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# Loading rate effect on the creep behavior of metallic glassy films and its correlation with the shear transformation zone

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ABSTRACT

The room temperature creep behaviors of metallic glassy films with a series of loading rates were systematically investigated by a spherical-tip nanoindentation. The results show that the role of loading rate on the creep deformation is material-dependent and can be intrinsically related to the shear transformation zone (STZ). Smaller STZs facilitate the more dependency of creep on the loading rate and, interestingly, a rate independency was observed in Ni–Nb, whose STZ volume is particularly larger than those in other samples. The deformation mechanism of the creep flow is discussed in the light of STZs combined with extra free volume, providing detailed explanation for the creep diversity on the loading rate in different metallic glass systems.



## 1. Introduction

Metallic glass, which is relatively a new member of glass family, is scientifically defined as amorphous alloy that has a non-crystalline but short-range order structure [1–3]. Due to the unique atomic configuration and the corresponding excellent mechanical properties [4–6], metallic glass is widely used as an important part of model system in the condensed matter physics researches and has a great potential to be utilized as an engineering material. However, the limited size and brittleness are currently the two critical bottlenecks that hinder the development of metallic glass for commercial applications [7,8]. In the last decade, a strong size effect was validated in both simulation and experiment to enhance the plasticity in the micro/nano-sized metallic glasses, whilst the shear banding event can be avoided within a large plastic strain [9–11]. As a consequence, it attracts numerous attentions on the metallic glass with its form of nano-pillar or film for the purpose of revealing the underlying mechanism of the plastic deformation modes (localized deformation and homogeneous flow) at room temperature [12–15]. Nevertheless, the studies of time-dependent plastic deformation also referred to creep at the micro/nano-sized samples are quite limited [16–19]. Nanoindentation is the most powerful technology to study the mechanical properties and the deformation process of small-sized materials [20]. Compared to the traditional uniaxial compression or tension test, it is more accurate and time-saving to study creep behavior by the nanoindentation technique. On the other hand, the stress distribution underneath the indenter is more complex and severe plastic deformation always occurs before the holding stage, hence metallic glasses with high $T_g$ can creep even at room temperature [19,21]. Furthermore, the creep behavior of metallic glass in the nanoindentation is difficult to predict and susceptible to the experimental conditions (e.g., imposed initial strain or indenter size). Though the deformation mechanism in metallic glass is under debate, the loading rate effect on the plastic deformation has been studied in detail [22–25]. While for the time-dependent plastic deformation, the role of loading rate was always qualitatively discussed and simply attributed to the free volume. From the scientific point of view, characterization of loading rate effect on the creep deformation merits more lucubrate. With this in mind, four metallic glassy thin films with distinct physical and mechanical properties are selected in this work and creep tests are performed upon four loading rates which span two orders of magnitude. We aim to reveal the inner connection between the creep behavior and the loading rate for the small-sized metallic glasses.

## 2. Experiment

The metallic glassy thin films (La–Co–Al, Cu–Zr–Al, Ni–Nb and W–Ru–B) were deposited on silicon wafer in a DC magnetron sputtering system (Kurt J. Lesker PVD75) at room temperature in pure argon gas. The alloys of 2-inch targets adopted in the

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chamber respectively are $La_{55}Co_{20}Al_{25}$, $Cu_{45}Zr_{48}Al_7$, $Ni_{60}Nb_{40}$ and $W_{46}Ru_{37}B_{17}$, which were prepared from high purity (99.99%) elements by vacuum casting. The target was installed at the bottom while the silicon wafer was stuck on the sample platform, which was right above the target. The target-to-substrate distance was kept constant, equal to 100 mm. The base pressure of the chamber was kept about $5 \times 10^{-7}$ Torr before deposition and the working argon pressure was set about 1 m Torr. The power on the target was fixed at 200 W during the deposition. The thicknesses of the as-deposited thin films with various compositions are about 2000 nm by controlling the depositing time with which the film by sputtering grows linearly, albeit sputtering productivities of the four alloys are different due to the different bonding strengths and atomic weights. The thicknesses were measured by a surface profilometer (Dektak 150). By means of X-ray energy dispersive spectrometer (EDS) attached on the SEM, the chemical composition of the films can be detected. X-ray Photoelectron Spectroscopy (XPS) was further utilized to measure the composition of W–Ru–B film due to the insensitivity of B element in EDS. The amorphous nature of the samples was confirmed by X-ray diffraction (XRD) with Cu $K_\alpha$ radiation and differential scanning calorimetry (DSC, Perkin Elmer &NETZSCH) with heating rate of 20 K/min.

Nanoindentation creep experiments were conducted at constant temperature of 20 °C on Agilent Nano Indenter G200 with a spherical indenter, whose effective radius is 3.5 μm upon the calibration on standard fused silicon. The displacement and load resolutions of the machine are 0.01 nm and 50 nN, respectively. The constant load holding method was used in this work, the displacement of indenter into the surface at a prescribed load was continuously recorded. The applied maximum loads for the samples are various, 10 mN for La–Co–Al, 20 mN for Cu–Zr–Al, 25 mN for Ni–Nb and 30 mN for W–Ru–B, thus the initial strains before the holding stage for these samples are almost the same. The indenter was held for 250 s at a series of loading–unloading cycles with different loading rates 5, 1, 0.2 and 0.04 mN/s. It should be mentioned that the creep tests were carried out until thermal drift reduced to below 0.03 nm/s. Meanwhile, drift correction which is calibrated at 10% of the maximum load during the unloading process would be strictly performed. Furthermore, the reliability of the creep results was confirmed by conducting twelve independent measurements.

## 3. Results and discussion

The chemical compositions of the as-deposited films are $La_{57}Co_{18}Al_{25}$, $Cu_{44}Zr_{44}Al_{12}$, $Ni_{60}Nb_{40}$ and $W_{40}Ru_{29}B_{31}$. A little composition deviation between the film and target in W–Ru–B was inevitable due to the metalloid constituent B. All the films are fully amorphous that only a broad diffraction peak can be detected in each sample as shown in Fig. 1, as well as the DSC scans that the glass transition temperature $T_g$ and crystallization temperature $T_x$ can be clearly observed.

Fig. 2 exhibits the typical *P–h* curves of creep obtained from the tests with four different loading rates. It should be noted first that the maximum indentation depths in all the samples are about 10% of the film thickness, therefore the substrate influence is negligible. It is clear that larger indentation depth is required to reach the same load at the slower loading test for all the films, indicating that hardness decreases with the increase of loading time in this work. The corresponding creep curves during the holding stage are shown in Fig. 3, in which the displacements are plotted with the holding time. Two distinct stages can be divided for the creep curves as transient creep and steady-state creep. At the transient stage, the press displacement increases relatively fast but the creep rate drops rapidly. Then the creep displacement turns to be slowly and almost linearly increased

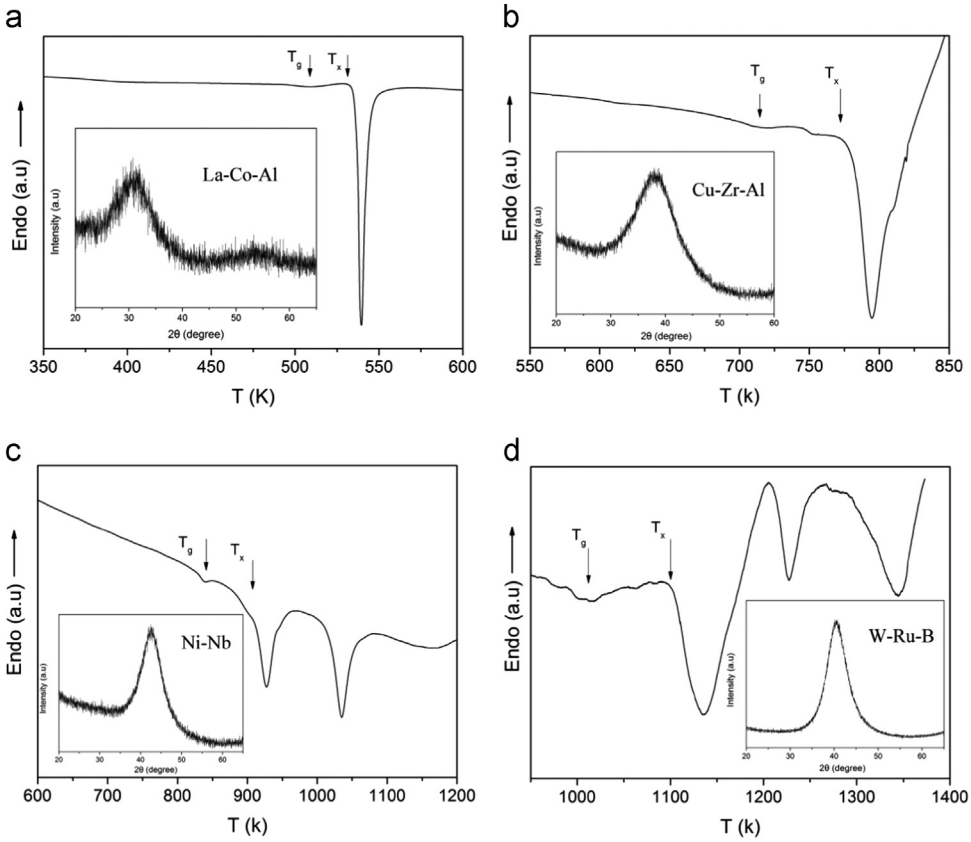


**Fig. 1.** DSC curves and XRD patterns of the La–Co–Al (a), Cu–Zr–Al (b), Ni–Nb (c) and W–Ru–B (d).