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Self-organization of ripples on Ti irradiated with focused ion beam

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ABSTRACT

30 keV focused Ga^+ ions were used to raster the metallographically polished surface of commercially pure Ti (CP Ti) at various FIB incidence angles over a wide range of doses (10^{16}–10^{18} ions/cm^2) at room temperature. The sputtered surfaces were observed in situ using FIB imaging and later carefully characterized ex situ under scanning electron microscope (SEM) and atomic force microscope (AFM). Ripples were observed on the irradiated surfaces even at the normal FIB incidence angle. The ripple evolution is analyzed as functions of surface diffusion, surface crystallographic orientation, ion dose and incidence angle. It is found that the ripple orientation was progressively influenced by the ion beam direction with incidence angle increasing and in some cases curved ripples or fragmented rods viewed from different angles occurred at high ion doses. The morphological evolution from the well-developed straight ripples to the curved ones is never observed. The formation of ripples is attributed to the competition between the formation of ripples due to anisotropic surface diffusion and the formation of incidence-angle dependent ripples determined by Bradley–Harper (BH) model.

1. Introduction

Periodic height modulations can develop after off-normal incidence ion bombardment. Surface nanopatterning by ion beam irradiation may show properties of high technological interest and has attracted much attention. Ripples have been observed on various surfaces irradiated with ions at an off-normal angle, e.g., metals [1], semiconductors [2–5], and insulators [6]. Depending on the ion incidence angle, the ripple orientation can be either perpendicular or parallel to the projected ion beam direction. These results have been theoretically explained by linear instability caused by the competition between surface curvature dependent sputtering and smoothening due to surface diffusion [7]. However experiments on metals, such as Cu [8], Au [9], Ag [10] and Sn [11,12], show ripple formation not only at oblique incidence angle but also at normal incidence angle. At normal incidence, the features produced by ion sputtering reflect the surface symmetry and are aligned along energy preferred crystallographic orientation [13]. Off-normal sputtering generates ripples depending on crystallographic orientations [14]. In contrast, the ripple direction depends only on ion beam direction at grazing angle [14,15]. Both the substrate temperature [14] and dwell time [11] can be used to select and enhance certain diffusion processes and consequently to tune the final surface morphology. Surface anisotropy and Ehrlich–Schwoebel barrier are added to the linear model to account for some phenomena on metals [16]. Although the research in this field is intensive, the basic mechanism controlling these pattern formation processes remain poorly understood and warrants further efforts [17].

In this paper, pattern formation is investigated on ion irradiated Ti surfaces at room temperature. Compared with metals studied before, Ti has relatively high melting point and complicated nonsymmetrical crystal structure. Study on Ti is of significance to understand pattern formation on ion irradiated surfaces.

2. Experimental

The material used in the study was commercially pure titanium. The sample surfaces were metallographically polished and then irradiated at room temperature with 30 kV Ga^+ ion using the Micrion dual beam FIB system (model 9500EX). The current was fixed at 1 nA. The ion beam spot size was 60 nm in diameter at full width and half maximum (FWHM) at this current level. The beam spot overlap was 67% and the distance between two adjacent FIB spots was around 20 nm. Various FIB incidence angles (0°, 15°, 30°, 45° and 60°) were used to study the effect of incidence angle on ripple formation, and sputtering time was varied to investigate the evolution of surface morphology over a wide range of doses (10^{16}–10^{18} ions/cm^2). The irradiated surfaces were examined using scanning electron microscope (SEM) and atomic force microscope (AFM) (model Nanoscope IIIa). Tapping mode was used in all the AFM measurements to make sure the consistency and repeatability of the results.

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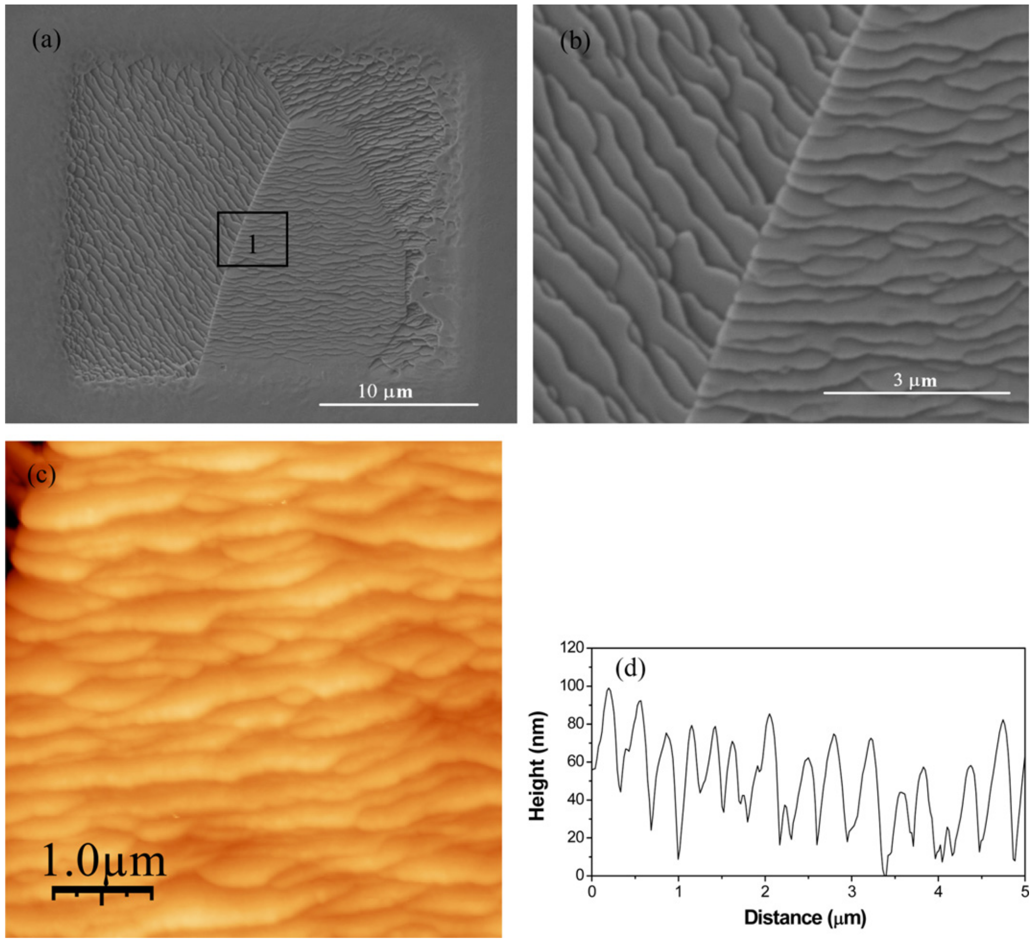

Fig. 1. Images showing ripple formation at normal angle (ion dose = 2.40×10^{18} ions/cm^2). (a) SEM image of irradiated area; (b) close-up; (c) AFM image of area 1 labelled in (a); (d) AFM line profile along the direction perpendicular to ripple direction shown in (c).

3. Discussion

3.1. Effect of surface diffusion on ripple formation

In contrast to usual expectation, ripples were observed forming at normal incidence angle in the experiments. Fig. 1(a) is an overall SEM picture of irradiated area. It is clear that the sputtered surface is composed of several parts with different morphologies. Each part corresponds to one grain. And the ripple orientation is corresponding to energy favoured crystallographic orientation. After irradiation, ripples formed in different grains are aligned along different orientations. Fig. 1(b) is a close-up image showing the grain boundary effect. Fig. 1(c) shows AFM images of area 1 labelled in Fig. 1(a). And the line profile along the direction perpendicular to the ripple orientation is shown in Fig. 1(d).

The melting point of Ti is 1660 °C. The ratio of substrate temperature versus melting point (15.5%) is low compared with the ratio calculated on Ag (18.6%) and Cu (18.4%). This might be due to the highly nonsymmetrical crystal structure of Ti (hexagonal close-packed). Anisotropic surface diffusion induces the ripple formation even at normal incidence angle and at relatively “low” temperature.

3.2. Effect of crystallographic orientation on ripple formation

Ripple-like structures were obtained on Ti surfaces at off-normal incidence, whose direction was found to be independent of ion beam direction. The surface morphology irradiated at 15° incidence angle is displayed in Fig. 2. The overall morphology of irradiated area is shown in Fig. 2(a). Dots and ripples are formed in different grains under the same sputtering condition. Seen from Fig. 2(b), the dots are decorated on the ridge-like ripples. The diameter of dots varies from 200 nm to 400 nm. Ripples forming in one single grain have the same orientation, but the directions are varied in different grains, as shown in Fig. 2(c) and (d).

All the facts shown above imply that crystallographic orientation plays important role in surface morphology evolution. After irradiation, dots were formed in the grain with certain crystallographic orientation, whereas ripple-like structures were formed in the grains with other orientations. The ripple direction is determined by crystallographic orientation rather than ion beam direction.

3.3. Effect of incidence angle on ripple formation

When the Ti surface was irradiated at normal incidence angle (see Fig. 1) or at a small incidence angle of 15° (see Fig. 3), the ripple orientation was determined by the crystallographic orientation rather than the ion beam direction. With incidence angle increasing, the role of incidence angle on ripple formation is more and more important. Fig. 4 shows surface patterns formed at 30° incidence angle at ion dose of 4.15×10^{18} ions/cm^2. It should be noted that the ripples in different grains orientate nearly perpendicular to the ion beam direction as predicted by the classical BH model. This fact reveals that ion beam direction plays greater role in the ripple direction rather than crystallographic orientation at this incidence angle. However, it should be noted that crystallographic