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Surface Nanofeatures Induced by High-Energy Heavy Ions Irradiation

A. GARCÍA-BÓRQUEZ^{a,*}, C. CAMACHO-OLGUÍN^a,
L. HERRERA-COLÍN^a, G. RUEDA-MORALES^a
AND W. KESTERNICH^b

^aESFM-IPN, 07738 México D.F., Mexico

^bIFF-FZ Jülich, Germany

During neutron, ions or electron irradiation of materials, the surface morphology can be microroughened in different forms. Using a tandem accelerator with high current capability, 3.66 MeV Ni and Al ions were implanted into Ni-22at%Si alloy at 650°C and into α -Al₂O₃ at 1000°C, respectively. Scanning electron microscopy observation revealed surface nanofeatures induced by the irradiations. On the Ni-Si alloy, long parallel channels are formed having a periodicity near to 2 μ m and a mean depth of 30 nm (atomic force microscopy measurements). On α -Al₂O₃ nanopyramides with around 50 nm basis length and similar height were detected on some grains forming periodic chains. We assume that preferential sputtering produced both induced surface features.

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1. Introduction

Understanding surface microerosion in materials caused by particle bombardments is important for near surface studies on radiation damage of materials. It is further of high interest in studies using particle bombardment as means of analytical techniques such as the Auger spectroscopy, or the Rutherford backscattering. A new, exciting area, which is recently opening up, is the controlled production of surface nanofeatures and their correlation with surface property changes. Special interest began about ten years ago in the quantum confinement effects studied in objects of nanometric size [1–3], and also in Ni-based nanostructures [4].

With respect to Ni-Si alloys, no reports were found, relating to radiation induced surface nanofeatures. Tanaka et al. reported [5] on effects at the surface area surrounding the area bombarded by a 25 keV Ga⁺-focused ion beam on Ni₂Si/Si thin films, but did not report on the changes within the irradiated area.

*corresponding author; e-mail: borquez@esfm.ipn.mx

Most of the works on irradiated alumina up to now have reported on bulk changes such as macroscopic swelling, vacancy formation [6, 7], phase transformation [8], crystallization [9], amorphization [10], ion track formation, ionizing effects [11], etc. There is only very scarce information concerning the surface changes caused by irradiation with energetic particles. Nakao et al. [9] have shown atomic force microscopy (AFM) images on alumina films before and after 1.5×10^{17} Si⁺/cm² implantation, revealing granular features caused by the irradiation. Auciello and Kelly [12] describe pyramid as termed, regular and crystalline feature forming on high sputter yield materials, such as Cu, Au, Ag, Al, and Pb [12]. A more recent report by Skuratov et al. [13, 14] describes nanostructures, which were induced by 245–710 MeV Bi and Kr ions, as hillock-like defects.

2. Experimental

Using a high intensity ion beam tandem accelerator, 3.66 MeV Ni and Al ions were implanted in a Ni-22at%Si alloy at 650°C and in α -Al₂O₃ at 1000°C, respectively. The respective specimen temperatures were achieved by resistance heating in the first case, and by radiation with a halogen lamp, in the second case. The temperatures were held constant within 10°C using an infrared pyrometer focused (1 mm in diameter) at the middle of the irradiated area (6 mm in diameter). All experiments were carried out under a 10^{-7} mbar pressure [15]. The samples were irradiated with ion fluences in the range of 10^{17} – 10^{18} ions/cm², up to extremely high dose: 100 dpa for NiSi and 250 dpa for α -Al₂O₃. To study the radiation-induced features (RIF) at the surface of the studied materials, several surface techniques were employed. In particular, direct observation using scanning electron microscopy (SEM) and atomic force microscopy (AFM) were carried out. For the SEM measurements, quite different conditions were necessary to visualise the RIF at Ni-Si and α -Al₂O₃. For Ni-22at%Si, long work distance (WD) and high accelerating voltage, i.e. 12–14 mm and 20 kV, respectively, were used to discern the presence of channels. Whereas for α -Al₂O₃ very short WD and low accelerating voltage, i.e. 2 mm and 1 kV, respectively, were very successful to obtain high resolution without charging effect (no conductive recovering was necessary). In AFM, a standard cantilever working with 0.05 N/m, 22 kHz and, a 3 μ m–35° pyramidal tip, were used in a non-contact mode.

3. Results

SEM observation gives evidence for surface nanostructures induced by the ion irradiation at fluences between 10^{17} – 10^{18} ions/cm². On the surfaces of the Ni-22at%Si alloy, long parallel channels (Figs. 1a, b) are formed having a periodicity near to $\lambda = 2 \mu$ m and a mean depth of 30 nm as measured by AFM (Fig. 1c). On the surface of α -Al₂O₃, nanopillars, with around 50 nm basis length and similar height were detected on some grains. The pillars tend to be arranged in periodic chains (Fig. 2).

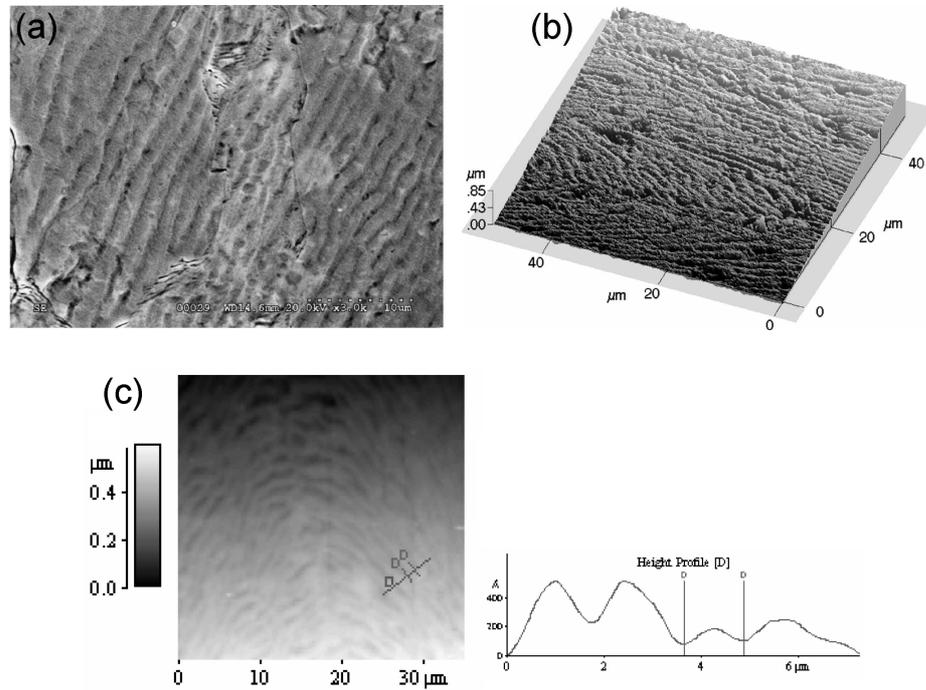


Fig. 1. (a) SEM micrograph showing a groove pattern preferential sputter to a fluence of 10^{17} – 10^{18} Ni ions/cm² at 650°C from a mirror polished Ni-22at%Si surface. (b) AFM 3-dimensional image of a groove pattern. (c) Image and diagram as an AFM measurement of periodicity is taken. Similar process is followed by depth measurement.

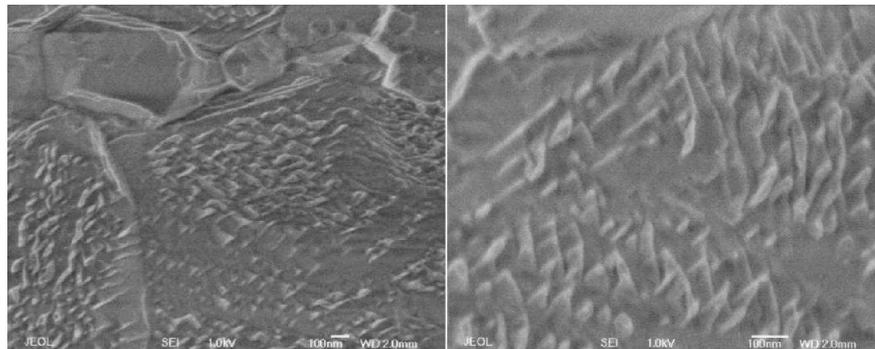


Fig. 2. SEM micrographs showing details of the nanopylramids (50 nm) after 250 dpa dose induced by 3.66 MeV Al ions at 1000°C.

4. Discussion

The Ni-22at%Si alloy presents lamellar structure before irradiation as expected for an eutectic composition and confirmed by metallographic test. X-ray

diffraction (XRD) revealed the lamella to consist of disordered Ni–Si and Ni₃Si phases, respectively. We assume that the 3.66 MeV Ni ion bombardment produces channels by sputtering preferentially one of the two phases, presumably the disordered Ni–Si phase. Supporting these considerations is the fact that this phase has more abundance of the weak Ni–Ni metallic bonds, because it is richer in Ni than the ordered Ni₃Si phase. Similar periodical ($\lambda = 700$ nm) features, forming subtle ripples on (111) surfaces in Ge during etching by 5 keV Xe ions, have previously been reported [16]. It was concluded by the authors that the curvature dependence of the sputtering yield underestimates the observed periodicity and that the possibility of preferential sputtering of step-edge atoms as a function of step orientation need to be considered.

The sharp, faceted pyramids and their periodic arrays, induced by 3.66 MeV Al ion bombardment of α -Al₂O₃, is thought to be due to crystallographic preferential sputtering (CPS) taking place during irradiation. Features similar in form were reported as sputter cones formed on Cu surfaces by 1 keV Ar⁺ bombardment at 300°C at a fluence of 2×10^{18} ions/cm², but they had around 700 nm basis length and 1 μ m height and were formed as result of an impurity-initiated mechanism owing to the presence of W surface impurities [17]. Features similar in size were reported as hillocks formed on the surfaces of α -Al₂O₃ and MgO irradiated with high energy Kr and Bi ions, and have been suspected to form by plastic deformation due to defects created by the Coulomb explosion mechanism in the target subsurface layer [13, 14].

5. Conclusions

1. Both observed grooves at Ni–22at%Si alloy and nanopyramids at α -Al₂O₃ are radiation-induced features.
2. Our RIFs were produced by preferential sputtering (PS) by two different mechanisms not reported up today in these materials.
3. PS has left grooved patterns on the Ni–22at%Si surface by eroding faster one of the two-phases forming the eutectic crystallisation structure.
4. The long parallel channels forming the grooved patterns have a periodicity near to 2 microns and a mean depth of 30 nm.
5. In α -Al₂O₃, sharp faceted pyramids sometimes aligned in periodic chains, have been formed by CPS.
6. The nanopyramids show projected basis, height and top angle between 40–100 nm, 60–90 nm and 55–75°, respectively.

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References

- [1] K.F. Braun, K.H. Rieder, *Phys. Rev. Lett.* **88**, 096801 (2002).
- [2] S.M. Pons, J.Y. Veuillen, *Phys. Rev. B* **64**, 193408 (2001).
- [3] F.M. Leisble, *Surf. Sci.* **514**, 33 (2002).
- [4] J.Y. Veuillen, P. Mallet, L. Magaud, S. Pons, *J. Phys., Condens. Matter* **15**, S2547 (2003).
- [5] M. Tanaka, K. Furuya, T. Saito, *MRS Symp. Proc.* **438**, 313 (1996).
- [6] R.A. Youngman, T.E. Mitchell, F.W. Clinard, G.F. Hurley, *J. Mater. Res.* **6**, 2178 (1991).
- [7] G.V. Kornich, G. Betz, A.I. Bazhin, *Tech. Phys. Lett.* **26**, 445 (2000).
- [8] C. Kinoshita, Y. Tomokiyo, K. Nakai, *Ultramicroscopy* **56**, 216 (1994).
- [9] S. Nakao, P. Jin, G. Xu, M. Ikeyama, Y. Miyagawa, *J. Crystal Growth* **237-239**, 580 (2002).
- [10] C.J. McHargue, P.S. Sklad, J.C. McCallum, C.W. White, *Mater. Res. Soc. Symp. Proc.* **157**, 555 (1990).
- [11] S.J. Zinkle, V.A. Skuratov, D.T. Hoelzer, *Nucl. Instrum. Methods Phys. Res. B* **191**, 758 (2002).
- [12] O. Auciello, R. Kelly, *Ion Bombardment Modification of Surfaces*, Elsevier, New York 1984, p. 301.
- [13] V.A. Skuratov, S.J. Zinkle, A.E. Efimov, K. Havancsak, *Nucl. Instrum. Methods Phys. Res. B* **203**, 136 (2003).
- [14] V.A. Skuratov, S.J. Zinkle, A.E. Efimov, K. Havancsak, *Surf. Coat. Technol.* **196**, 56 (2005).
- [15] A. García-Bórquez, Ph.D. thesis, Instituto Politécnico Nacional, México 1994, p. 20.
- [16] G.C. David, R.S. Averback, *Phys. Rev. B* **67**, 045404 (2003).
- [17] R.S. Robinson, S.M. Rossnagel, *J. Vac. Sci. Technol.* **21**, 790 (1982).