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Instrumented indentation of a Pd-based bulk metallic glass: Constant loading-rate test vs constant strain-rate test

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ABSTRACT

Most of the previous nanoindentation experiments on bulk metallic glasses (BMGs) were made under a constant 'loading rate,' although 'strain rate' is a more useful parameter than loading rate to analyze the inhomogeneous plasticity in the BMG according to the classic free-volume theory. Here, we explore the strain-rate dependency of plastic characteristics in a Pd-based BMG through nanoindentation tests under a variety of constant strain rates (0.01–0.25 s^{-1}). The results are compared with those from nanoindentations under various constant loading rates (0.05–5 mN/s) and discussed in terms of the influences of strain rate on the plastic flow characteristics in the BMG.

1. Introduction

In bulk metallic glasses (BMGs), plastic deformation at low temperature is very inhomogeneous and highly localized into thin shear bands having initial thickness of ~10 nm [1,2]. As a result, stress–strain curves obtained from compression tests of BMGs often exhibit serrated flows that are very similar to Portevin–Le Chatelier phenomena observed in some crystalline materials such as Al and Ni alloys, if the deformation mechanism is totally different. The serrated flow behavior due to the inhomogeneous deformation has been also reported in load–displacement (P–h) curve recorded during instrumented indentation (especially nanoindentation) experiments of BMGs. Since Schuh and Nieh [3] declared through systematic examination that the serrated flow during nanoindentation is seriously dependent not only on the chemical composition of the tested BMG but also on the indentation rate, there have been many works on the rate dependency of the inhomogeneous deformation (for example, see [4–8]). From the extensive investigations, it is now well accepted that the serrated flow during nanoindentation of metallic glasses is highly dependent on "strain-rate ($d\varepsilon/dt$)" as in compression or bending experiments [1,2]. Nevertheless, it is interesting to find that most of the indentation experiments in the previous works were made under constant loading rate (dP/dt) condition (possibly due to the instrumental limitation).

During indentation at a constant loading rate, the indentation strain rate, which is defined as $h^{-1}(dh/dt)$ [9], is non-linearly diminishing with indentation depth. So, it is hard to directly convert a constant loading rate to a representative strain rate that might be a more useful parameter than loading rate for analyzing the inhomogeneous deformation according to the classic free-volume theories [10,11].

With this in mind, we performed nanoindentation experiments on a Pd-based BMG under constant strain-rate condition. The purpose of this paper is to report our preliminary results on the 'strain-rate' dependency of inhomogeneous plasticity during nanoindentation of the BMG. Additionally, the results were compared with those from constant loading rate nanoindentation on the same BMG.

2. Experimental

Nanoindentation experiments were performed on a mechanically polished $Pd_{40}Cu_{30}Ni_{10}P_{20}$ BMG using a Nanoindenter-XP (MTS Corp., Oak Ridge, TN) with a common Berkovich indenter. The indentation rate was controlled under either constant loading rate condition (from 0.05 to 5 mN/s) or constant strain-rate condition (from 0.01 to 0.25 s^{-1}), while the maximum indentation load (P_{max}) was fixed as 100 mN. To properly capture the small-scale serrations, thermal drift was maintained below 0.05 nm/s.

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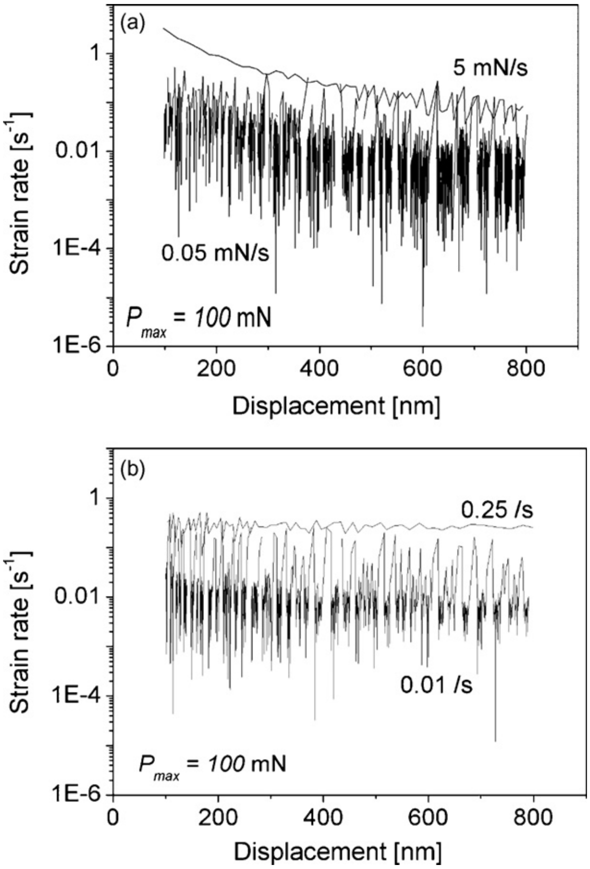

Fig. 1. Typical relationship between strain rate and displacement during nanoindentation tests under (a) constant loading rate condition and (b) constant strain-rate condition.

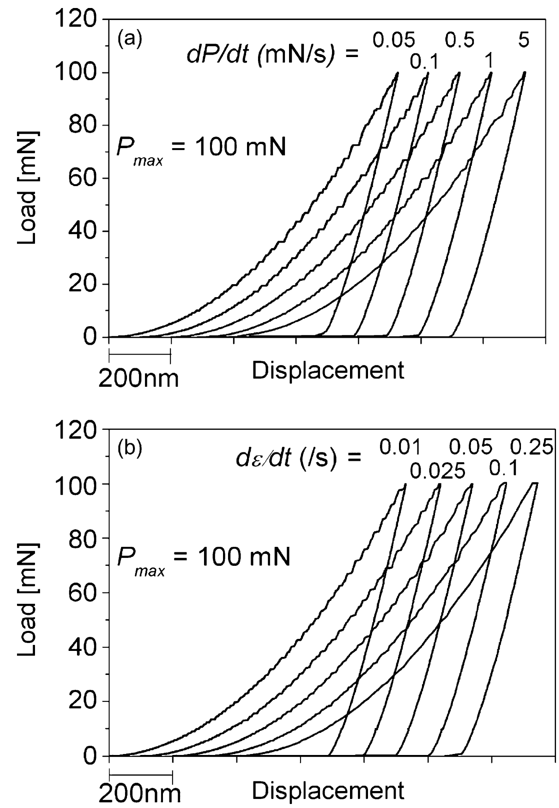

Fig. 2. Representative load–displacement curves obtained from nanoindentations made under (a) constant loading rate and (b) constant strain rate.

3. Results and discussion

Fig. 1 plots the indentation strain rate as a function of the indentation depth for both cases of constant loading rate condition and constant strain-rate condition. In the figure, early part of strain (i.e., $h < 100$ nm) is excluded due to the possible uncertainty of the indenter geometry. During nanoindentation of the BMG at a constant loading rate [Fig. 1(a)], strain rate is decreasing with displacement as expected from the definition of indentation strain rate, $h^{-1}(\mathrm{d}h/\mathrm{d}t)$ [9]. As reported previously [3], the curve for strain rate vs. displacement is not monotonically decaying but shows many peaks in strain rate that appear to increase in size as (1) the depth increases and (2) the loading rate decreases. Schuh and Nieh [3] suggested that the strain-rate peaks exactly correlated with the serrations observed in nanoindentation load–displacement curve. Typical relationship between strain rate and displacement from nanoindentation under a constant strain rate is shown in Fig. 1(b). Unlike in Fig. 1(a), the strain rate is indeed maintained as a constant independent of indentation depth. Interestingly, however, there are still many strain-rate peaks (which are bigger at slower rate) and the strain fluctuation decreases in size as the indentation proceeds, which is an opposite trend to that observed from constant loading rate test.

Fig. 2(a) exhibits the representative *P*–*h* curves recorded during nanoindentation to P_{max} = 100 mN at a variety of constant loading rates ($\mathrm{d}P/\mathrm{d}t$ from 0.05 to 5 mN/s). Each *P*–*h* curve has been shifted for clarity. The rate-dependency of the serrations in the figure is consistent with that reported previously [3]; the serrations are more pronounced at lower loading rate. The *P*–*h* curves measured during nanoindentation at various strain rates ($\mathrm{d}\varepsilon/\mathrm{d}t$ from 0.01 to 0.25 s^{-1}) is represented in Fig. 2(b). Compared with Fig. 2(a), although the similar rate dependency of the serrated flow is seen in this figure, the serrations are a little less clear. It is interesting to note that in Fig. 2(b) the serration size is increasing with indentation depth, as under constant loading rate condition. This trend is opposite to a possible expectation based on the fact that the size in strain-rate fluctuation diminishes with the depth under constant strain-rate condition [see Fig. 1(b)]. Therefore, we believe that a more accurate description of the relationship between the strain-rate fluctuation and the serrations is desirable in future work.

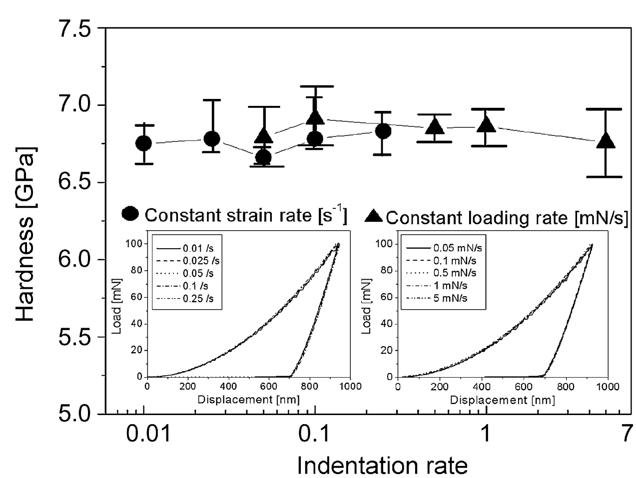

Fig. 3. Dependence of nanoindentation hardness on the indentation rate and testing mode. Inset figures show the superposition of the *P*–*h* curves recorded from different testing conditions.

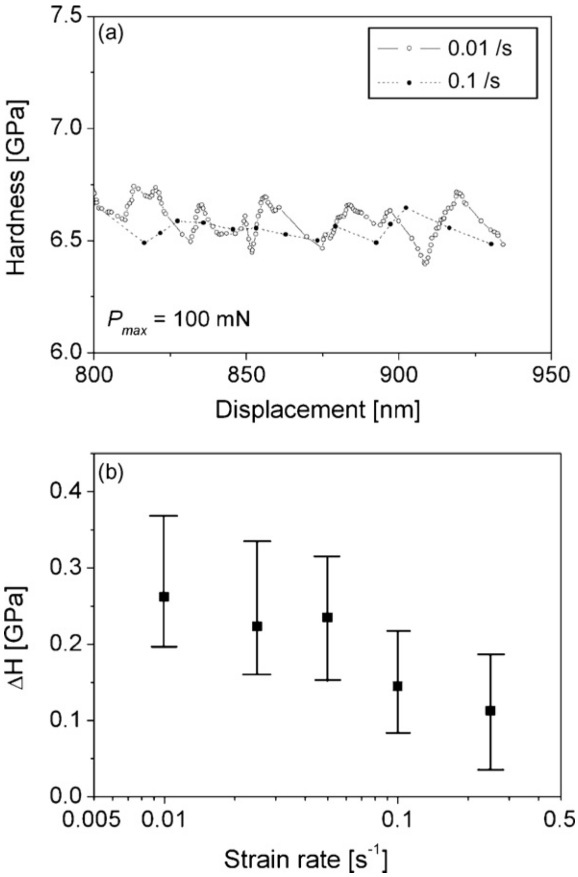

Fig. 4. Results from CSM mode nanoindentations at constant strain rate: (a) typical example of the fluctuations in hardness; (b) change in hardness serration size as a function of the logarithmic strain rate.

Next, we investigated the strain-rate dependency of nanoindentation hardness. There has been a dispute on the rate dependency of BMG hardness. For instance, in the case of $Pd_{40}Cu_{30}Ni_{10}P_{20}$ BMG examined in present work, Concustell et al. [6] reported the decrease in hardness of BMGs with decrease in the loading rate (from 6.4 to 0.04 mN/s), while Golovin et al. [12] observed no evidence for the loading-rate dependency of hardness. All the *P*–*h* curves obtained in this work are superposed in inset figures of Fig. 3. In the Pd-based BMG examined here, the total plastic deformation does not change with either loading rate or strain rate. The nanoindentation hardness values calculated according to Oliver–Pharr method [13] are also summarized in Fig. 3, indicating that the hardness is ~6.75 GPa and independent of both strain rate and loading rate. The hardness values of $Pd_{40}Cu_{30}Ni_{10}P_{20}$ BMG measured here are consistent with those by Golovin et al. [12], but are higher than those by Concustell et al. [6], ~5 GPa. This difference in hardness might arise from the difference in the applied maximum load (100 mN and 85 mN in this paper and [12], respectively, vs. 5 mN in [6]), but this is not well understood.

Very recently, Yang and Nieh [7] demonstrated a clear fluctuation in hardness of a Au-based BMG during indentation, and argued based on the classic free volume theory [10,11] that there is a correlation between hardness serration size, ΔH (which appeared to be independent of indentation depth at a given strain rate) and strain rate as $[\ln(d\varepsilon/dt) - A] \propto -\Delta H$ where A is a constant at a given testing condition. However, their experiments [7] were done only under constant loading rate and they did not provide any clue for how they could directly convert a loading rate to a strain rate for entire displacement range.

Here, in order to directly analyze the relationship between the ΔH and strain rate, we measured the hardness through continuous-stiffness-measurement (CSM) nanoindentation tests [13] (which can continuously provide the hardness with increasing indentation depth) at various constant strain rate. In Fig. 4(a), a number of hardness serrations are seen, and the size of serration exhibits a clear dependency on the strain rate. Fig. 4(b) illustrates the ΔH as a function of the logarithmic strain rate. The ΔH appears to decrease linearly with the logarithmic increase in the strain rate, which is in very good agreement with Yang and Nieh's suggestion [7], $[\ln(d\varepsilon/dt) - A] \propto -\Delta H$. It is noteworthy that the similar relationship between stress serration size ($\Delta\sigma$) and strain rate during compression tests of BMGs was recently reported [14,15].

4. Summary

In this work, to investigate the strain-rate dependency of plastic characteristics in a Pd-based BMG, we performed nanoindentation tests under a variety of constant strain rates (0.01–0.25 s^{-1}) and compared the results with those from constant loading rate tests (0.05–5 mN/s). It was revealed that the serrated flow is strongly dependent on the strain rate while the hardness is almost independent of the rate.

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