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Helium ion irradiation-induced microstructure evolution on the surfaces of thin nickel foils

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ABSTRACT

Polycrystalline nickel foil (sample I) with thickness of ~950 nm was irradiated with 0.5–1.2 MeV helium ions at room temperature. Another piece (sample II) with same thickness was mounted behind to receive the irradiation of transmitted helium ions (~0.026–0.537 MeV). Morphology evolutions on irradiated surfaces were investigated by scanning electron microscopy (SEM) and atomic force microscopy (AFM). Results show that the open cracks, which were located mainly at grain boundaries, occurred on the surfaces of both irradiated samples. Interestingly, ripple patterns were observed to be regularly arranged on the front surface of sample I. The compressive stress resulting in the sliding on close-packed (1 1 1) planes was regarded as the origin of the ripple formation. Moreover, protrusion islands and its surrounding microstructures were observed on the front surface of sample II. The mass transport driven by the lateral stress generated in the helium ion irradiation were discussed as possible reasons.

1. Introduction

Bombardment of solid materials with energetic ions is often used to modify surfaces [1,2], to change the state and the microstructure of surface layers [3,4], and to synthesize buried epitaxial layers [5,6]. In the regime of ion energies where electronic energy loss is dominant, several interesting features of solid modification have been observed [7–9]. A prominent effect was the formation of ripple patterns on irradiated surfaces. For example, ripples were observed on surface of amorphous materials that were irradiated by MeV xenon ions [7]. It was demonstrated that the prerequisite for the ripple formation is the irradiation at off-normal incident angle ($15^\circ < \theta < 20^\circ$). The shear flow, driving the transient mobile atoms that were generated along the passage of each fast-heavy ion, was explained as the key reason for the formation of observed mounds and valleys [7].

For metals, it was reported by Rusponi et al. that ripples can be produced by low temperature ion sputtering on surfaces of Ag(1 1 0) [9] and Cu(1 1 0) [10]. It was explained that the ripple phenomenon is related to the interplay between ion erosion and diffusion of adatoms (vacancies), which induces surface re-organization [11]. The conditions for ripple formation varied with different materials, ion species, energies and fluences. As reported so far, most of ripples were induced by heavy ions, for instance, Ar^+ [9,10,12–17], Si^+ [8] and Xe^+ [7]. Light ions, such as helium ions, were usually not considered in the study of surface modification of materials, since their ability of sputtering was limited. The irradiation effects on surfaces by light ions were always neglected especially in the MeV energy range, due to the relatively weak interaction between the ions with target atoms at surface.

In this study, we report that ripple patterns can be also induced by helium ions in the energy range of 0.5–1.2 MeV on the surface of polycrystalline nickel foil. It was demonstrated that the origin of their formation differs from those that have been reported so far. For the specific case of irradiation using helium ions, it was often reported that surface layers can be deformed by blisters [18–21], or even removed by exfoliation process [22,23]. The present study shows some new features associated with the formations of open cracks and protrusion islands.

2. Experimental procedure

The samples used in this study are well-annealed polycrystalline nickel foils (99.95 wt%) with thickness of ~950 nm, which were provided by the Goodfellow Cambridge Limited, UK. A sandwich-like sample holder was designed using three pieces of stainless steel plates with a square opening (6 mm × 6 mm) in the middle, as schematically

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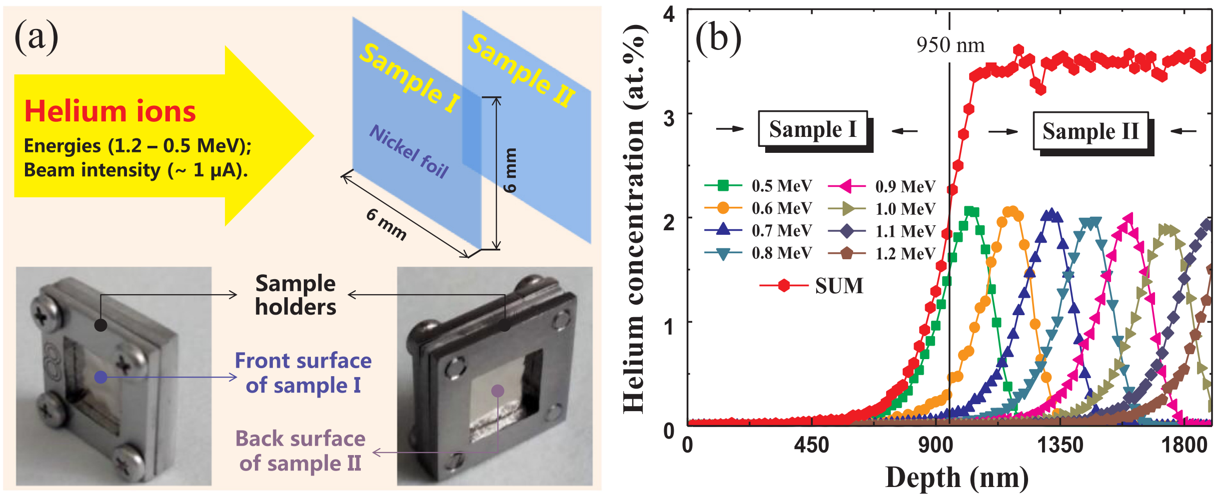

Fig. 1. Irradiation experiments for nickel foils. (a) Experimental geometry setup of helium ion irradiation and the sandwich-like sample holder. (b) The depth profile of helium concentration calculated by SRIM-2013.

shown in Fig. 1(a). Prior to the ion irradiation, a piece of nickel foil (sample I) was mounted in front of another nickel foil (sample II) between three pieces of the stainless-steel plates. That means both the front and back surfaces of each sample contacted with the stainless-steel plates, but the two samples did not contact with each other, as shown in Fig. 1(a). This experimental setup is used to investigate the effects of ion energies and helium concentrations on the evolution of surface morphology.

The samples were firstly irradiated by 1.2 MeV helium ions to the fluence of 6.75×10^{16} ions/cm^2 and subsequently to the same fluence of 4.50×10^{16} ions/cm^2 at the following energies of 1.1, 1.0, 0.9, 0.8, 0.7, 0.6 and 0.5 MeV, using a 4 MV Pelletron accelerator located at the Shanghai Institute of Applied Physics, Chinese Academy of Sciences (SINAP-CAS). The corresponding energies of helium ions penetrating through the sample I to reach the surface of sample II were estimated to be ~537, 443, 353, 266, 190, 121, 67 and 26 keV, respectively, according to the calculations using the SRIM-2013 code [24]. The irradiation adopts a scanning mode, in which the ion beam with a spot size of ~2 mm in radius covers the whole sample surface in a scanning cycle. The scan frequencies were 39.17 Hz in the X direction and 17.13 Hz in the Y direction. These parameters were selected to ensure the irradiation uniformity in the sample. The irradiation was performed at room temperature and the ion beam intensity was kept ~1 μA during the irradiation. At the back side of sample holder where the thermocouple was located, the temperature was continuously monitored (< 33 °C). In consideration of the scanning irradiation and relatively low beam intensity, the real temperature on sample surface may be slightly higher than 33 °C. The depth profile of helium concentration was calculated using SRIM-2013 software, as shown in Fig. 1(b). It is clear that little helium ions stopped at the front surface of sample I, whereas the helium amount at the front surface of sample II was much higher with a concentration higher than 2 at.%.

3. Results and discussion

3.1. Open cracks and protrusion islands

Fig. 2 shows the comparison of morphologies between the pristine surface and the front surface of sample I, which was irradiated by 0.5–1.2 MeV helium ions. It can be noticed that the grain boundaries (GBs) can be roughly identified by the difference in image contrast between neighboring grains, as shown in Fig. 2(a) and (b). Obviously, several open cracks formed on the irradiated front surface and they are mainly located at GBs, as indicated by the circles in Fig. 2(c). Another interesting feature is the formation of ripple structure (indicated by the parallel lines in Fig. 2(c)), which will be discussed in the following section.

As calculated by the SRIM code, the energies of helium ions reaching the surface of sample II were in the range of 0.026–0.537 MeV. It was found that similar crack structures also occurred on the front surface of the sample II, as shown in Fig. 3(a). In addition, some small protrusion islands were detected by AFM experiments, as shown in Fig. 3(b). The cross-section views in Fig. 3(c) and 3(d) revealed that the nominal height of these structures was ~6 nm, with average size of ~120.9×68.4 nm^2, which approximately agrees with the size of the structures indicated by the arrows in Fig. 3(a). Another interesting feature observed is the formation of 3-4 circles of waves around the protrusion structures (Fig. 3(b)). Moreover, some low-lying areas surrounded by these waves were also detected with nominal height varying from ~−4 nm to ~−8 nm. It seems that the protrusion islands formed at the places where open creaks have occurred. Such cases can be clearly found on the irradiated surface, as indicated by the circle in Fig. 3(a).

The open cracks and protrusion islands found in present work were much like the exfoliation and blisters structures, which were widely reported in previous studies using helium ion irradiation [18–21]. However, it should be mentioned that the formation of helium blisters on implanted surfaces generally take place at peak helium levels of more like 30 at.%. In the present work, the helium concentration at the front surface of sample I was rather low (Fig. 1(b)). Even at the front surface of sample II, the concentration (~2 at.%) was still much lower than the value that was needed for the blister formation. Therefore, blisters and exfoliation cannot occur in present study.

It was known that ion implantation can introduce defects (by displacement damage) together with the injection of a large number of foreign (helium) atoms into thin layers. Hence, the surface stress would be generated in both samples due to the volume expansion. It was reported that mass transport can occur through the formation of island structures because of the stress relaxation during the ion irradiation at room temperature [8]. The mass transport has been previously explained by momentum transfer from ion beam to solid targets, and this effect would become prominent during heavy ion irradiation with ion energies exceeding a few MeV [25]. Although the ion irradiation used in this study was not the case of heavy ion irradiation with ion energies in MeV range, considerable stress can be still introduced into the sample by the aggregation of implanted helium ions. Here, a deduction can be given that the mass transport, driven by the lateral stress generated in the helium ion irradiation, play a role in the formation of