



Structure relaxation effect on hardness and shear transformation zone volume of a Ni–Nb metallic glassy film

H. Chen^a, Y.X. Song^a, T.H. Zhang^a, M. Wu^b, Y. Ma^{a,*}

^a College of Mechanical Engineering, Zhejiang University of Technology, Hangzhou 310014, PR China

^b College of Materials Science and Engineering, Zhejiang University of Technology, Hangzhou 310014, PR China

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ABSTRACT

The annealing effect below glass transition temperature (T_g) on nanoindentation hardness and shear transformation zone (STZ) volume was studied in a Ni–Nb metallic glassy thin film. The indentation size effect (ISE) and loading rate effect on hardness were systematically investigated. It was found that ISE was less pronounced and strain rate sensitivity (SRS) was higher in the sample suffered longer annealing time. As annealing time reached a critical value, hardness value, ISE and SRS changed quite limited which could be due to full structure relaxation in samples. The hardness spatial distribution was more scattered with increasing annealing time, indicating a more pronounced mechanical heterogeneity at microscale. Based on SRS values and cumulative probability distribution of hardness, STZ volumes were estimated upon two distinct methods, respectively. A tight correlation between STZ volume and structure state was revealed. As increasing annealing time, STZ volume was continuously decreased and approached down to a stable value.

1. Introduction

Owning to the unique atomic configuration, metallic glass owns excellent mechanical performance, such as super large elastic limit, high strength and strong wear resistance [1]. However, the limited forming size and lack of ductility hinder the development of this new alloy on engineering application. Tremendous efforts have been made in order to enlarge the critical sample size through improving fabricating technology and developing new compositions. Though some compositions were modulated to break through the maximum size of 70 mm, their brittleness was further deteriorated [2, 3]. In recent years, size effect on mechanical properties of metallic glass has attracted numerous attentions [4–6]. As reducing sample dimension down to micrometer and/or nanometer scale, both plasticity and strength were effectively enhanced in comparison with those of bulk counterpart. Such size effect was attributed to the restriction of shear banding, which could even be replaced by homogeneous flow temporarily [7, 8]. However, the explicit mechanism for size effect in metallic glass was unclear and even controversial due to that its internal structure is actually independent on physical dimension.

Free volume and shear transformation zone (STZ) are currently the two mostly adopted modes of deformation mechanism in metallic glass [9, 10]. It is well recognized that free volume content has a strong correlation with plasticity [11]. While for STZ, being different from

structure defect, was defined by its transience, i.e., it can be only identified from the atomic structures before and after deformation. The details of STZ evolution are always studied by computer simulation, on its shape, configuration and activation mechanism [12]. STZ could also be regarded as deformation unit with a local rearrangement of atoms. Recently, experimental methods relying on nanoindentation were developed to estimate STZ volume based on the cooperative shear model (CSM) by Johnson and Samwer [13–17]. It was suggested that large STZ volume facilitates ductility in bulk metallic glasses [14].

For micro-size metallic glass, it has been revealed that sub- T_g annealing could decrease the critical sample size for deformation mode transition (localize to homogeneous) and elastic strain limit [8, 18], which could be intuitively detected by high-resolution observation technology. While the correlation between strength and structure state in micro-size metallic glass has not been fully studied. It is well known that free volume would be annihilated by thermal treatment induced structure relaxation. However, the structure relaxation effect on STZ volume is still unclear that both enlargement and reduction effects have been reported in bulk metallic glasses [13, 19]. With this in mind, a Ni–Nb metallic glassy film was prepared and annealed at sub- T_g with different durations in our study. Hardness was carefully measured relying on nanoindentation, by applying various indentation depths and loading rates. Spatial distribution of nano-hardness at micrometer-scale region was also detected upon an express testing module. The STZ

* Corresponding author.

E-mail address: may@zjut.edu.cn (Y. Ma).

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volume was calculated experimentally by rate-jump and statistical methods, respectively. Here, we aim to study structure relaxation effect on nano-hardness and STZ volume in micro-size metallic glass, for further bridging the correlation between plastic mechanism and structure state.

2. Experimental procedures

The Ni–Nb films were deposited on silicon wafer in a DC magnetron sputtering system (Kurt J. Lesker PVD75) at room temperature in pure argon gas. The 2-in. target alloy adopt in the chamber is $\text{Ni}_{60}\text{Nb}_{40}$, at.%, which was prepared from high purity (99.99 wt%) elements by vacuum casting. The target is installed at the bottom while the silicon wafer is stick on the sample platform, which is right above the target. The target-to-substrate distance is kept constant, equal to 100 mm. The base pressure of chamber was kept about 5×10^{-7} Torr before deposition and working argon pressure was set about 1 mTorr. The power on target was fixed at 200 W during the deposition. The thickness of 2 h-deposited Ni–Nb film is $2.2 \mu\text{m}$ measured by a surface profilometer (Dektak 150). The annealing treatment was performed following the deposition by a heating apparatus attached on the sample platform with argon shielding. The annealing temperature was set to be 623 K (T_g was 841 K [20]) and the durations were 30 min, 60 min, 120 min and 240 min, respectively. By means of X-ray energy dispersive spectrometer (EDS) attached on the SEM, the chemical composition of as-prepared Ni–Nb film was detected as $\text{Ni}_{59.8}\text{Nb}_{40.2}$, which is almost the same to alloy target. The amorphous nature of all the as-prepared and annealed films were confirmed by X-ray diffraction (XRD) with $\text{Cu } K_\alpha$ radiation.

Nanoindentation experiments were conducted at constant temperature of 20 °C on Agilent Nano Indenter G200 with the dynamic contact module (DCM), by which higher resolution in both force and displacement and less sensitivity to the environmental can be attained. Hardness measurements were conducted at displacements of 50 nm, 100 nm, 150 nm, 200 nm and 250 nm for all the samples with a constant loading rate of 0.2 mN/s. Nano-hardness was also measured at a constant maximum load of 5 mN with four different loading rates, respectively were 2.5 mN/s, 0.5 mN/s, 0.1 mN/s and 0.05 mN/s. More than 16 measurements were conducted in each test, in order to reduce the systematic errors in the data. Furthermore, spatial distribution of nano-hardness was detected by express testing (about one second for one measurement) with a maximum load of 5 mN on the area of $100 \mu\text{m} \times 100 \mu\text{m}$, in which 625 tests (25×25 matrix with $4 \mu\text{m}$ spacing) were conducted uniformly. The distance between two adjacent testing positions was roughly 25 times of the pressed depth. For each sample, the express testing was performed at 5 different areas. It's worthy mention that surface roughness of metallic glassy film is extremely low ($R_a \sim 0.5 \text{ nm}$ for Ni–Nb) [20], which could guarantee the nano-hardness mapping test is correct.

3. Results and discussion

3.1. Indentation size effect

Fig. 1(a) shows the typical P - h curves of as-cast and 240-mins annealed samples at maximum depths of 50 nm, 100 nm, 150 nm, 200 nm and 250 nm. Clearly, higher load was required to reach the same displacement after annealing. Fig. 1(b) exhibits the variation trend of nano-hardness with pressed depths for all the samples. Nano-hardness was effectively enhanced by annealing, which could be due to free volume annihilation, atomic arrangement changes and release of tensile residual stress during structure relaxation [21–23]. As the annealing time was beyond 60 mins, hardness increase was quite limited that there was no visible difference between 120 mins-annealed and 240 mins-annealed samples. It is suggested structure relaxation could be fully occurred as annealing duration reaches a critical value under T_g .

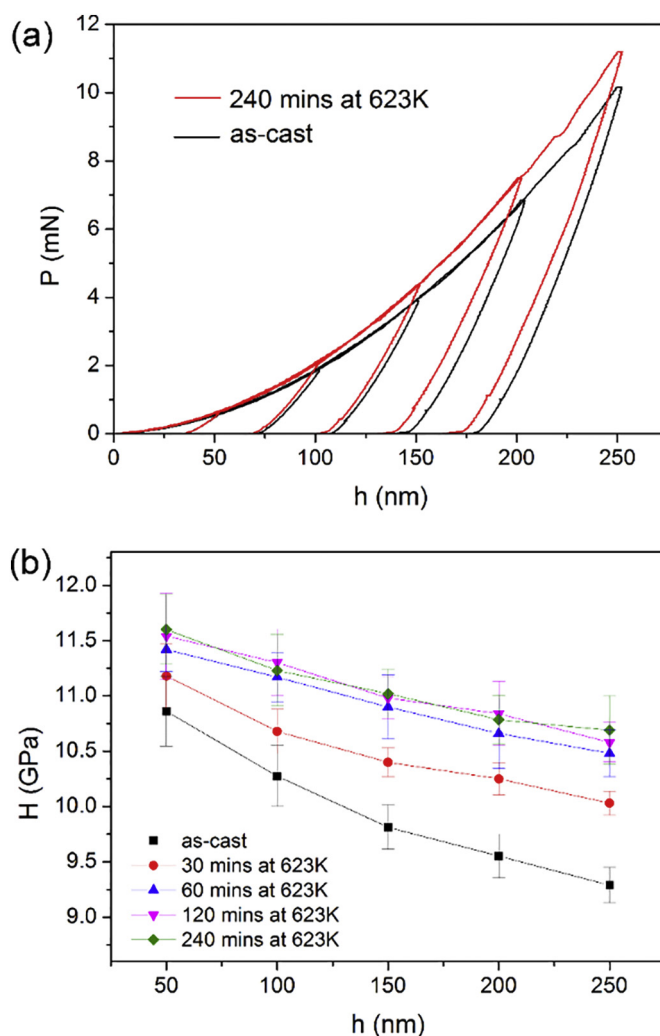


Fig. 1. (a) Typical P - h curves at various peak loads with a constant loading rate of 0.2 mN/s for as-cast and 240 mins annealed samples; (b) Nano-hardness at various displacements for all the samples.

In Dmowski et al.'s report, the nano-hardness of a Zr-based metallic glass quickly tended to a stable value after 10 mins annealing, of which temperature 40 K below T_g [24]. Apparently, hardness enhancement would be more precipitously with annealing time as adopting higher sub- T_g annealing temperature, which facilitates the process of structure relaxation in metallic glass.

The well-known indentation size effect (ISE) could be clearly observed that nano-hardness was evidently decreased with increasing pressed depth in all the as-cast and annealed samples. Though it was commonly appeared in crystalline alloys, it was also reported that ISE could occur in metallic glasses [25]. And several mechanisms concerned with plastic deformation have been proposed to explain the ISE phenomenon in metallic glasses, e.g., low defects at small plastic zone and/or more excess free volume generated at deep pressed depth, though it was not fully understood [25]. Qualitatively, shear banding propagation would be more sufficient and induce soften effect on hardness at larger pressed depth. In the present study, ISE was less pronounced in the annealed samples than that in as-cast one. From the perspective of deformation mode, shear banding would be suppressed at small length scale and the critical size for deformation mode transition was $\sim 50 \text{ nm}$ in as-cast Ni–Nb film according to the author's previous work [7]. While in annealed metallic glassy films, localized deformation was easy to occur that much smaller critical length scale was required for the deformation mode transition from shear banding

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