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Tensile properties and fracture reliability of a glass-coated Co-based amorphous microwire

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Abstract: $Co_{68.15}Fe_{4.35}Si_{12.25}B_{15.25}$ (at%) amorphous microwires with a smooth surface and a circular cross-section were fabricated by the glass-coated melt spinning method. Their mechanical properties were evaluated through tensile tests of the glass-coated amorphous microwires, and their fracture reliability was estimated using two- and three-parameter Weibull analysis. X-ray diffraction and transmission electron microscopy results showed that these glass-coated Co-based microwires were mostly amorphous. The coated Co-based microwires exhibit a tensile strength of 1145 to 2457 MPa, with a mean value of 1727 MPa and a variance of 445 MPa. Weibull statistical analysis showed that the tensile two-parameter Weibull modulus of the amorphous microwires is 4.16 and the three-parameter Weibull modulus is 1.61 with a threshold value as high as 942 MPa. These results indicate that the fabricated microwires exhibit good tensile properties and fracture reliability, and thus appear to be good candidates for electronics reliability engineering applications.

Keywords: cobalt alloys; amorphous alloys; glass; fracture; tensile testing

1. Introduction

Amorphous ferromagnetic microwires have aroused much interest for their potential use in a range of scientific research and engineering applications, including the giant magneto-impedance (GMI) effect, magnetic bistability, microwave absorption, electromagnetic interference (EMI) shielding [1–5]. Compared with their counterparts, amorphous glass-covered microwires exhibit more interesting magnetic properties, and their use in several sensors and electronic devices has been proposed [6–9]. The microstructure and magnetic properties of these rapidly solidified fine microwires can be tailored during their fabrication process or after their fabrication via annealing and mechanical processes [10–16]. A maximum magneto-impedance, $\Delta Z/Z \approx$ 600%, was reported for glass-coated $Co_{68.25}Fe_{4.5}Si_{12.25}B_{15}$ amorphous microwires when measured at a frequency of 15 MHz after the microwires were subjected to Joule annealing under an applied tensile stress [17]. Annealing under the applied tensile stress was proposed to induce magnetic anisotropy perpendicular to the wire axis of glass-coated $Co_{68.25}Fe_{4.5}Si_{12.25}B_{15}$ amorphous microwires and to lead to an enhancement of their magnetic properties. A substantial improvement in the GMI effect in Co-rich melt-extracted amorphous microwires was also achieved via an external axial tensile stress because of the rearrangement of the domain wall induced by tensile stress and the increase in circular anisotropy and permeability [18].

Mechanical investigations of such amorphous microwires are important for enhancing their magnetic properties and for solving the problems related to their use in microelectromechanical systems. To our knowledge, the mechanical properties of these glass-coated amorphous microwires have not been adequately investigated. Earlier reports on the mechanical properties of glass-covered microwires were mostly devoted to determining the tensile strength and Young's modulus of Fe-rich and stainless steel microwires [19–21]. High tensile strength (approximately 3900 MPa) was observed in amorphous $Fe_{77.5}Si_{7.5}B_{15}$

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microwires [22]. However, the deformation behaviors as well as the fracture reliability of microwires are not well understood. The aim of this investigation was to fabricate high quality Co-based glass-covered amorphous microwires and to investigate the mechanical properties of these amorphous microwires when they are subjected to uniaxial tensile deformation. The fracture reliability was also evaluated using two- and three-parameter Weibull distributions.

2. Experimental procedure

The glass-coated microwires of a nominal composition $Co_{68.15}Fe_{4.35}Si_{12.25}B_{15.25}$ (at%) were fabricated by a rapid solidification method through two steps. The master alloy ingot was prepared by arc-melting a mixture of pure Co (99.99%), Fe (99.99%), Si (99.999%), and B (99.9%) in a Ti-gettered argon atmosphere, and a CoFeSiB alloy rod with a diameter of 6 mm was subsequently obtained by suction casting. The polycrystalline master alloy rod was then placed into a special quartz glass tube with a transition temperature close to the melting point of the ingot, and the rod was remelted using a high-frequency-powered induction coil. The glass tube itself became viscous under the heat of the molten alloy, and the inner metallic alloy was drawn together with the glass shell. The drawing speed was constant at 24.5 m/min. The wire, whose diameter ranged from 20 to 30 μm, was formed after the molten glass coating had cooled. All the processes in the second step were conducted on a custom-built glass-coated spinning apparatus, as schematically shown in Fig. 1(a). Fig. 1(b) shows the macromorphology of the as-cast glass-coated wires, which have smooth surfaces and cross-sections with high roundness (Figs. 1(c) and 1(d)). Fig. 1(d) provides a clear view of the microwire, which is composed of a distinct metallic core and a glass coating. Also, the Taylor-Ulitovski technique used to prepare these wires was highly effective in achieving uniform evenness of the metallic core and glass coating, as well as good bonding between them. All the micromorphology and fracture surfaces of the wires after they were subjected to tensile testing were observed using a field-emission scanning electron microscope (SEM, Helios Nanolab600i). The microstructural characteristics of the wires were examined by X-ray diffraction (XRD); the diffraction patterns were obtained on a D/max-rb diffractometer equipped with a Cu K_α radiation source. The microstructure evolution of the glass-coated wires was identified using transmission electron microscopy (TEM, Tecnai G2 F30). A regularly rectangle gauge length of 10 mm was chosen for the preparation of tensile samples, which were tested on an Instron tensile tester (Instron-5500R1186) with special clamps and a load cell of 50 N, in accordance with ASTM standard D3379-75. Uniaxial tensile tests were conducted at room temperature with a constant strain rate of 4.2×10^{-4}. The engineering strain for each sample was calculated from the crosshead displacement after correction for machine compliance.

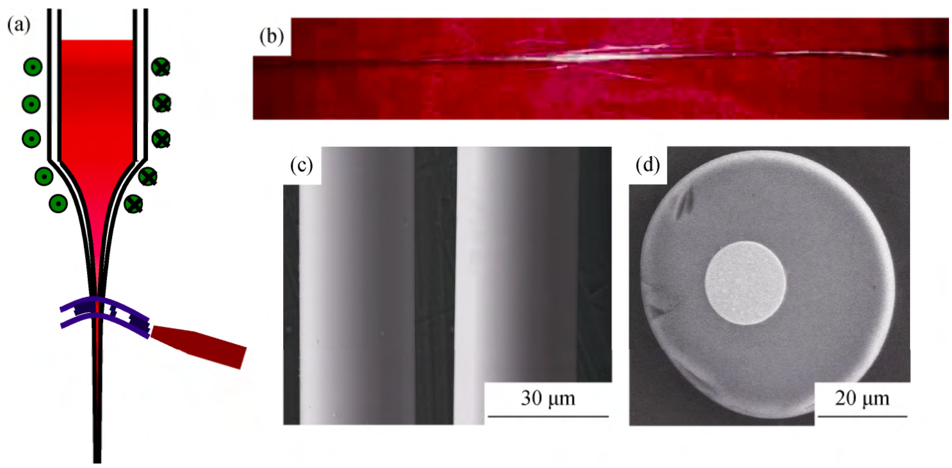

Fig. 1. Schematic illustration of the glass-coated spinning apparatus (a), optical graph of the fabricated wire bundles (b), and SEM images of a side-view (c) and a circular cross-section (d).

3. Results and discussion

3.1. Structural analyses

The intense heat transfer between a cooling medium and a liquid metal stream results in a cooling rate of a few million degrees Celsius per second, which leads to the formation of a nanocrystalline or amorphous structure during the microwire fabrication process. Fig. 2(a) shows the XRD pattern of the coated microwires. The pattern contains a single broad peak and no crystalline peaks, indicating the amorphous nature of the microwires. Fig. 2(b) gives the corresponding TEM image of the Co-based glass-coated microwires. No contrast or obvious lattice fringe was detected, demonstrating their fully amorphous nature. The high resolution transmission electron microscopy (HRTEM) image in Fig. 2(c) further confirms this claim, and the selected-area electron diffraction (SAED) pattern (inset) also consists of only a broad halo without any diffraction rings or spots. Thus, both the XRD and TEM results indicate that glass coating technology can be used to fabricate rapidly solidified Co-based amorphous wires.

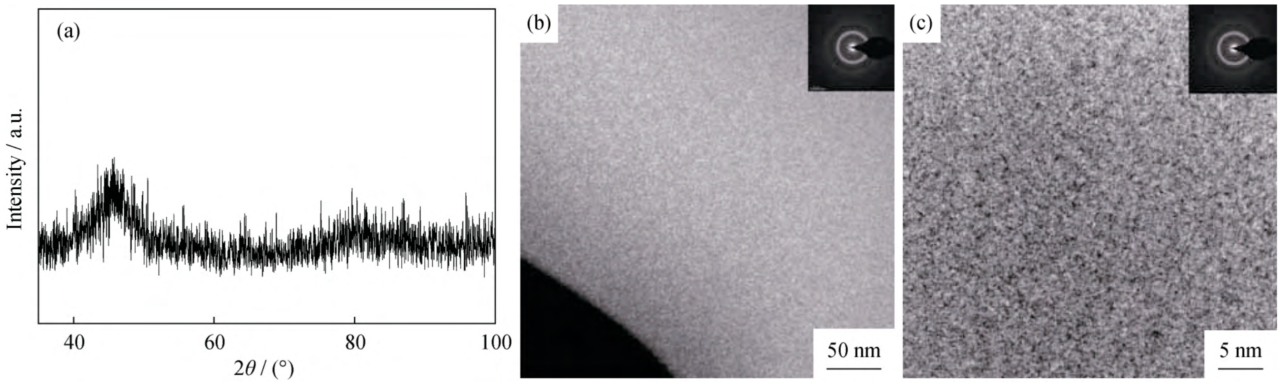

Fig. 2. Microstructural characterization of the glass-coated $Co_{68.15}Fe_{4.35}Si_{12.25}B_{15.25}$ microwires: (a) XRD pattern; (b) TEM image; (c) HRTEM image (the insets of (b) and (c) show the corresponding SAED patterns).

3.2. Tensile properties

Fifteen samples of the amorphous microwires were tested; twelve of the resulting stress-strain curves are displayed in Fig. 3. Notably, all of the tested specimens exhibited brittle behavior, and almost all the samples failed catastrophically without exhibiting any plasticity. The sudden decreases in stress-strain curves indicate the breakage of glass coatings, followed by the failures of metallic cores. The ultimate strengths of these amorphous microwires were scattered. The apparent fracture strength σ_f ranged from 1145 to 2457 MPa, with a mean value of 1727 MPa and a variance of 445 MPa. The variation in strength of these amorphous microwires results from the distribution of their strength-limiting flaws. In the case of as-cast microwires, possible flaws include the poor interface bonding strength between the glass and the metallic cores and the presence of casting pores, inclusions, or surface irregularities. Notably, the existence of these flaws results in a severe deterioration of their mechanical properties, especially the tensile strength and fracture reliability, which limit their application in microelectromechanical systems.

3.3. Weibull analysis

The strength data of brittle materials have been known to exhibit a wider degree of scatter compared to those of ductile materials, which has vital implications for the reliability of these materials in structural applications. The statistical method commonly used to describe the distribution of fracture stresses in brittle materials is given by Weibull [23]. The cumulative probability function of the Weibull distribution is expressed as follows:

$$P_f = 1 - \exp\left[\int_V \left(\frac{\sigma - \sigma_\mu}{\sigma_0}\right)^m \mathrm{d}V\right] \quad (1)$$

where P_f is the probability of failure at a given uniaxial stress or lower, which can be calculated using the equation $P_{f,i} = \frac{i-0.3}{N+0.5}$ [24], where N is the total number of tested samples and i is the sample ranking in ascending order of failure stress; the threshold value σ_μ is the value below which no specimen is expected to fail; σ_0 refers to a characteristic strength defined as the stress at which P_f is 63.2%; m is a parameter known as the Weibull modulus; and V is the volume of the tested samples.

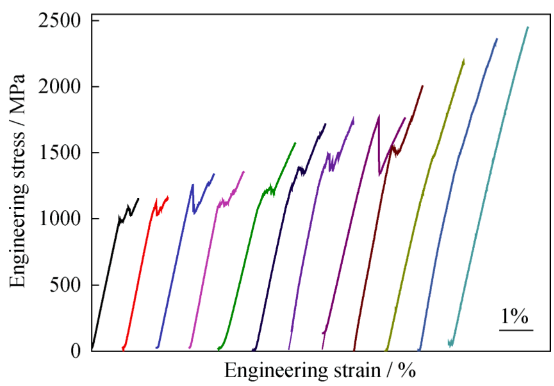

Fig. 3. Tensile stress-strain curves of the studied glass-coated Co-based amorphous microwires.

After rearrangement, Eq. (1) can be written as

$$\ln\left[\ln\left(\frac{1}{1-P_f}\right)\right] = m\ln(\sigma - \sigma_\mu) - m\ln\sigma_0 \quad (2)$$

When $\sigma_\mu = 0$, the distribution becomes the two-parameter

Weibull distribution:

$$\ln\left[\ln\left(\frac{1}{1-P_\text{f}}\right)\right] = m\ln\sigma - m\ln\sigma_0 \quad (3)$$

The Weibull modulus *m*, threshold value σ_μ, and characteristic strength σ_0 can thus be obtained by fitting experimental data ($\ln(\sigma_{\text{f},i})$, $\ln\left[\ln\left(\frac{1}{1-P_{\text{f},i}}\right)\right]$) using two- and three-parameter methods.

Fig. 4 shows the Weibull plots in the fashion suggested by Eqs. (2) and (3) for the glass-coated Co-based microwires. The data fits yield the following estimated parameters that describe the distributions:

Two-parameter: $m = 4.16$, $\sigma_0 = 1910$ MPa;

Three-parameter: $m = 1.61$, $\sigma_\mu = 942$ MPa, $\sigma_0 = 915$ MPa.

Despite the advantageous mechanical and functional properties of various amorphous alloys, the flaw/damage tolerance and reliability of amorphous metallic materials have long been ignored; some attention has only recently been paid to this perspective. The *m* value calculated in the present work is larger than that of brittle glass, similar to that of A707 steel welds [25], but smaller than that of Zr-based [26] and Mg-based [27–28] monolithic amorphous alloys under compression. The Weibull modulus *m* actually reflects the reliability of the tested samples: a greater value of *m* represents a narrower dispersion of the fracture strength and, hence, greater reliability. Notably, most of the monolithic samples generally fail under compression loading conditions via the generation of shear bands, which are probably insensitive to the sample flaws. In the present case, the samples failed under tension; however, other failure modes, such as crack opening (mode I or mixed mode), are possibly operative. Consequently, the reliability calculated on the basis of tensile-test data is probably overoptimistic. As shown in Fig. 4 (the solid line), the fitting based on the three-parameter model follows the trend of the data much better than the fitting achieved using the two-parameter model.

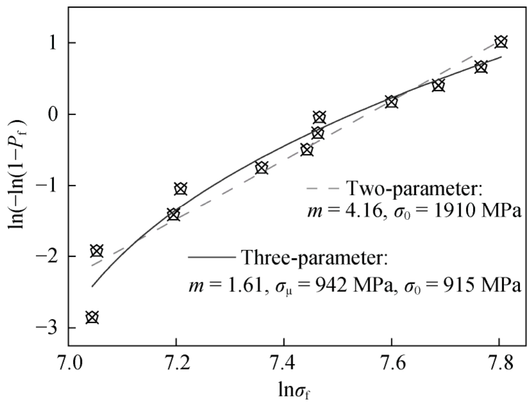

Fig. 4. Weibull plots of the tensile strength of the amorphous Co-based amorphous microwires.

3.4. Fracture morphology

Figs. 5(a) and 5(b) illustrate the two fracture morphologies

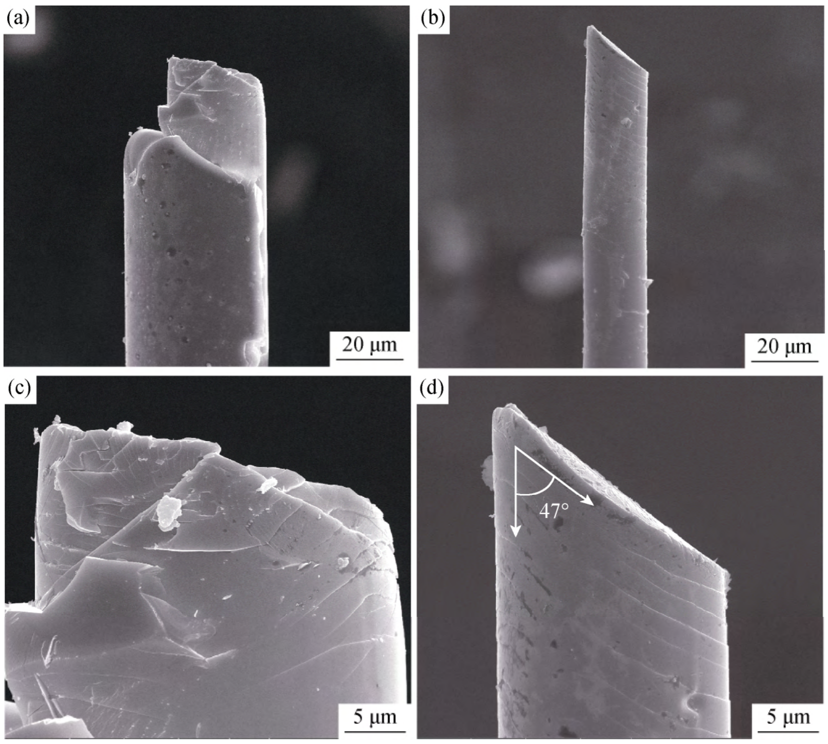

Fig. 5. Fracture morphologies of glass-covered amorphous microwires with diameters of 30 (a) and 20 (b) μm; images (c) and (d) show the magnified versions of images (a) and (b), respectively.

observed in the samples with diameters of 30 and 20 μm, respectively. The angle between the fracture surface and stress axis varies from 68° to 47° when the diameter of the metallic core decreases from 30 to 20 μm. A large number of pronounced shear bands, which are typical for the fracture feather of amorphous alloys, are observed on both sides of the lateral fracture surface, and the shear zone runs parallel with the fracture surface in the case of the small-sized sample, as shown in Figs. 5(c) and 5(d). However, even in the case of samples with significant shear banding, no obvious tensile plasticity was detected in their stress-strain curves, thereby indicating the brittle fracture by analogy to other monolithic amorphous alloys.

4. Conclusions

In the present work, highly uniform and circular $Co_{68.15}Fe_{4.35}Si_{12.25}B_{15.25}$ amorphous microwires were fabricated using a Taylor–Ulitovsky technique, and the tensile properties and fracture reliability were systematically investigated. These glass-coated Co-based microwires exhibit a tensile strength of 1145 to 2457 MPa with a mean value of 1727 MPa. The tensile Weibull modulus of the amorphous microwires is 4.16, and the threshold value is 942 MPa, indicating their high tensile property and fracture reliability. The high mechanical strength and fracture reliability of these microwires, together with their excellent reported magnetic properties, make them attractive for use as miniaturized components in electronics for magnetic recording, biomedical implantation, etc.

Acknowledgements

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