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Ion-bombardment modification of the surface of mirrors fabricated of ZrTiCuNiBe amorphous alloys

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Abstract. When preparing mirror samples of amorphous metal alloys some inhomogeneities of the structure became to be seen on the surface. These inhomogeneities were modified during bombardment with ions of deuterium and argon plasma. Besides, a new blister-like type of inhomogeneities was found on the mirror surface in one experiment. In the paper a short description of obtained results are presented.

1. Introduction

Not long time ago the Bulk Amorphous Alloys (BAA) appeared [1,2] which have some properties different from properties of polycrystalline materials that are used at present time in technology and industry. Because of rather short time past since the BAA appeared, not many their characteristics were studied in detail, what limit their use in everyday life. Among characteristics that are not well known yet are those ones important for interaction of BAA with ions, like sputtering rate, gas absorptivity, modification of the near-surface layer, etc.

The looking for the answers these questions is the main objects of this paper. We present here some results of investigations directed to obtaining the data on peculiarities of impact of ions (deuterium and argon) with different energy on properties of mirror samples fabricated of BAA, namely, surface modification under long term ion bombardment, on deuterium uptake, reflectance, and sputtering yield.

2. Samples

The experiments were provided with mirror specimens fabricated of moldings of amorphous materials prepared by:
- Liquidmetal Technology Corporation (LTC) in the USA,
- Institute of Solid State Physics, Materials Science and Technologies of National Science Center “Kharkov Institute of Physics and Technology” (ISSPMT), and
- Hahn-Meitner-Institute (HMI) in Berlin, Germany.

The composition LTC and ISSPMT samples was – Zr(41.2)Ti(13.8)Cu(12.5)Ni(10)Be(22.5) and the composition of HMI samples was a little different – Zr(46.2)Ti(8.2)Cu(7.5)Ni(10)Be(27.5).
The round shape LTC and ISSMT samples were 22 mm in diameter, and the diameter of HMI samples was 5 mm; the thickness of all samples ~2.5 mm. After polishing to the mirror quality with gradual decrease of the abrasive particles, the samples were washed in acetone and saved in air. Before providing any experiment with exposing samples to ions of deuterium or argon plasma, the surface of every sample was cleaned with low energy (50–60 eV) ions of deuterium plasma (typical fluence $5 \times 10^{19}$ ion/cm²) to clean the surface from the organic film appeared due to washing in acetone.

The cleaning procedure and experiments with ion impact on mirror samples were provided in the DSM-2 installation described elsewhere [3], with parameters of ECR discharge plasma: $n_e \sim 10^{10}$ cm⁻³ and $T_e \leq 5$eV.

3. Experimental results
Several results on behavior of LTC mirror samples subjected to long-term bombardment with deuterium plasma ions have been already published in [4,5]. Here we repeat some of them for comparison with data obtained in experiments with samples of two other producers.

3.1. Surface of LTC mirror samples
3.1.1. After polishing and cleaning
After polishing and cleaning on surfaces of LTC samples some inhomogeneities became clearly seen [4]. In figure 1a the SEM photo obtained with scattered electrons of similar inhomogeneities is shown. They are a little above the matrix and occupy small part of the whole sample surface (1–2 %), therefore do not affect noticeably the reflectance. The fact they do protrude over the matrix indicates on higher local hardness in comparison to the hardness of the matrix. To clear up the reason of appearance of these inhomogeneities, the SEM photos were made with elastically backscattered electrons and the micro-probe analysis of the composition was provided (by measuring the characteristic X-ray radiation in X-ray spectrometer system attached to TEM). The photo of the same part of the surface shown in figure 1a is reproduced in figure 1b by registration of elastically reflected electrons. The darker color of the inhomogeneities is a qualitative indication on abundance of elements with lower atomic number. This qualitative result was proved by quantitative data obtained with application of microanalysis. It should be noted that, as we know, such inhomogeneities have not been described in literature previously.

**Figure 1.** SEM photos with secondary (a) and backscattered electrons (b) of inhomogeneities on the LTC sample surface; c) photo of interferometer microscope after the layer of ~2 μm was sputtered with deuterium plasma ions.

3.1.2. After long-term ion bombardment.
After long-term ion bombardment the inhomogeneities shown in figure 1 turned into shallow ‘pits’, what indicates that their sputtering rate is higher than the sputtering rate of the matrix, in agreement with lower portion of zirconium measured with micro-probe method. The photo of interferometer
microscope in figure 1c was made when the ion sputtered layer thickness (bombardment with deuterium plasma ions) was estimated as ~2 μm. It can be seen that the depth of the ‘pit’ is ~200 nm. The matrix maintained the initial smoothness of the surface.

When the effect of ion energy (deuterium plasma) was investigated, the new inhomogeneities of the surface microrelief were found out [5]. They were observed only on a rather small area of the part of mirror sample bombarded with 300 eV ions. The photos in figure 2a show the view of these ‘blister-like’ artifacts in different scales, and figure 2b,c – the reconstruction made from AFM data.

![Figure 2](image)

Figure 2. SEM photo of blister-like features (a), and the AFM-images of different form ‘blisters’ and their profiles (b, c).

3.2. Surface of ISSMT mirror samples

3.2.1. After polishing and cleaning

After polishing, washing and cleaning in deuterium plasma, the ISSMT samples viewed in SEM very peculiarly as figure 3a shows. The shallow pits were almost uniformly distributed along the surface. Their depth, according to data of interferometer and atomic-force microscopes, was 60–70 nm. At the same time, the SEM photos made in backscattered electrons, figure 3b, demonstrate a very homogeneous picture without any compositional inhomogeneities, what was supported also by results of microanalysis. So, at present we do not have a non-controversial explanation of appearance of such ‘pits’. However, it should be mentioned that because they are not deep and have a quite plane bottom, there is no measurable effect on reflectance at normal incidence in the range where optical measurements were done (250–650 nm).

3.2.2. After long-term ion bombardment

The long-term sputtering of ISSMT amorphous mirror sample with argon ions resulted in decreasing the contrast for observation of the pits that were clearly seen before sputtering (compare figure 3a and figure 3c). This is probably connected with gradual decrease of the depth of pits relatively the matrix level. Important that the surface continues to be smooth and reflectance does not change even after sputtering the layer of 13.4 μm in thickness, what is the experimental confirmation of the hypothesis stated in [6].
Figure 3. SEM photos of the polished mirror surface of a ISSPMT sample obtained with scattered electrons (a) and with backscattered electrons (b); SEM photo after layer of ~7 µm was sputter eroded with Ar ions of 1000 eV (c).

3.3. Surface of HMI samples
On the HMI polished samples small inhomogeneities were observed on the surface after polishing and they continued to be seen after ion bombardment. Majority of them are probably similar to that shown in figure 4, but were seen only partly. The shape of inhomogeneity of figure 4 is very much similar to the shape of the crystal, the photo of which was published in paper [7]. Therefore we can suppose that these inhomogeneities are crystals in the matrix and their composition is different in comparison to the matrix of the alloy, as was found in [7].

Figure 4. SEM photo of the polished mirror surface of a HMI sample after sputtering with argon ions the layer ~5 µm in thickness.

4. Conclusion
Some inhomogeneities were found out on the surfaces of mirror samples prepared of materials fabricated by 3 producers. The inhomogeneities differed from the main matrix by composition and structure (LMC and HMI samples) and are of a not known nature yet on the surface of ISSMT samples.

In the case of LMC material the inhomogeneities are abundant with elements of lower Z and their sputtering yield is a little higher than the sputtering yield of a matrix. As a result, the shallow pits (~0.2 µm) become to be visible after a layer of ~2 µm in thickness was sputtered by ions of deuterium plasma.

After sputtering by Ar$^+$ ions of the layer ~13.4 µm the surface of amorphous ISSMT mirror sample continued to be smooth.

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