SEM and AFM analysis of the shear bands in Zr-based BMG after HPT

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Abstract. The surface relief formed by shear bands in bulk metallic glasses (BMG) under high-pressure torsion (HPT) has been investigated by the method of scanning electron microscopy (SEM) and atomic force microscopy (AFM). For this purpose, two halves of disks of the bulk metallic glass were jointed together and processed by HPT. The SEM examination of the internal surfaces of two joint halves of an HPT-processed disk allowed to study the formation and accumulation of shear bands under an increased imposed strain. The maximum density of the shear bands is observed at the edges of the HPT samples and in areas adjacent to the upper anvils. The observed minimum shear band spacing is equal to 0.5 μm after HPT processing for 5 revolutions.

1. Introduction

Bulk metallic glass (BMG) [1, 2] attract significant scientific and practical interest. However, during their deformation, the plastic flow is localized in thin shear bands (SB), which leads to brittle fracture during deformation by tension. One of the effective ways to transform the structure and properties of amorphous materials is deformation using high-pressure torsion. Studies [3-6] showed that under the effect of HPT on initially amorphous alloys, partial nanocrystallization and the formation of a nanocluster structure can occur, a growth of free volume and an increase in speed sensitivity of deformation is noted. The urgent task is to study the formation of shear bands in BMG with HPT. However, during TEM studies of BMG after HPT, shear bands are often not detected [6], due to the small band thickness (10 nm) and since the difference in the amorphous structure of the bands and the matrix is not sufficient to form a visible contrast. When deformed by pressing or rolling, the SBs emerge onto the surface of the BMG in the form of steps, which are well observed by SEM and AFM [7]. However, with HPT, the sample is clutched between the anvils, and there are no “free surfaces” for the SB exit in this scheme. In [8], a new method of HPT of BMGs was proposed, which made it possible to conduct SEM study of shear bands on a free surface. In the current article we continue to use the AFM method.
2. Experimental results and discussion

The Zr$_{52.5}$Cu$_{17.9}$Ni$_{14.6}$Al$_{10}$Ti$_{5}$ (at. %) bulk metallic glass rods 10 mm in diameter and 40 mm in length (hereinafter referred to as Vit105) were used in this study. Disks with a thickness of 1 mm were cut out of the initial BMG ingot. Each disk was cut into two halves (figure 1a), and the internal surfaces of both halves were polished. Then the halves of the disks were joined together with a thin layer of a lacquer in order to prevent the adhesion of the two parts, and processed by HPT, as shown in figure 1b, under a pressure of 6 GPa at room temperature.

![Figure 1.](image)

**Figure 1.** (a) Scheme of the two halves of the as-cast HPT disk. (b) Scheme of constrained HPT processing: the lower anvil has a groove with a depth of 0.5 mm, the upper anvil is flat. (c) SEM image of the internal surface of the sample HPT 30°.

The observed structure obtained by HPT with various regimes is inhomogeneous across the internal surface of the sample. Shear bands with a much higher density are formed on the side of the sample (figure 1c). The observed bands can be conventionally divided into primary shear bands, less intense secondary shear bands that intersect primary ones, and even thinner tertiary shear bands that are visible at high magnifications (figure 3). Such a division of SB in BMGs samples after rolling was also observed [7]. In the main area of the sample subjected to HPT with a rotation of 30° (HPT 30°) a system of parallel primary SB is formed, directed at an angle of 45° to the upper surface (figures 1, 2), with an average distance between the lines about 10 µm. In the marginal regions of the sample, the SB density is noticeably higher. This is attributed to the larger strain in this area.

An increase in the number of revolutions of HPT leads to an increase in the density of SBs. The distance between the bands in the areas of r = 2.5 mm was about 0.3 µm after HPT for turns n = 5. In the samples after HPT n = 5, the SB of the first type have an angle of about 45° to the upper surface of the sample, and the shear bands of the second type are nearly parallel to the surface of the sample. These SB thus divide the sample volume into numerous units (figure 3).
Figure 2. SEM and AFM images of the internal surface of the Vit105 BMG subjected to HPT 30°, and the step height distribution at the exit of the shear bands (AFM).

Figure 3. SEM and AFM (lateral forces) images of the internal surface of the Vit105 BMG subjected to HPT n=5.

The density and distribution of the shear bands, stated by AFM, correspond to the pattern observed by SEM. However, the use of AFM made it possible to estimate the height of the shear bands, which in the sample HPT 30° for the primary bands were about 400 nm, the secondary ones - about 50 nm. In the HPT sample deformed to n = 5, it is more difficult to analyze the AFM pattern due to the high density of the bands and a rougher surface profile of the sample. According to the first estimates, the height of the primary bands after HPT n = 5 reached 1000 nm. Hence, with an increase in the number of revolutions (shear angle) of HPT, both the density of the shear bands and their intensity (the height of the steps) increase.

3. Conclusions
Two halves of disks of the BMG were processed by HPT, and the parameters of SB evolution with deformation were examined using the SEM and AFM imaging of the internal surfaces of two joint halves of the HPT-processed disk. HPT processing leads to the formation of a high density of the SB in the BMG. An increase in HPT strain leads to a significant increase in the SB density, and the spacing between SB after HPT n = 5 is 0.5 µm. The maximum density of the SB is observed at the edges of the HPT samples. The use of AFM made it possible to estimate the height of the shear bands, which in the sample HPT 30° for primary bands was about 400 nm, after HPT n = 5 - about 1000 nm, for secondary bands - about 50 nm.
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