



The impact of elastic and plastic strain on relaxation and crystallization of Pd–Ni–P-based bulk metallic glasses

Yu.P. Mitrofanov,^{a,b,*} M. Peterlechner,^a I. Binkowski,^a M.Yu. Zadorozhnyy,^c I.S. Golovin,^c
S.V. Divinski^a and G. Wilde^{a,d,*}

^a*Institute of Materials Physics, University of Münster, Wilhelm-Klemm Str. 10, Münster 48149, Germany*

^b*Department of General Physics, State Pedagogical University, Lenina Str. 86, Voronezh 394043, Russia*

^c*Department of Physical Metallurgy of Nonferrous Metals, National University of Science and Technology “MISIS”,
Leninskii pr. 4, Moscow 119049, Russia*

^d*Institute of Nanochemistry and Nanobiology, School of Environmental and Chemical Engineering, Shanghai University,
Shanghai 200444, PR China*

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Abstract—The effects of deformation and subsequent heat treatment on the low-temperature heat capacity, enthalpy relaxation rate and mechanical losses of two Pd–Ni–P-based bulk metallic glasses of slightly different compositions and different thermal stabilities have been investigated. It was found that the crystallization temperatures decreased significantly with imposed strain and the effect was more pronounced for the alloy with a higher thermal stability. The boson heat capacity peak increases with increasing strain in both alloys. However, after annealing treatments above room temperature, it relaxes to a lower enthalpy state as compared to that of the as-quenched state for the alloy with a lower thermal stability. The existence of two counteracting processes that might be related to different shear band structures within one homogeneously deformed sample is suggested. These results agree with the internal friction data, which indicate different regimes of mechanical damping as a function of the strain amplitude, while the critical amplitude of a transition between the regimes depends on the imposed strain. The results are interpreted within the energy landscape approach and advocate that the composition-dependent local atomic configurations affect significantly the response of the glass to an applied strain.
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1. Introduction

According to the most generally accepted approach, inhomogeneous deformation of metallic glasses occurs through the localization of free-volume by external mechanical stress [1,2], which leads to a local decrease of the viscosity and, thus, to shear localization in narrow low-viscosity layers (i.e. shear bands) [3–5]. Such layers might be characterized by short-range low internal stresses. There is also an alternative description based e.g. on dislocation-like notions, which leads to the expectation of the presence of high long-range internal stresses within the shear bands [6–8]. Yet, neither of the theoretical approaches is able to predict the changes of state variables (such as enthalpy or volume) of the shear bands or to

allow calculating the activation barrier for shear band activation or the relaxation behavior within the shear bands and the impact on the matrix. In that respect, careful measurements by acoustic emission spectroscopy during the deformation of a metallic glass have shown that the generated shear bands have a rather wide spectrum of properties [9]. Recent results obtained by combining different quantitative analytical transmission electron microscopy methods on a deformed Al-based metallic glass have verified this result and have shown that even densification can occur in a fraction of the shear bands, which is counter-intuitive with respect to the free-volume based descriptions [10]. Thus, in spite of considerable experimental and theoretical efforts, there is no accepted viewpoint on the microscopic mechanism of inhomogeneous plastic deformation of metallic glasses and the related shear banding phenomenon.

After inhomogeneous plastic deformation accompanied by strain localization, the volume of the deformed metallic glass contains modified regions (i.e. the shear bands). Consequently, the physical properties of deformed glassy samples that contain shear bands are different in

* Corresponding authors at: Department of General Physics, State Pedagogical University, Lenina Str. 86, Voronezh 394043, Russia (Yu.P. Mitrofanov). Institute of Materials Physics, University of Münster, Wilhelm-Klemm Str. 10, Münster 48149, Germany (G. Wilde); e-mail addresses: mitrofanovyup@gmail.com; gwilde@uni-muenster.de

comparison with the properties of undeformed samples. For example, the shear bands induced by cold rolling in a CuZr-based bulk metallic glass lead to a decrease of the activation energy for crystallization [11] and cold rolling of a Pd-based metallic glass was found to influence the relaxation of the electrical resistance [12] in the temperature range below and near the glass transition temperature. Also, the compressive and tensile mechanical properties of Zr-based bulk metallic glasses were reported to be modified by pre-deformation via cold rolling [13,14]. On the other hand, some investigations reported an absence of (or inefficient) influence of shear bands on the properties of metallic glasses [14,15]. Thus, the properties of a deformed metallic glass are or are not affected by the shear bands.

It has recently been found [16,17] that room-temperature plastic deformation of bulk metallic glasses affects one important characteristic of the glassy state, namely the low-temperature boson heat capacity C_p contribution that results in a peak in $C_p(T)/T^3$ at low temperatures and that, according to the generally accepted view, is based on an increase of the low-energy vibrational density of states. The boson heat capacity peak of metallic glasses increases with strain, i.e. the shear band formation is apparently accompanied by an increase of the number of low-energy vibrational states. Furthermore, the boson heat capacity peak in the deformed glass relaxes faster compared to the undeformed glass, possibly indicating “the presence of two distinct relaxation processes that affect the boson heat capacity contribution in an opposite way in comparison to the undeformed glassy state” [16,17]. Correspondingly, the shear band structure is probably heterogeneous, in agreement with the microscopic investigations [10] and also in accordance with recent work [18] illustrating the appearance of a sandwich-like heterogeneous structure of shear bands in bulk Pd_{40.5}Ni_{40.5}P₁₉ metallic glass near fracture; the shear bands were found to contain solid-like and liquid-like zones. It was concluded [18] that the observed heterogeneous structure of shear bands can be related to both, short-range ordering and middle-range ordering in the glassy system. In the microscopy study [10], quantitative analyses showed variations in both, the local specific volume and the local medium-range order for different shear bands and even for different parts of one shear band. Thus, it seems safe to assume that depending on the amount of specific excess volume occupied by the first and the second zones, the properties of a single shear band can be different. Consequently, the properties of deformed metallic glasses, containing different structural types of shear bands, will be different from each other, even at identical thermodynamic conditions. Since the property variations are intimately connected with the local atomic structures, it seems likely to expect that modifications of the composition of the glass affect the formation of different zones within the shear bands.

In this paper, we present the results of low-temperature heat capacity measurements, enthalpy relaxation rate and damping characteristic measurements of two (Pd–Ni–P)-based bulk metallic glasses with similar but not identical chemical compositions in both, the as-quenched states and after deformation by rolling at room temperature. All of the obtained results yield a consistent picture of the deformation-induced structural transformations in Pd–Ni–P-based metallic glasses.

2. Experimental

Pd–Ni–P-based bulk metallic glasses were chosen for investigation, since these alloys offer a very high glass forming ability [19] and high thermal stability [20] against crystallization in the glassy and the deeply undercooled liquid states. Two master alloys were prepared by direct melting of palladium (purity 99.95%) and Ni₂P powder (purity 99.5%) in an alumina crucible using an induction furnace in a pure argon atmosphere. The chemical compositions of the alloys were measured by atomic absorption spectroscopy (Mikroanalytisches Labor Pascher, Germany) as Pd₃₈Ni₄₀P₂₂ (G38) and Pd₄₀Ni₄₀P₂₀ (G40). The compositions are given in at.%. These crystalline master alloys were subsequently re-melted and then chill-cast into a copper mold. The castings were carefully checked by X-ray diffraction (XRD, Siemens D5000) to be fully X-ray amorphous.

2.1. Plastic deformation

The glassy samples were plastically deformed by cold rolling in one step at room temperature to various strains. The deformation degree was determined by the thickness reduction as $\varepsilon = (h - h_0)/h_0$, where h_0 is the initial thickness of the sample and h denotes the thickness of the samples after rolling. The strain rate $\dot{\varepsilon}$ was estimated to be $\approx 10 \text{ s}^{-1}$. Cold rolling of the glassy samples led to shear band formation, which was detected by optical microscopy. The density of shear bands, ρ_{SB} , was increased with increasing strain and $\rho_{\text{SB}} \approx 0.03 \mu\text{m}^{-1}$ and $\approx 0.2 \mu\text{m}^{-1}$ at $\varepsilon \approx 0.05$ and 0.4, respectively. Since the relative volume occupied by one shear band is low (i.e. the shear band thickness is about 5–20 nm) [10,21], the volume fraction of material inside shear bands in the strongly deformed glass was estimated to be lower than 1%.

It has to be noted that in spite of the intended equal deformation path and level of the two sample types (G38 and G40), it was impossible to impose the same strain on different samples using similar rolling conditions. Still the differences are not appreciably high and they do not affect the conclusions derived here.

2.2. Calorimetric measurements

The relaxation behavior of the undeformed and deformed glasses was investigated by differential scanning calorimetry (DSC) using a Perkin–Elmer Diamond instrument and applying linear heating with a heating rate of 20 K/min. The DSC curves, characterizing the enthalpy relaxation, were calculated as the difference between two subsequent runs taken on the same sample in the glassy and fully crystalline states. Low-temperature ($2 \leq T \leq 30 \text{ K}$) heat capacity measurements were performed by a physical property measurement system (PPMS™, Quantum Design Inc.). Before the measurements, some samples were pre-annealed by linear heating up to temperatures $T_a = 450, 550, 620, 700$ and 823 K in a pure argon atmosphere with a heating rate of 5 K/min and subsequent cooling to room temperature at the same rate. After the respective treatments (cold rolling and/or annealing) the samples were analyzed by XRD.

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