

Letter to the Editor

## Observation of unique blister-like surface features on amorphous metallic alloys following bombardment with deuterium ions

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### Abstract

When investigating the impact of deuterium plasma ions (with energy 300 eV) on mirror specimen fabricated of bulk amorphous alloy Zr(41.2)Ti(13.8)Cu(12.5)Ni(10)Be(22.5) some unusual surface features were observed. Their shape differs from blisters observed on the surface of amorphous foils bombarded with high energy helium and hydrogen ions (Refs. [9–11]). In this Letter a short description of characteristics of these 'blister-like' features are presented.

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### 1. Introduction

Bulk amorphous metallic alloys (AMAs) first appeared about 10 years ago, and since this time, the technology involved in their production has moved forward significantly. Also, many properties of these materials have now been investigated with some properties being superior to the corresponding properties of the constituent metals, or more importantly, the same alloy in crystal form.

Of particular interest, related to their possible application as first mirrors in fusion reactors, is the response of AMAs to ion bombardment. Polycrystalline materials typically display a step-like structure, reflecting the differing sputtering yield associated with differing crystal orientations, e.g., [1–4]. In contrast, it was found that mirrors of the alloy Zr(41.2)Ti(13.8)Cu(12.5)Ni(10)Be(22.5) (Vitreloy-1) maintained their initial reflectivity, even after

$\sim 2 \mu\text{m}$  was sputtered by ions from a deuterium plasma [5]. This is evidence of a uniform erosion process associated with the lack of a grain sub-structure. Other properties of AMAs related to ion bombardment, such as ion trapping, sputtering rates, sputter-induced surface morphology, etc., have received little attention so far. The objective of the present letter is to describe unusual blister-like surface features observed following ion bombardment of Vitreloy-1 mirror specimens.

### 2. Experiment

AMA mirror specimens (22 mm in diameter) were mechanically polished to optical quality with gradually decreasing size of abrasive polish. After an acetone rinse, specimens were cleaned by exposure to low-energy (50–60 eV) deuterium ions produced by a electron-cyclotron resonance deuterium plasma ( $T_e = 3\text{--}5 \text{ eV}$ ,  $n_e = 10^{10} \text{ cm}^{-3}$  [4]) with  $\text{D}^+$ ,  $\text{D}_2^+$  and  $\text{D}_3^+$  as the primary plasma components. After the initial optical and surface characteristics

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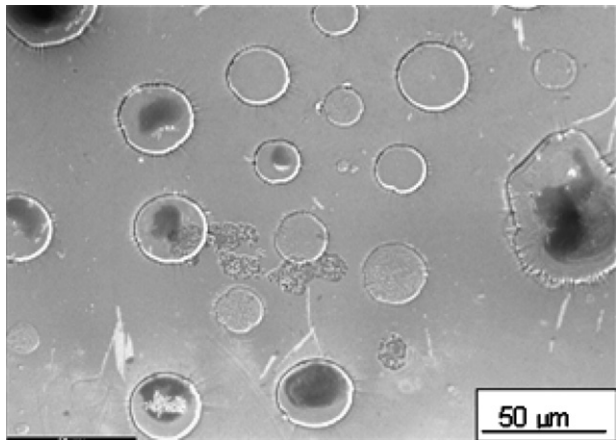


Fig. 1. The SEM photo of the AMA mirror surface with characteristic surface features. The roughen parts seen a little below the center are the structure defects described in [5].

of energy 0.3, 0.6, 1.0 or 1.35 keV, and current densities  $\sim 2 \text{ mA/cm}^2$ . After various ion fluences, surfaces were analysed by scanning electron microscopy (SEM) and atomic force microscope (AFM).

### 3. Results

Following exposure to 300 eV ions (fluence:  $2.4 \times 10^{20} \text{ ion/cm}^2$ ), many small surface features were observed on the very smooth specimen surface, see Fig. 1. These features covered approximately 15% of the irradiated surface, and generally had a round shape. The size (diameter) distribution was obtained by measuring the dimensions of 617 such features observed in the view field of the microscope,  $\sim 0.4 \text{ mm}^2$ . The mean diameter was  $\sim 10.5 \mu\text{m}$  with sizes ranging from  $\sim 3 \mu\text{m}$  up to  $60 \mu\text{m}$  (one subject).

Several peculiarities of these surface features are noted: (i) their location on the surface appears to be random, not depending on the presence of defects in the AMA described in [5]; (ii) around practically every feature, there is evidence of stress-induced ductile deformation; both of these properties are clearly distinguishable in Fig. 1; (iii) some of the features have flat 'lids' which show up with little contrast in the SEM photographs (Fig. 1), while others have a dark region on the lid; (iv) the inspection of several hundred features did

of the specimens were measured, the specimens were subjected to bombardment by non-mass-separated plasma ions accelerated by a negative potential applied to the specimen. During bombardment, specimens were kept at  $\sim 300 \text{ K}$  by a water-cooled copper specimen holder. Specimens were bombarded through an 8 mm aperture with ions

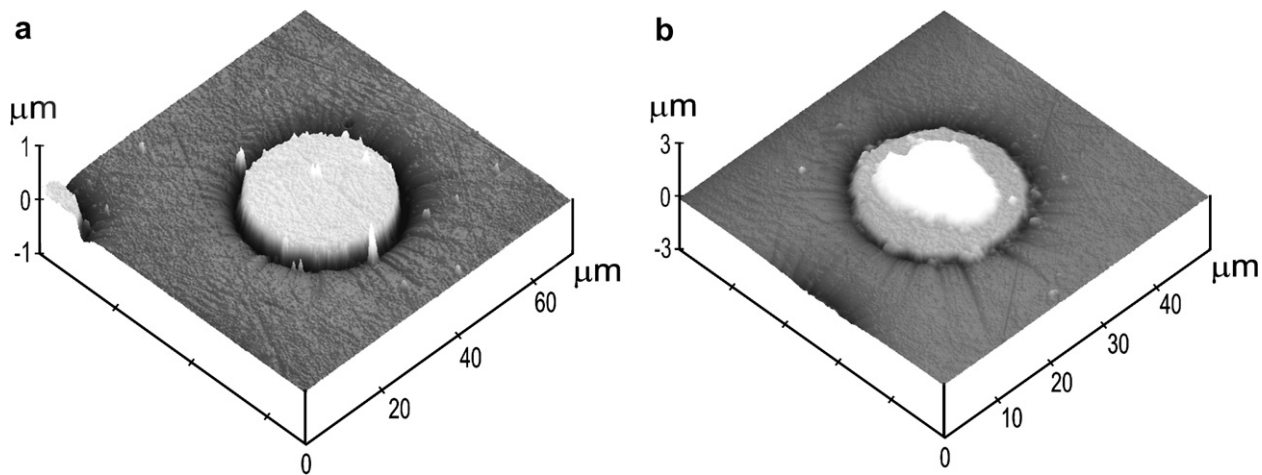


Fig. 2. The AFM images of two different kinds of features: (a) with flat lid and (b) with a dome-like lid.

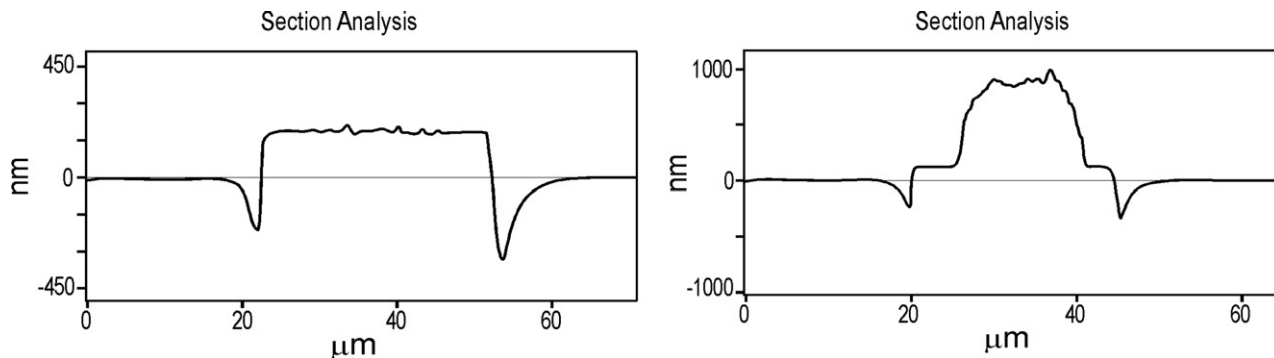


Fig. 3. Cross-sectional profiles of the surface features shown in Fig. 2.



Fig. 4. SEM photo of a feature with a dome-like lid.

not result in the observation of any cavities such as might be found following the bursting of blisters.

Fig. 2 shows AFM images of the two different types of surface features and Fig. 3, the corresponding cross-section profiles. The height of the flat-topped feature (Fig. 3(a)) is  $\sim 0.2 \mu\text{m}$  and it is surrounded by an annular groove of similar depth. A surface feature of similar size, but with a black central area (Fig. 1) is shown in Figs. 2 and 3(b). This time, there is an irregular-shaped dome on a flat lid base; the dome is seen as the black color in Fig. 1. Compositional SEM photographs suggest that the elemental composition of the dome differs from that of the main AMA matrix; however, microprobe analysis indicates a similar composition to the matrix. SEM photographs with higher magnification, Fig. 4, may indicate a more porous structure for the dome, and this may be affecting the SEM images.

#### 4. Discussion and conclusions

The saturation concentration of hydrogen in AMAs is high [6–8], significantly exceeding that for the crystal state of the same material (e.g., factor 17.6 for the Ti(60)Zr(15)-Ni(15)Cu(10) alloy [7]); therefore, we consider blistering to be an unlikely event.

Previously, the appearance of blisters was observed on metallic glasses of several kinds and their crystal analogues following bombardment with  $\text{H}^+$  and  $\text{He}^+$  ions of energies up to 100 keV [9–11]. It was found that on such amorphous materials, blisters appear at ion fluences approximately three times greater than for the same materials in crystal-line state. For these materials, the blisters had dome-shaped lids with diameters in the range 0.5–2.5  $\mu\text{m}$  (mean value 1.25  $\mu\text{m}$ ) for  $\text{H}^+$  ions [11]. These dimensions correlate well with the implantation range of the  $\text{H}^+$  ions, thus suggesting the formation of ‘classical’ blisters [12] due to accumulation of hydrogen in the near-surface region.

In the present case, the projective range of 300 eV  $\text{D}^+$  ions is  $\sim 6 \text{ nm}$ , so the size of ‘classical’ blisters would be much less than 1  $\mu\text{m}$ . This difference in size, along with the atypical shape (like flat lids, annular grooves, the lack of broken lids, etc.), indicate that the observed features are not blisters in common understanding of this word [12].

It is known from theory [12] that the process of blister formation is strongly influenced by material properties, such as bulk structure and elastic modulus. However, below the crystallization temperature, AMAs are found to deform by a process similar to viscous flow [13]. For example, according to [14] the fracture toughness of Vitreloy-1 was significantly higher in the amorphous state than after crystallization. We note that the characteristic stretching around the surface features of the current specimens is indicative of high ductility, but it is unclear what mechanism is leading to these features being formed.

In this paper, we have presented for the first time observations of unique blister-like surface features appearing on the surface of amorphous metallic alloys following ion bombardment. Additional experiments will be necessary to provide a better understanding of the nature of these surface features, and to correlate their appearance with ion energy, ion fluence, sample temperature, etc.

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