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# Cooling rate induced radial gradient structure in cylindrical metallic glasses



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#### ABSTRACT

The mechanical property dependence of a typical  $Zr_{52\cdot5}Cu_{17.5}Ni_{14\cdot6}Al_{10}Ti_5$  metallic glass on micro-structure has been investigated using nanoindentation. Heat flow equation was chosen to study cooling rate and the microstructure during the casting process. Results show that cylindrical sample with larger dimension possesses a lower cooling rate, which leads to larger elastic modulus and higher hardness accordingly, and so is the center region compared to boundary in the cross section of a cylindrical specimen. To a certain extent, cylindrical metallic glasses are radial gradient structure induced by the cooling rate in a small range.

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#### 1. Introduction

As a rule, metals and alloys exist in crystalline state. It was first discovered that rapid solidification of the melt resulted in the appearance of alloys with the amorphous structure in 1960, from which metallic glasses (MGs) have emerged in our view [1]. Different from their crystalline counterparts, the plastic deformation of metallic glasses is highly localized into shear bands (SBs) within a nanometer scale zone, while the SBs are preferential sites for further deformation leading to the final fracture [2,3]. The nucleation of shear bands is determined by the free volumes produced during the rapid cooling [4–6], since there is no sufficient time for ordered arrangement of atoms. A promising new way of enhancing the plasticity by introducing large amount of randomly distributed free volumes into MGs with a high cooling rate has been proposed [7]. The surface softening in metallic glasses may be dependent on the excess freezing free volumes near the surface compared to the interior during the solidification [8,9]. Y. Meng systematically studied the heating methods and the cooling rate by both experimental and numerical ways and found that the solidification process have great

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effects not only on the crystalline structure but also on the thermal stability of the amorphous phase [10]. Investigation on the free volumes is of great help to reveal the mechanical properties and deformation mechanism of metallic glasses. Enlightened by this point, the aim of the current work is to study the correlation between cooling rate and the mechanical parameters using nanoindentation. Moreover, the micro-structure dependence of free volumes will be explored.

#### 2. Materials and methods

Cylindrical ingots of Zr<sub>52.5</sub>Cu<sub>17.9</sub>Ni<sub>14.6</sub>Al<sub>10</sub>Ti<sub>5</sub> (Vit105) had been prepared by melting of the raw metals according to the method used in our previous work [10,11]. The disk samples, with three different diameters (i.e., 2 mm, 3 mm, and 6 mm), were cut from the ingots which behaved amorphously for sure with a thickness of 2 mm respectively. Nanoindentation tests were performed on the Agilent Nano-indenter G200 test system with a triangular pyramid Berkovich diamond indenter at room temperature. Strain rate control mode of 0.05 s<sup>-1</sup> with a depth limitation of 2000 nm and dwell period of 10 s were carried out at points with radius of r = 0,  $r = 1/5r_0$ ,  $r = 2/5r_0$ ,  $r = 3/5r_0$ ,  $r = 4/5r_0$  on three samples respectively, where  $r_0$  is the sample radius. The tested values were averaged from at least eight points positioned by a rotational increment of 45° around the disk center.

In Oliver and Pharr's nanoindentation model [12], the contact area,  $A_c$ , is defined by a polynomial function ( $A_c = \sum_{n=0}^{8} C_n h_c^{\frac{1}{n-1}}$ ) of the contact

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Fig. 1. (a) Elastic modulus and (b) hardness versus the distance from disk center to boundary by nanoindentation.

depth,  $h_c$ , which is given by:

$$h_c = h_{\max} - \varepsilon \frac{P_{\max}}{S} \tag{1}$$

where  $P_{\text{max}}$  is the peak load,  $h_{\text{max}}$  is the displacement at the maximum load,  $\varepsilon$  is a constant equal to 0.75 and *S* is the contact stiffness. Based on the continuous stiffness measurements technique, *S* can be obtained as:

$$S = \left[\frac{1}{(P_0/Z_0)\cos\phi - (K_s - mw^2)} - \frac{1}{K_f}\right]^{-1}$$
(2)

where  $P_0$  is the amplitude of the harmonic excitation force,  $Z_0$  is the response to the displacement amplitude ( $\approx 2$  nm),  $\Phi$  is the phase shift between the harmonic displacement and the harmonic excitation force,  $\omega = 2\pi f$  is the angular frequency (45 Hz), and  $K_s$ ,  $K_f$ , and m are the spring constant in the vertical direction, frame stiffness, and mass of the indenter, respectively.

For a Berkovich indenter,  $A_c$  is given by:

$$A_c = 24.56h_c^2$$
. (3)

The hardness is defined as:

$$H = {}^{P}/_{A_{c}}.$$
(4)

The reduced modulus,  $E_{\rm r}$ , is calculated from the unloading data as:

$$E_r = \frac{\sqrt{\pi}}{2\beta} \frac{S}{\sqrt{A_c}} \tag{5}$$

where  $\beta$  is a constant equal to 1.034 for a Berkovich indenter. The modulus, *E*, of the sample can easily be determined from the following relationship:

$$\frac{1}{E_r} = \frac{1 - v^2}{E} + \frac{1 - v_i^2}{E_i} \tag{6}$$

where  $E_i$  and  $v_i$  are Young's modulus and Poisson's ratio of the material and the indenter, respectively ( $E_i = 1141$  GPa and  $v_i = 0.07$ ).

#### 3. Results and discussion

Fig. 1 shows the dependence of elastic modulus and hardness on the radial position from the central region to the boundary of the disk, error bars were added to describe the average values and standard deviation of each position. For the sample with 6 mm diameter, both elastic modulus and hardness exhibit a monotonic decreasing tendency from the disk center to the exterior. The average elastic modulus reduces from 105 GPa (r = 0) to 98 GPa ( $r = 4/5r_0$ ) with ~6% decrement, while the hardness reduces from 7.25 GPa to 6.59 GPa with ~9% decrement. With regard to the 2 mm and 3 mm specimens, there is no obvious declining trend from the interior to the surface. Nevertheless, the central values are higher than those of the boundary. Furthermore, 6 mm sample has a guite higher value than that of 2 mm and 3 mm samples, this is because big rod results in low cooling rate, which is always accompanied by the decrease in free volumes. As shown in Fig. 1, it doesn't appear to be much different from each other between 2 mm and 3 mm samples, especially in hardness, suggesting that the two samples are carrying close contents of free volumes. Mechanical parameters such as hardness and elastic modulus are essentially related to the microstructure, i.e., concentration of free volumes. The difference in



Fig. 2. Temperature distribution profiles in the center region (a) and at the time of 0.15 s (b) during the heat conduction process for different radii of 1.5 mm and 3 mm, respectively.

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