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# Deformation-strengthening during rolling Cu<sub>60</sub>Zr<sub>20</sub>Ti<sub>20</sub> bulk metallic glass

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## Abstract

Mechanical strength evolutions during rolling the  $Cu_{60}Zr_{20}Ti_{20}$  bulk metallic glass (BMG) at room temperature (RT) and cryogenic temperature (CT) have been investigated by measuring the microhardness. The hardness slightly increases during the initial rolling stage as a result of the gradually enhanced microinhomogeneity of chemical composition, and then dramatically rises owing to phase separation at CT or phase separation plus nanocrystallization at RT. It is revealed that the Cu-rich separated amorphous phases from the matrix possess higher strengths than the original and Cu-poor separated amorphous phases. As the deformation-induced nanocrystallites contain lots of crystal defects, their resistance to yielding is deteriorated. Consequently, as partial phase-separated regions crystallize during RT-rolling, the increase rate of microhardness slows down as compared with that in CT-rolling. It is proposed that phase separation may be a more effective way to strengthen the BMG than the incorporation of the nanocrystallites with crystal defects.

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# 1. Introduction

Without long-range translational symmetry in the atomic assembly and crystal defects, such as grain boundaries and dislocations, bulk metallic glasses (BMGs) exhibit many unique properties, including excellent corrosion resistance, remarkably high strength and hardness, and large elastic deformation limit [1–4]. However, their low ductility and brittle fracture at temperatures far below the glass transition temperature extremely limit the application as structural materials [5]. A localized shear occurs as soon as stresses exceed the yield strengths of metallic glasses, and the unrestricted rapid propagation of shear bands leads to a catastrophic failure [6]. In order to improve the ductility and strength, heterogeneous microstructures have recently been designed by combining glass matrix with crystalline second phase particles. The second phase in metallic glasses can be introduced by partial nanocrystallization of the amorphous precursors [7], in situ crystallizing the primary den-

0921-5093/\$ - see front matter © 2006 Elsevier B.V. All rights reserved. doi:10.1016/j.msea.2006.12.025 dritic bcc phase in the solidification [8,9], or directly adding crystalline particles in glass-forming melts [10]. Owing to the interaction with the crystalline phase, shear bands are multiplied and branched, and their rapid propagation is inhibited. Besides, crystalline/amorphous composites can also be produced by plastically deforming the metallic glasses, namely mechanically driven crystallization [11]. While nanocrystals precipitate from the mother glass owing to plastic deformation, different from other crystallization processes, shear bands are also created in the glass matrix. Considering that introduction of shear bands in metallic glasses generally decreases the strength [12], but nanocrystallization improves it [13], the variation of mechanical properties of metallic glasses with plastic deformation may be very sophisticated, and cannot be predicted without sufficient experiments. In addition, the changes of free-volume content and phase transformation during plastic deformation are closely correlated with the deformation temperature [5,14–18], which makes the evolution of the mechanical property become more complicated. Recently, while investigating the microstructure evolutions of the Cu<sub>60</sub>Zr<sub>20</sub>Ti<sub>20</sub> BMG during the rolling at room temperature (RT) and cryogenic temperature (CT) [17], the present authors have found that the rolling deformation at

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CT leads to the occurrence of only phase separation, but phase separation plus nanocrystallization takes place in the RT-rolling when the thickness reduction exceeds a critical value. Therefore, it is very interesting to reveal how the mechanical properties change during such deformations.

#### 2. Experimental procedures

The master alloy ingot with nominal composition of Cu<sub>60</sub>Zr<sub>20</sub>Ti<sub>20</sub> was prepared by arc melting the high purity Cu (99.99%), Zr (99.9%) and Ti (99.9%) under a Ti-gettered argon atmosphere. The ingot was inverted and remelted six times to ensure its compositional homogeneity and then suck-cast into a water-cooled copper mold to produce a 40 mm long cylindrical rod with a diameter of 2 mm. The amorphous structure was ascertained by X-ray diffraction (XRD) and high-resolution transmission electron microscopy (HRTEM). The rods were cut using a diamond saw into short cylinders with a thickness of 1.5 mm for rolling. Both ends of the cylinders were mechanically polished to make them parallel to each other prior to the rolling experiment. Details of RT-rolling and CT-rolling procedure were described elsewhere [17]. The degree of deformation was denoted by the reduction in thickness  $\varepsilon = (h_0 - h)/h_0$ , where  $h_0$  and h represented the specimen thicknesses before and after rolling, respectively. Many small deformation passes were used with progressively narrowing the gap between two rollers, and the decrease of the gap during the deformation was carefully controlled so that the strain rate was about  $5.0 \times 10^{-3} \text{ s}^{-1}$ .

The microstructures of the as-rolled specimens were examined by Philips PW1820 X-ray diffractometer with monochromatic Cu K $\alpha$  radiation and JEOL JEM-3000F HRTEM instrument operating at 300 kV. The difficulty in preparing a good TEM specimen of the Cu–Zr–Ti alloy is well known just as the previous studies on this alloy system have pointed out that a nanocrystalline microstructure might form when the specimen was prepared without special attention [19]. To prevent the damage caused by the ion milling at RT and the contamination from electro-chemical twin-jet polishing, the thin foil specimens for TEM were prepared by low-energy ion milling at 2.5 keV and 5 mA with liquid nitrogen cooling. The specimens were observed in TEM immediately after the preparation since the Cu–Zr–Ti thin foil readily oxidizes upon exposure in air atmosphere.

A Perkin-Elmer Pyris Diamond differential scanning calorimeter (DSC) was used to isothermally anneal the specimens into different crystallization degree under a flow of purified argon, during which the specimens were heated at 20 K/min in a flowing argon atmosphere up to 708 K and held for various times, and then rapidly cooled to room temperature. The crystallized fraction in the annealed specimens was determined by reheating them in the DSC at 20 K/min to the completion of crystallization.

The microhardness of the rolled and the annealed specimens was measured by a Leitz Durimet Vickers hardness tester that consisted of a square-based pyramidal diamond indenter with a  $136^{\circ}$  angle between opposite faces of the indenter. The static loads were 50, 100 and 200 g, respectively, and the dwell time



RT-rolled specimen

-rolled specimen

RT-rolled specimen

40

Ī

20

80

100

60

ε (%)

Fig. 1. Microhardness of the RT-rolled and CT-rolled  $Cu_{60}Zr_{20}Ti_{20}$  specimens as a function of  $\varepsilon$  at a load of 200 g using the substrate material of quenched carbon steel (a) and Fe–10Cr–3Al–3Si stainless steel (b).

of loading was 15 s. The substrate material used for sustaining the specimens during the measurement was a quenched Fe–0.45 wt.% C steel. Twenty indentations were made for each specimen.

### 3. Results and discussions

#### 3.1. Microhardness of as-rolled specimens

The as-cast Cu<sub>60</sub>Zr<sub>20</sub>Ti<sub>20</sub> rods have been verified by XRD and HRTEM to be fully amorphous [17]. By carefully controlling the strain rate, a maximum thickness reduction of the BMG as high as 97% was achieved at both RT and CT. The specimens with the highest deformation degrees have no cracks, and remain ductile, as shown by the  $180^{\circ}$  bending without fracture. Fig. 1(a) shows the microhardness  $H_{\rm v}$  of the rolled specimens with different  $\varepsilon$  at a load of 200 g. The results show that their variations with  $\varepsilon$  are quite similar and can be divided into two stages: a slow increase from 5.89 GPa for the as-cast specimen to 5.99 GPa or so for the CT-rolled specimen with  $\varepsilon = 89\%$  and the RT-rolled specimen with  $\varepsilon = 87\%$ , and a rapid increase up to 6.33 and 6.27 GPa for the CT-rolled and RT-rolled specimens with the highest  $\varepsilon$ , respectively. It is observed that the hardness of the RT-rolled specimen is slightly higher than that of the CTrolled specimen with the same  $\varepsilon$  when  $\varepsilon$  is less than 96%, while

6.4

6.2

6.0

5.8

6.4

6.2

6.0

0

H<sub>,</sub> (GPa)

(a)

H (GPa)

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