

Densification contribution as a function of strain rate under indentation of terbium-doped aluminophosphate glass

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Abstract In this work, the strain rate effect on the deformation processes under Berkovich indentation of Tb-doped aluminophosphate glass has been investigated. It is shown that both densification and shear flow, adopted as main mechanisms of plastic deformation for oxide glasses, are strain rate sensitive. Moreover, the shear flow is assumed to be responsible for the strain rate sensitivity of densification. The densification contribution to the total plastic deformation is found to be greater for lower strain rate, and the same tendency is observed for the plastic flow. This, in turn, leads to the influence of the strain rate on the hardness values, manifesting as a softening of the glassy matrix with the decrease of strain rate caused by more intensive development of the densification and shear flow. The decrease of hardness with load increase is attributed to the involving and increasing contribution of the shear flow and fracture to the total deformation process.

Introduction

Deformation mechanisms of amorphous and vitreous materials under concentrated load action (indentation) remain a topic of great interest in terms of better understanding of the relationship between mechanical behavior, structure, and processes that take place in non-crystalline materials during highly localized deformation. It was suggested [1] that different mechanisms may be responsible for the plastic deformation of glassy matrix, the main of which are the particular modes of shear flow [2–7] and permanent densification caused by the compression of material [8, 9]. The contribution of each of these mechanisms to the total plastic deformation strongly depends on the composition and the resulting glass structure, particularly, on the atomic packing density [8, 10]. Less dense structure on the one hand favors the development of the permanent densification mechanism and on the other hand, may hamper the development of shear flow. Glasses based

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on 3D structural units such as amorphous silica are characterized by low atomic packing density, whereas layer (2D), chain (1D), or cluster (0D) glass structures, proper to multi-component oxide glasses, chalcogenide, or metallic ones, have relatively more dense atomic packing. It was shown that the lower the atomic packing density, the higher the contribution of the densification mechanism is [8].

A method to estimate the contribution of densification mechanism to the total plastic deformation of the glass matrix under indentation is the heating of the indented sample up to $0.9T_g$ (glass transition temperature), that causes the recovery of the indentation depth, the measure of which is believed to assess what part of the imprint volume is formed due to the permanent compression of material [8, 11]. Using this method, it was shown that not only the structure of the glass is responsible for the development of one or another deformation mechanism, but also the geometry of the indenter [12, 13] is responsible for the creation of a specific stress state of the deformed region [14].

The aim of this work is to explore the influence of the strain rate during indentation on the deformation processes in phosphate glasses. The strain rate sensitivity is well investigated for crystalline, poly-, and nanocrystalline materials based on such notions like activation volume, and dislocation or grain boundary movement mechanisms. Among glassy materials, most of research concerning strain rate sensitivity relates to bulk metallic glasses [15–17]; however, little work has been done on oxide glasses [18, 19].

The aluminophosphate glasses doped with rare-earth (RE) elements (Tb in this study) are promising candidates for high energy lasers, magneto-optical sensors, and micro-opto-electro-mechanical systems—MOEMS. The optical, structural, and thermal properties [20], and also phase transformation and microstructure evolution after heat treatment of phosphate glasses containing terbium ions were recently reported [21]. Taking into account the mechanical impact in some of the above-mentioned applications, it has become important to investigate the peculiarities of their mechanical behavior [1, 22, 23]. It is known that phosphate glasses possess a polymeric-like structure, but the addition of network modifiers changes this 3D structure into 1D one. In this context, it is of interest to investigate the role of densification mechanism for these glasses and the influence of strain rate on this process under point contact loading.

Experimental

Tb-doped aluminophosphate glass with oxidic composition in raw materials (mol%) of $17.88\text{Li}_2\text{O}-8.93\text{Al}_2\text{O}_3-6.31\text{BaO}-1.27\text{La}_2\text{O}_3-63.72\text{P}_2\text{O}_5-1.89\text{Tb}_2\text{O}_3$ was prepared

from analytical grade reagents: Li_2CO_3 , BaCO_3 , Al_2O_3 , La_2O_3 , H_3PO_4 (85 % solution in water), and Tb_2O_3 , using a wet non-conventional raw materials preparation method followed by the melt quenching of the glass. Details of a similar preparation procedure adopted for phosphate glasses doped with different rare-earth oxides have been reported elsewhere [24].

All the reagents were introduced in H_3PO_4 solution at the beginning of the batch preparation process under continuous stirring and dried on a heating plate, until solidification appeared, at around 423 K. This homogenization and drying step is accompanied by the release of gases resulting from chemical reactions that promote the formation of homogeneous metaphosphates, which additionally improves the chemical homogeneity of the final glass [20, 24]. Subsequently, the dried mixture was heat treated in an alumina crucible during two sequential steps: (i) at a heating rate of 100 K h^{-1} from room temperature up to 973 K, to eliminate volatile components, in a Lenton electrical furnace, provided with silicon carbide (SiC) rods and (ii) at a heating rate of 250 K h^{-1} up to 1523 K with 4 h holding at this temperature to achieve glass melt and refining, in a Nabertherm electrical furnace, using molybdenum disilicide as heating elements. The improvement of glass melt quality was assured by a mechanical stirring device, equipped with an alumina stirrer. The rotation speed varied between 100 and 250 rot/min, depending on the temperature and viscosity of the glass melt. Then the glass was cast in graphite molds and annealed at 723 K for 6 h to remove internal stresses. The obtained glass had the following composition (wt%): $4.99\text{Li}_2\text{O}-8.47\text{Al}_2\text{O}_3-8.97\text{BaO}-3.82\text{La}_2\text{O}_3-67.31\text{P}_2\text{O}_5-6.44\text{Tb}_2\text{O}_3$. The glass composition was calculated from the raw materials oxide composition, considering a loss by volatilization of 25 % for phosphorus oxide. This volatilization loss fits well to data from a previous work [25], where similar phosphate glasses doped with other several rare-earth oxides were investigated. In that work, the induced coupled plasma-atomic emission spectroscopy and induced coupled plasma-mass spectrometry were used to measure the oxide composition of glass samples.

The as-prepared glass block was cut with diamond disk to obtain thin glass sample with a thickness of about 2 mm, which was polished using 150-, 300-, and 600-grade SiC followed by cerium oxide—CEROX abrasive. Before nanoindentation, the samples were finally polished by Cr_2O_3 powder to reduce the surface defects and to obtain a sufficiently smooth surface.

The local deformation of glasses was carried out on the instrumented nanoindentation device (Nanotester PMT-3NI-02) equipped with a Berkovich indenter of 200-nm tip radius. The calibration of the indenter area function was made by using the Oliver–Pharr method [26] on a fused