



Effect of hydrogen on the yielding behavior and shear transformation zone volume in metallic glass ribbons

Yakai Zhao^a, In-Chul Choi^a, Moo-Young Seok^a, Min-Hyun Kim^{b,c}, Do-Hyang Kim^c,
Upadrasta Ramamurty^{d,e}, Jin-Yoo Suh^{b,*}, Jae-il Jang^{a,*}

^a Division of Materials Science and Engineering, Hanyang University, Seoul 133-791, Republic of Korea

^b High Temperature Energy Materials Research Center, Korea Institute of Science and Technology, Seoul 136-791, Republic of Korea

^c Department of Materials Science and Engineering, Yonsei University, Seoul 120-749, Republic of Korea

^d Department of Materials Engineering, Indian Institute of Science, Bangalore 560012, India

^e Center of Excellence for Advanced Materials Research, King Abdulaziz University, Jeddah 21589, Saudi Arabia

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Abstract

The effect of hydrogen (H) charging on the shear yield strength (τ_{\max}) and shear transformation zone volume (Ω) of Ni–Nb–Zr metallic glass ribbons, with varying Zr content, were studied through the first pop-in loads during nanoindentation. Weight gain measurements after H charging and desorption studies were utilized to identify how the total H absorbed during charging is partitioned into mobile and immobile (or trapped) parts. These, in turn, indicate the significant role of H mobility in the amorphous structure on the yielding behavior. In high-Zr alloys, τ_{\max} increases significantly whereas Ω decreases. In low-Zr alloys, a slight decrease in τ_{\max} and increase in Ω were noted. These experimental observations are rationalized in terms of the mobility of the absorbed H in the amorphous structure and the possible role of it in the shear transformation zone dynamics during deformation of the metallic glass.

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1. Introduction

Since the discovery of amorphous alloys by Klement et al. [1], there has been considerable interest in these materials due to their unique properties and functionalities, including their potential as an effective hydrogen (H) storage and separation medium [2,3]. Recently, metallic glass (MG) membranes that are permeable to H have gained interest due to the increasing importance of H separation in a number of applications, e.g., H-powered fuel cells. In particular, MG-based membranes are considered as an

attractive replacement for expensive Pd and Pd-based alloy membranes because of their comparable H permeability but lower cost [4,5]. In addition to such technological importance, the possible influence of H on the structural characteristics of MGs has also gathered scientific interest due to the small size of H and the open interatomic spaces in the amorphous structure [6,7].

Generally, the compositions of MGs that are suitable for H separation have hydride-forming elements such as Zr and Ti due to their strong affinity to H. A representative example is the Ni–Nb–Zr alloy system in which Zr is the key element [8,9]. It has been reported that with increasing Zr content in this system, the interatomic spacing, H solubility and diffusivity increase whereas the crystallization temperature (T_x), hardness and fracture strength decrease

* Corresponding authors.

E-mail addresses: jinyoo@kist.re.kr (J.-Y. Suh), jjjang@hanyang.ac.kr (J.-i. Jang).

[5,10,11]. Additionally, pronounced H embrittlement (HE) was also reported in alloys with high Zr content, which seriously compromises the utility of MGs in the intended application. Consequently, excellent resistance to HE is one of the primary requirements for MG-based H selective membranes.

In crystalline metals and alloys, the influence of H on the mechanical behavior (including HE) has been a subject of extensive studies over the past century [12–15]. Since deformation mechanisms in MGs are completely different from those in crystalline alloys [16–19], the mechanisms utilized to rationalize the H effects on mechanical properties in crystalline metals may not be applicable to those in MGs. While the mechanical behavior of hydrogenated MGs is investigated to some extent [20–22], no attempt to examine the influence of H on the yield behavior of amorphous alloys has been made until now. In this study, towards this end, we examine the spherical-tipped nanoindentation responses of a series of MG alloy ribbons that have varying Zr contents. The influence of H on the first pop-in load, P_I , and in turn the maximum shear yield stress underneath the indenter, τ_{\max} , was examined [23,24]. Statistical analysis of the τ_{\max} data was utilized to estimate the volume of the shear transformation zone (STZ), Ω , and how it is influenced by the H absorption in the alloys with different Zr content in the alloy. Our results show that the mechanical response of hydrogenated MGs is sensitive to the Zr content in them. These were then coupled with the results of the H desorption experiments to discuss the mobility of absorbed H in the amorphous structure and on local structural packing.

2. Experimental

Melt-spun MG ribbons, ~ 30 – $60 \mu\text{m}$ thick, with the compositions (in at.%) of $\text{Ni}_{45}\text{Nb}_{30}\text{Zr}_{25}$, $\text{Ni}_{33}\text{Nb}_{22}\text{Zr}_{40}\text{Co}_5$, $\text{Ni}_{27}\text{Nb}_{18}\text{Zr}_{50}\text{Co}_5$ and $\text{Ni}_{35}\text{Nb}_{30}\text{Zr}_{15}\text{Ti}_{10}\text{Fe}_5\text{Co}_5$, were prepared. These alloy compositions were selected on the basis of the $(\text{Ni}_{0.6}\text{Nb}_{0.4})_{100-x}\text{Zr}_x$ scheme, which has been shown to combine high H permeability and mechanical stability [5,11], and are hereafter referred to as Zr_{25} , Zr_{40} , Zr_{50} and $\text{Zr}_{15}\text{Ti}_{10}$, respectively. In the latter two, 5 at.% Co is added as it suppresses severe HE that commonly occurs in high-Zr alloys [11]. Note that in $\text{Zr}_{15}\text{Ti}_{10}$, the Ti content was also used for the abbreviation since Ti also exhibits a strong affinity to H just as Zr [8]. Samples with an area of $\sim 50 \times 10 \text{ mm}^2$ were cut from the ribbons and one side of them was coated with a $\sim 150 \text{ nm}$ thick pure Pd via RF magnetron sputtering. The purpose of this catalytic layer is to enhance the surface kinetics for the dissociation/recombination of H molecules during charging. Subsequently, H charging was performed in a Sievert-type apparatus at $300 \text{ }^\circ\text{C}$ and under constant pressure (0.5 MPa) of gaseous H_2 for 3600 s, followed by water-quenching of the specimen-containing cylinder. The alloys were weighed before and after charging for estimating the mass fraction of H in each sample. While the specimens

weigh $\sim 100 \text{ mg}$ and the weight change due to hydrogenation is $\sim 1 \text{ mg}$, the precision balance, MSE3.6P-000-DM (Sartorius, Göttingen, Germany), can weigh with the resolution of μg , leading to an accuracy of 0.001% in weight gain measurement.

Nanoindentation tests were conducted at room temperature using a Nanoindenter-XP (formerly MTS; now Agilent, Oak Ridge, TN, USA) with a spherical indenter (tip radius, $R = 5.73 \mu\text{m}$) on the side of the sample surface that was not coated with Pd and was polished with $0.3 \mu\text{m}$ alumina to a mirror finish. Prior to testing, R was calibrated through the Hertzian contact analysis [25,26] of indentations made on fused quartz. About 50 tests for each condition were conducted in load-control mode at a constant loading rate of $dP/dt = 1 \text{ mN s}^{-1}$, where P is the indentation load and t is time. In every case, the maximum depth of penetration (h_{\max} , at largest $\sim 400 \text{ nm}$) was much lower than one-tenth of the ribbon thickness. Hence, the effect of the substrate on the measured P vs. depth of penetration, h , can be neglected. All H-charged specimens were tested within 72 h of charging.

For investigating the correlation between Zr contents and H solubility in the ribbons, H desorption behavior was analyzed by thermal desorption spectroscopy (TDS) studies with a thermal conductivity detector (TCD). For TDS measurements, AutoChem HP model 2950 Chemisorption Analyzer (Micromeritics Instrument Corporation, Norcross, GA, USA) was used. The flow rate of Ar carrier gas was 50 ml min^{-1} and the H release behavior was monitored during heating with a constant heating rate of $20 \text{ }^\circ\text{C min}^{-1}$.

3. Experimental results

3.1. Estimation of critical shear yield stress

The P – h curves often exhibit either sudden bursts of displacement or load drops, when nanoindentation is performed under load or displacement control, which are often referred to as “pop-ins” [17,18,27,28]. If a spherical tip is used, the deformation prior to the first pop-in is purely elastic and hence follows Hertzian contact theory [26]. The first pop-in then corresponds to the elastic-to-plastic transition, and the load, P_I , at which it occurs can be utilized to compute τ_{\max} , which represents the critical shear strength for the onset of plasticity, as [25]:

$$\tau_{\max} = 0.31p_0 = 0.31 \left(\frac{3}{2}p_m \right) = 0.31 \left(\frac{6E_r^2}{\pi^3 R^2} P_I \right)^{\frac{1}{3}} \quad (1)$$

where p_0 and p_m are maximum and mean pressures of the contact, respectively, and E_r is the reduced modulus that can be determined by $E_r = \left(\frac{E_s}{1-\nu_s^2} + \frac{E_i}{1-\nu_i^2} \right)^{-1}$, where E and ν are the elastic modulus and Poisson's ratio, with the subscripts s and i indicating the sample and the indenter, respectively.

Fig. 1 shows representative examples of the P – h curves with pop-ins. When the peak load was lower than the P_I

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