneously precipitating micro-scale ductile crystalline particles in an amorphous matrix.[2,3)] On the other hand, it is reported that as-cast two-phase microstructures of micro-sized crystals in an amorphous matrix exhibit significantly reduced ductility.[4)] The interactions between an amorphous matrix and a micro-sized crystal have yet to be thoroughly examined.

In a previous study, we developed a precision electrolytic polishing apparatus to fabricate micro-sized tensile specimens from bulk samples.[5,6)] Using this apparatus, we successfully fabricated micro-sized tensile specimens with diameters of less than 100 µm from crystalline materials. This technique of fabricating micro-sized specimens from bulk metallic glass samples containing micro-scale crystals allows us to directly investigate the interactions between an amorphous matrix and a micro-sized crystal.

The purpose of the present study was to determine whether or not this electrolytic polishing technique for fabricating micro-sized specimens could be applied to amorphous materials.

2. Experimental

2.1 Precision electrolytic polishing apparatus

Ohm's law can be generalized as

$$i = \kappa \,\mathrm{grad}\, f(x, y, z)$$

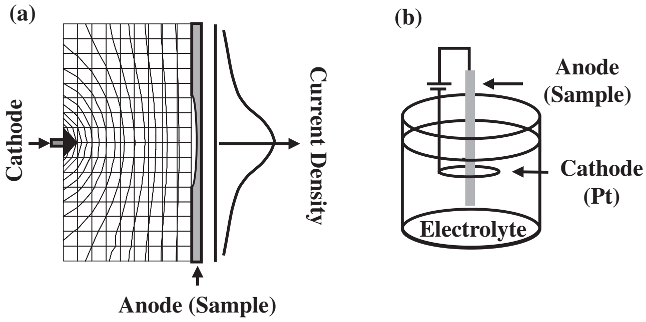

Fig. 1 (a) Simulated electric potential distribution; (b) schematic illustration of the arrangements of electrodes in the electrolyte cell.

where i is the current density, κ is the specific conductivity and f is the electric potential. This equation indicates that the current density i, which is identical to the polishing rate, is controlled by the gradient of the electric potential f when the specific conductivity κ is constant.

Figure 1(a) shows the electric potential distribution simulated on conductive paper when the cathode of 0 V is arranged in one point to the long anode of 6 V. Vertical and horizontal lines indicate a lattice drawn at intervals of 50 mm on conductive paper. The curved lines are equipotential lines drawn at intervals of 0.2 V. The sample is locally polished in an electrolyte by achieving the electric potential distribution shown in Fig. 1(a). Therefore, we can fabricate a symmetric tensile specimen by performing electrolytic polishing with the electrode arrangements shown in Fig. 1(b) in an electrolyte with a constant specific conductivity.

In a previous investigation, we developed a precision electrolytic polishing apparatus based on this idea and used it to fabricate micro-sized tensile specimens from bulk crystalline materials.[5,6)] The cathode of this apparatus is a platinum ring 0.3 mm in thickness and 4 mm in diameter. The apparatus maintains a specific conductivity on the surface of the sample during electrolytic polishing by applying a

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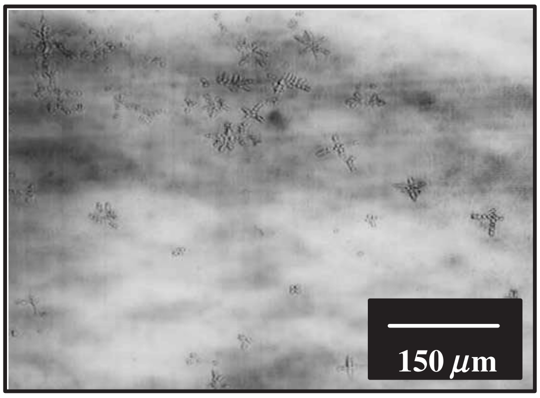

Fig. 2 Laser micrograph of the partially crystallized sample surface after etching, showing the presence of dendrites in the amorphous matrix.

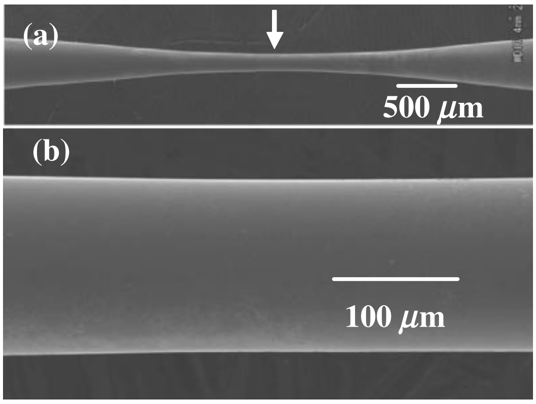

Fig. 3 Scanning electron micrographs of the micro-sized tensile specimen fabricated by means of electrolytic polishing. A white arrow indicates the central position of the ring-shaped cathode. (a) Whole image; (b) magnified image of the center point of the specimen.

rectangular-wave voltage to the sample in a flowing electrolytic solution.

2.2 Material and specimen preparation

The material selected for the present study was Zr_{55}-$Al_{10}Ni_5Cu_{30}$ bulk metallic glass (atomic percent). Two kinds of samples were used. One was a fully amorphous sample supplied by the Institute for Materials Research, Tohoku University. The other was a partially crystallized sample which had crystalline particles that precipitated out during casting. Both samples were plates produced using a copper mold casting method. As received, the plates were 50 mm in width, 60 mm in length, and 2 mm in thickness.

Figure 2 is a laser micrograph of the surface of the partially crystallized sample after it had been etched with an electrolytic solution consisting of 10 vol% percholoric acid and 90 vol% acetic acid. Image analysis showed dendrites with an average size of 33 μm distributed in the amorphous matrix. Energy dispersive X-ray spectrometer analysis revealed that these crystallites were enriched in aluminum whereas the concentration of the other constituents was lower compared with the composition of the amorphous matrix. Micro-indentation experiment on the crystalline particles indicated that these were ductile crystals.

Square rods ($2 \times 2 \times 50$ mm) were cut from the plate using a fine cutter and mechanically ground using emery papers until they became round bars with a diameter of 1 mm. Micro-sized tensile specimens with circular cross-sections were fabricated from these round bars using the electrolytic polishing apparatus. A sample rod was placed vertically at the center of a ring-shaped counter electrode, then a rectangular wave voltage with maximum voltage of 12 V, minimum voltage of 0 V, period of 6 s, and duty cycle of 15% was applied to the sample in an electrolytic solution flowing at a rate of 1.2 mL/s. The polished specimens were cleaned by alternately rinsing them with distilled water and ethanol. Each specimen was then placed in an ultrasound bath for a few seconds to remove metal oxides that had been deposited on the surface during electrolytic polishing. The fabricated samples were examined using scanning electron microscopes.

2.3 Tensile tests of micro-sized specimens

Tensile tests were carried out using a SHIMAZU tabletop type tester (EZTest) in an ambient atmosphere at room temperature. All tests were performed at the same crosshead speed of 0.5 mm/min. After the tensile tests, the fracture surfaces were examined using scanning electron microscopes.

2.4 Analytical equipments

After the specimens had undergone electrolytic polishing and tensile testing, the shapes and surfaces were examined using a laser microscope (1LM21, Lasertec Co.) and two scanning electron microscopes (FE-SEM, S-4300 and S-4500, Hitachi High-Technologies Co.). The composition of the samples was characterized by an energy dispersive X-ray spectrometer (EDX, HORIBA).

3. Results and Discussion

3.1 Micro-sized BMG specimen fabricated by electrolytic polishing technique

Figure 3 shows scanning electron micrographs of a micro-sized tensile specimen fabricated using electrolytic polishing. The white arrow in Fig. 3(a) indicates the center point of the ring-shaped cathode. Figure 3(b) indicates the magnified image of the center point of the specimen. Examination with a scanning electron microscope revealed that the surface of the specimen was smooth. The specimen was symmetric with respect to its center point, as shown in Fig. 3(a).

3.2 Tensile properties of the fully amorphous micro-sized specimen

Figure 4 shows the stress-displacement curve results of the tensile test on the fully amorphous specimen with a diameter of 145 μm. The fracture strength was 1,640 MPa. After yielding, a small amount of plastic deformation was observed. Figure 5 shows scanning electron micrographs of the fractured specimen. A tensile fracture occurred along the plane deviating from the maximum shear stress plane, which declines 45° in the direction of the tensile load. The fracture angle—the angle between the stress axis and the fracture

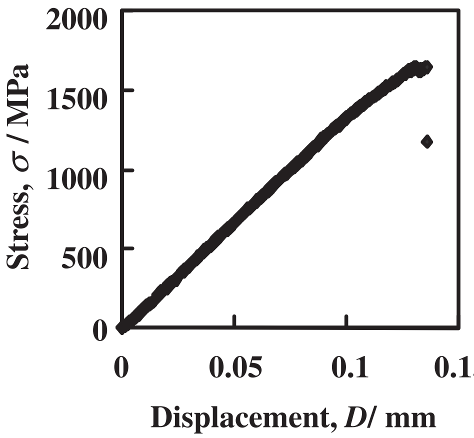

Fig. 4 Stress-displacement curve obtained from a tensile test on the fully amorphous specimen.

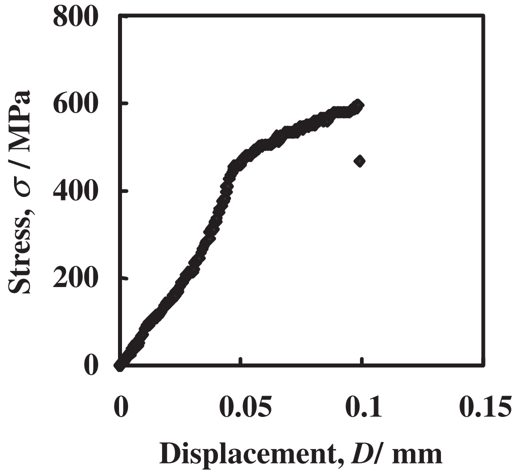

Fig. 6 Stress-displacement curve obtained from a tensile test on partially crystallized specimen.

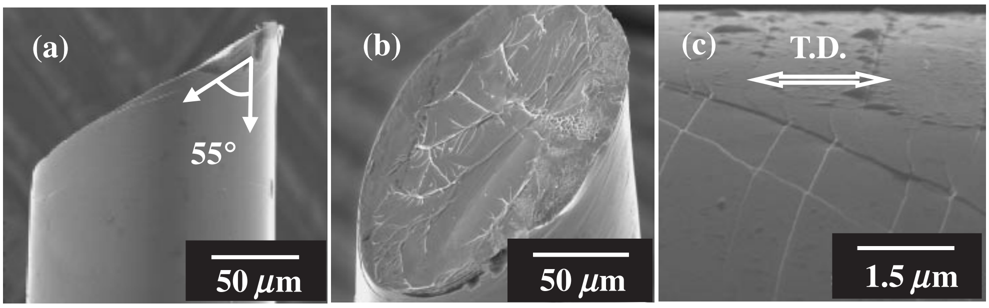

Fig. 5 Scanning micrographs of the fracture specimen having a fully amorphous phase. (a) Side view; (b) fracture surface; (c) multiple thin shear bands generated from a shear band on the specimen surface. T.D.: tensile direction.

plane—was 55°, which is consistent with previous observations of other metallic glasses under tensile deformation.[7] Vein patterns and smooth surface were clearly observed on the fracture surface, as shown in Fig. 5(b). Many thin shear bands generated from a shear band were observed on the side surface of the fractured specimen, as shown in Fig. 5(c). The generation of these thin shear bands caused a slight plastic deformation. These results are consistent with those of previous studies of Zr-based bulk metallic glasses[8–10] indicating that electrolytic polishing can be used to fabricate the Zr-based metallic glass specimens used in this study.

3.3 Tensile properties of micro-sized specimen containing dendrites

Figure 6 shows the stress-displacement curve of the tensile test on the partially crystallized specimen with a diameter of 69 μm. The yield and fracture strengths were 460 and 600 MPa, respectively, which are much lower than the strengths of the fully amorphous sample and nearly identical to the strengths of crystalline materials. After yielding, a large amount of plastic deformation and apparent work hardening were observed. This work hardening behavior is considered to originate in the crystalline particles because amorphous alloys are non-strain hardening materials. This specimen had a fracture angle of 55° and exhibited slight necking, as shown in Fig. 7(a). Vein patterns and brittle fracture region were observed on the fracture surface, as shown in Fig. 7(b). A crystalline particle likely existed at the brittle fracture region and became the starting point of the fracture. After fracturing, multiple shear bands were observed on the side surface of the specimen, as shown in Fig. 7(c).

Metallic glass specimens loaded under tension exhibit essentially no ductility and fail catastrophically by the propagation of a single shear band with plastic strain of 0.1% or less. Fracture stress of the crystallized specimen in present study was much lower than that of fully amorphous one, whereas the tensile ductility was improved by existence of crystalline phase. The enhanced tensile ductility of BMG containing micro-sized dendrites was also reported by C.C. Hays *et al.*[2,3]

In this study, the specimen fabricated from partially crystallized sample was 69 μm in a diameter and the dendrites were about half of the diameter. Therefore, it is considered that the specimen could not adequately reflect the normal stress condition that would be felt by a bulk specimen. The deformation of this specimen in consideration of size effects should be analyzed. A more detailed analysis of the interactions between the amorphous matrix and a crystalline particle is to be performed in the future.

4. Conclusions

We successfully fabricated micro-sized tensile specimens

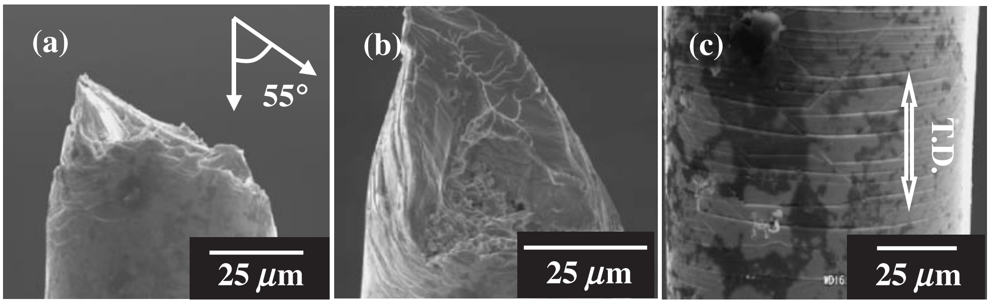

Fig. 7 Scanning micrographs of the fracture specimen containing dendrites. (a) Side view; (b) fracture surface; (c) multiple shear bands on the specimen surface. T.D.: tensile direction.

from $Zr_{55}Al_{10}Ni_5Cu_{30}$ metallic glass using electrolytic polishing technique. Micro-sized BMG tensile specimens were fabricated by electrolytic polishing from two kinds of $Zr_{55}Al_{10}Ni_5Cu_{30}$ samples. One specimen had a fully amorphous phase and the other contained dendrites within amorphous matrix. Tensile tests were performed on both specimens. The fully amorphous specimen exhibited a fracture strength of 1,640 MPa and slight plastic deformation. Multiple thin shear bands generated from a shear band were observed on the surface of the fractured specimen. The yield and fracture strengths of the specimen containing dendrites were 460 and 600 MPa, respectively. After yielding, the fractured specimen exhibited plastic deformation and work hardening as well as slight necking deformation. A brittle fracture region was observed on the fracture surface of the partially crystallized specimen. After fracturing, multiple shear bands were observed on the side surface of the specimen.

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