Effects of Low Ion Dose on SE Imaging and Orientation Dependent Ga-ion Channeling

Laxmikant V. Saraf

Electron Microscopy Laboratory, Clemson University, Clemson SC 29634 USA

ABSTRACT

Grain contrasts produced during ion induced secondary electron imaging depend upon grain orientation and ability of Ga-ion beam to channel in it. The current study aims to investigate critical Ga-ion dose for Ni-alloy where a transition from grain surface imaging to sputtering dominated Ga-ion channeling occurs. It is observed that critical dose is in the vicinity of 10^{15} ions/cm². Strong orientation dependent Ga-ion channeling effects were observed at dose values higher than 10^{17} ions/cm². When a normalized orientation dependent grain transparency factor was estimated for each grain in a limited sampling volume, it was observed that despite low sampling volume for a random polycrystalline orientations and variation in Ga-ion dose, the range of sputter depth variation during Ga-ion channeling remains above 80% indicating dose or sampling volume (for randomly oriented poly-crystals) independency in the sputter dominated dose region. This study attempts to establish a correlation between high resolution imaging, applied Ga-ion dose and orientation dependent Ga-ion beam channeling effects.

Keywords: Ga-ion channeling, Sputtering, EBSD, Grain orientation

INTRODUCTION

During SEM imaging, primary energetic electrons interact with the sample surface producing the secondary electrons. Secondary electrons could form the weakly bound conduction electrons from the target material with binding energies in the range of 2-5 eV. Typically, secondary electrons are identified with those energies below 50 eV. They escape from a very narrow surface depth region (of few nm) depending upon the primary electron beam energy. The secondary electrons can also be produced by backscatter electrons. Backscatter electrons could be generated from the depths as below as 1-2 microns in the primary electron beam energy range of 50 eV-30 keV. The reason backscatter electrons can reach energies as high as energy of primary incident electron beam is because the coulomb interaction with nucleus of target material results elastic diffusion with no energy transfer.

When ion-solid interaction is considered, interaction of energetic ions with solid surface often result in multiple reactions with emission of secondary electrons, secondary ions, ion induced X-ray emission, deposited metal atoms and sputtering of secondary (charged/uncharged) particles [1, 2]. Focused ion beam has been widely used in our past studies where surfaces were modified through ion-solid interaction [3-7]. The image contrast produced due to grain channeling is different than image contrasts observed during electron backscattering. Grain contrasts produced during electron backscattering are due to atomic mass dependent elastic scattering effects that emit backscattered electrons with a varying intensity. They provide information about chemical differences. Grain contrasts produced during ion induced secondary electron (secondary ion microscopy, SIM) imaging are due to ion beam channeling in those grains whose one of the crystallographic axis or atomic planes align well with in incident ion beam direction resulting partial or complete ion channeling. As a result, there is a reduction in electron backscattering. A

simple way to confirm if the imaging contrasts are due to ion channeling or differences in material chemistry is by changing incident ion beam angle (sample tilt). Grain contrast effects due to ion channeling are sensitive to incident ion beam angle whereas contrasts produced due to chemical differences during electron backscattering are relatively independent of incident beam angle. With the application of additional ion dose, the effects can be separated. Additional ion dose leads to excess sputtering from the sample surface. In this study, it is confirmed that by imaging the orientation dependent sputtering rates of individual grains from Ni-alloy, grain contrasts produced during SIM are due to orientation dependent grain channeling effects.

Using techniques like electron backscatter diffraction (EBSD), it is possible to accurately determine the orientations of channeled grains [3, 8-10]. EBSD combines ability to measure site specific grain orientation along with high resolution imaging. Slow sputtering process from some grains due to channeling, phase change or existence of surface layer can be accurately imaged with their orientation information. The critical ion dose, where a transition from imaging surface with and without sputtering effects depends upon various factors such as binding energy of surface atoms, type of surface (dense, porous, amorphous), ion beam energy, mass of ion and beam angle of incidence. At the lower ion beam energy, for the surface receiving ion dose, existence of surface layer could be an additional factor to influence such transition. Therefore, current investigation aims at establishing a correlation through microscopic analysis methods about the optimum Ga-ion dose required for a transition from purely imaging to sputter dominated imaging conditions. Because such studies constitute to low energy and low dose ion implantation region, effect of surface layer on the transition was also taken in to consideration. To better understand such effects and minimize multiple variables, Ga-ion beam dose receiving sample surface area, beam energy were kept unchanged and Ga-ion current density and the total

dose were varied. Strong orientation dependent Ga-ion channeling effects are directly imaged at high spatial resolution. It is observed that despite of variations in total Ga-ion dose, normalized orientation dependent grain transparency factor estimated for each grain and the degree of depth variations for each case stays high ruling out ion beam dose dependency in the sputter dominated region. These studies provide a direct correlation between orientation dependent ion channeling effects, applied Ga-ion dose and high resolution imaging for low dose conditions.

Anisotropic milling of Cu was studied during FIB milling process and the results were correlated to SEM, FIB and EBSD to better understand orientation dependent milling properties to generate surface topographies with specific properties [11]. Apart from beam energy and material density, angle of the Ga-ion beam with respect to the crystal axis is one of the most influential factors detecting the sputtering yield. During sputtering experiments, the direction dependent effects of a charged particle were observed for the first time by Rol et; al. [12]. The topic of ion beam channeling at low [11], medium [13] and high beam energies [14] and overall ion-solid interactions are well discussed in the literature. It was noted that for ion beam energies below 10 keV, the variations in sputtering yield alone cannot be predicted by traditional sputtering mechanism [15]. The displacement of atoms from their ideal positions in rows and columns could prohibit ion channeling. Recoiling of the channeled ions early on into the directions other than channeled direction reduces the channeling depth. In the current study, usage of higher beam energy than such a threshold energy ensures higher crystal orientation dependence on sputtering yield. At medium beam energies in the range 20-100 keV, critical channeling angle was defined by Lindhard et; al. [16(a-d)] as ..

$$\Psi_2 = [a\sqrt{3}/d\sqrt{2} (2Z_1Z_2e^2/dE)^{1/2}]^{1/2} \text{ for } \Psi > a/d$$

Where Ψ is the angular width, Z_1 and Z_2 are atomic numbers of ion and sample target material, 'a' is Thomas-Fermi screening length and d is the atomic spacing along the crystal axis. The beam angle of incidence can be estimated and changed depending upon crystal orientation to achieve channeling along specific planes.

It is important to know that secondary electrons are also generated as a result of primary Ga-ion beam interaction with target material. Compared to electron beam as a source, the primary Ga-ion beam does not excite backscatter electrons and the yield of the secondary electrons is directly proportional to the Ga-ion beam dose [17]. Because of heavier ion beam, the yield of secondary electrons in this case has crystal orientation dependence and brightness and contrasts in imaging are associated with degree of ion channeling.

EXPERIMENT

A focused Ga-ion beam scanning electron microscope (FIB-SEM) Quanta 3DFEG from FEI, Inc. was used for Ga-ion beam sputtering and SEM imaging analysis. In this dual-beam microscope, Ga-ion beam column is tilted at 52° w.r.t. vertical electron beam column. SEM images were recorded using forward scatter secondary electron detector. Ring shaped craters were milled on highly polished Ni-alloy (Ni, Cr, Fe) surfaces using Ga-ion beam deposition at various doses by tilting the sample stage to 52° with incident Ga-ions normal to the sample surface. The craters with 50 μm outer diameters and 10 μm inner diameters were formed after a total dose of 4.18×10^{16} ions/ cm^2 , 2.09×10^{17} ions/ cm^2 and 4.18×10^{17} ions/ cm^2 , respectively. The FIB energy during implantation was 30 kV @ 7 nA with a beam dwell time of 1 μs and volume per dose of $0.15 \mu\text{m}^3/\text{nC}$. The total Ga-ion dose was estimated using the formula..

$$\text{Ion Dose} = I_{\text{ion}} \times \text{time}_{\text{exposure}} / \text{Pattern Area} \times 1.602 \times 10^{-15}$$

Where I = ion current in pA, exposure time in seconds and crater area is considered in μm^2 . Because total sputtered area for all three craters was unchanged, average sputter depths were higher as a function of increase in Ga-ion dose. Maximum sputter depth for three craters with increase in Ga-ion beam doses were 23 nm, 66 nm and 146 nm, respectively. Actual sputter depths were measured using atomic force microscopy (AFM) in a tapping mode configuration with silicon cantilever tip on a Digital Instruments nanoscope III microscope. Other details of FIB sputtering experiment are shown in Table 1. The EBSD analysis on Ni-alloy was carried out using Hikari high speed camera from EDAX/TSL and by using OIM interface from EDAX. During EBSD measurements, the electron beam energy was maintained at 30 kV @ 4 nA. The measurements were done at 100 frames/sec with step size of 150 nm. Post data processing was done using OIM 5.0 interface from EDAX/TSL. For Hough pattern optimization, a classic algorithm was used with a 9 x 9 convolution mask. The binned pattern size was 96 with theta step size 2° . SIM measurements were done using Ga-ion beam source on Quanta 3D FEG microscope from FEI, Inc. The Ga-ion beam energy during imaging was 30 kV @ 1.5 pA.

RESULTS AND DISCUSSIONS

Minimum ion dose required for this study to remove natural surface contamination layer and image Ni-alloy grains was estimated using ion implantation at various beam currents. The set of images shown in Fig 1 (a-d) are time lapsed SIM images after Ga-ion beam exposure to the sample surface within first few milliseconds when sample surface was exposed to ion beam, when channeling effects are first observed and when channeling effects became clearly distinguishable. Ga-ion beam current for the four set of images in Fig. 1 (a-d) are 30 pA, 50 pA, 100 pA and 300 pA, respectively. Locations marked by arrows from middle column images show appearance of Ga-ion channeling contrasts near grain boundary regions. This stage also

indicates complete removal of natural surface contamination layer. Due to affinity with oxygen and exposure to atmospheric contaminants (mainly hydrocarbons), it is well known that air exposed metallic surface develop surface oxidation and contamination layer of few angstroms in thickness. A dose of 1.6×10^{15} ions/cm² at 300 pA removes contamination at the highest sputter rate. Since the maximum sputter depth is 23 nm for a dose of 4.2×10^{16} ions/cm², a dose of 1.6×10^{15} ions/cm² correspond to 0.88 nm material removed from the surface which is a reasonable thickness estimate of a contamination layer. Removal of surface oxide and/or contaminants are predicted from the presence of uniform amorphous surface layer prohibiting imaging of individual grains with defined boundaries, as observed from the areas surrounding circular crater. Graph in Figure 2 show total ion dose and time as a function of Ga-ion beam current to observe transition to SIM imaging with grain channeling. Because all parameters, including ion beam dwell time are same for all cases, time required to achieve ion dose for a transition to channeling effects will reduce with increase in ion beam current. Results indicate that for Ni-alloy in this study, ion dose of $1-2 \times 10^{15}$ ions/cm² is needed to start imaging grains with channeling effects. Low sputtering rates from crystalline metallic surface for the selected range of ion dose cause material removal at a reduced rate. Amorphous oxide and contaminated surface layer is expected to go through high sputtering rates for the same dose parameters. At low ion beam energies and low ion current densities, a difference in material sputtering rate due to changes in material density, bond energy, porosity and crystal ion channeling are expected [5, 11, 18, 19]. At high ion beam energies and low ion current densities, in addition to crystal ion channeling, reduction in number of energetic ions that cause knockout effect in sputter process also result in material removal with a reduced amount.

Within bulk crystalline Ni-alloy, assuming that there are no significant variations in the binding energies, material density or porosity, variation in sputtering rates can be directly correlated to Ga-ion channeling effects into the crystals. If variations in sputtering rate are indeed due to Ga-ion channeling effects, depending upon their crystal orientations, different grains should sputter out at different rates. This is assuming that single grain has single crystal orientation, the most likely case for high crystalline alloys. It is also expected that within a single grain of any orientation, the sputter rate should be highly uniform. To prove these effects, Ni-alloy samples were exposed to additional Ga-ion beam dose. AFM images shown in Figure 3 (a-c) represent craters created in Ni-alloy after Ga-ion beam implantation at 4.18×10^{16} , 2.09×10^{17} and 4.18×10^{17} ions/cm², respectively. Minimum depths for all three dose values were 2 nm, 11 nm and 7 nm, respectively. Maximum depths for all three dose values were 23 nm, 66 nm and 146 nm, respectively. From highly uneven sputtering rates, it can be concluded that strong Ga-ion beam channeling effects were dominant. Without significant thickness variations within a single grain surface, it can be concluded that the sputtering rate was uniform within a single crater (crystal). It is also observed that craters created during Ga-ion beam sputtering possess distinct shapes which are confirmed during EBSD analysis as individual grains with a unique single orientation.

The surface morphology related information in image quality maps during EBSD is more dependent on quality of crystalline surface than SEM and SIM imaging. Changes in the crystalline quality result in variations in the pattern intensity. Therefore, amorphous regions are not represented during EBSD image quality maps. Because SEM and SIM do not have significant dependence on the target material crystallinity, amorphous materials are imaged well. Detailed EBSD analyzes were performed to confirm unique grain orientations within FIB sputtered craters. A set of images shown in Figure 4 (a-e), Figure 5 (a-e) and Figure 6 (a-e) relate

to results for the craters created using dose, 4.18×10^{16} ions/cm², 2.09×10^{17} and 4.18×10^{17} ions/cm², respectively. The micrographs in (a-e) are SIM, Inverse pole figure (IPF) map, top view SEM, AFM and angular view SEM, respectively. For comparison purposes, AFM images in Figure 4(d), 5(d) and 6(d) are reproduced from AFM images in Figure 3. Unique colors in IPF map indicate single grain orientation. The shapes of these grains match very well with contrasts seen in SIM imaging as those contrasts represent Ga-ion channeling effects. Brighter gray colors in SI imaging represent more backscattering and should correlate to grain orientation misalignment with incoming Ga-ion beam that lead to more sputtering (darker regions in AFM). Relatively higher depths were measured at these locations. Similarly darker regions in SI imaging leads to more channeling (less backscattering). These regions appear bright in AFM images (reduced sputtering) with shallower depth measurements. Angular view SEM micrographs provide good depth perspective of the differences between partially channeled and unchanneled grains. They support AFM analysis where height differences among the grains were measured.

The perfections in lattice produce high quality diffraction bands. Detection of a grain boundary is easily possible in image quality map because mixture of two diffractions representing two orientations from neighboring grains create a dark region at the grain boundary. The surface roughness effects will appear as local intensity gradients, which could affect the ability of Hough transform to accurately locate the positions of the bands. The quality and accuracy of the EBSD patterns were estimated by automated indexing method developed earlier [20-22]. If the EBSD indexing quality is lower, a darker region would appear in the image due to distortions or surface non-uniformity. Information about the EBSD band quality indexing can also be found in our past work [3, 6]. The overlap of IPF and quality of EBSD band indexing images show if the detection

of a grain with orientation (h k l) (u v w) was done with higher indexing quality. Therefore, from the lack of intensity variations within a single grain surface IPF maps and AFM surface measurements (d), it can be concluded that the sputter rates within a single grain are uniform. EBSD image quality maps (not shown) also support this analysis. The individual grains in Figure 4(b), Figure 5(b) and Figure 6(b) are numbered to identify orientation relationship with Ga-ion beam channeling effects.

Lattice orientation dependence was observed between secondary electron and ion induced emission [1]. The authors noted anti-correlation between the two imaging techniques. The anti-correlation was attributed to the primary ion channeling disregarding surface effects. The secondary emission yield in the ion induced reactions reflects the rate of energy exchange in solid, typically termed as nuclear stopping power. If nuclear stopping power becomes larger than electronic stopping power, nuclear stopping initiates atomic collision cascade which results in secondary ion and secondary electron emission. Calculations using SRIM for 60 keV Ga-ions incident on Ni indicate that for amorphous sample, the electronic energy loss by the ion is 5% and by recoils 12%. The rate of energy loss for incident ions, nuclear and electronic loss depends upon material, secondary electrons, secondary ions, and sputtering effects provide structural information about the target material. The Tables 2-4 show orientation of each numbered grains in Figures 4-6 (b), misorientation angle for each crystals with respect to incoming Ga-ion beam (normal to surface) and measured sputter depths using AFM analysis. The normalized depth percentages considering the maximum observed depth as hundred percent are also shown in the bracket. Excellent correlation is observed for all image sets among AFM, SIM and angular view SEM data.

It is clear from the image sets of Figures 4-6 that the contrast and brightness during SIM directly correspond to the crystal channeling. It is expected that maximum sputter depth is observed for the highest dose case of 4.18×10^{17} ions/cm². By considering measured sputtered depths using AFM in individual grains for doses 4.18×10^{16} ions/cm², 2.09×10^{17} and 4.18×10^{17} ions/cm², data in Figure 7 is normalized. In other words, to understand the extent of grain channeling within selected grains and compare them with orientation and Ga-ion dose dependency, the data for normalized orientation dependent grain channeling is shown in Figure 7 (a-c). Individual crystal orientations for each grain (data point) are also shown schematically. This is a better way to correlate individual grain orientations with related channeling effects irrespective of the starting grain number. It is observed that the highest sputter depth differences between maximum and minimum grain channeling stay high (83% - 95%) irrespective of the Ga-ion dose. The maximum channeling (minimum sputter rate) conditions for the Ga-ion doses of 4.18×10^{16} , 2.09×10^{17} and 4.18×10^{17} ions/cm² were around 7%, 20% and 5%, respectively. Such maximum channeling conditions for any given dose cannot be pinpointed by sampling few grains. Therefore, minimum and maximum sputter depths observed in these cases are more dependent upon ability of local grain crystal alignment with incoming Ga-ion beam direction and does not represent minimum and maximum channeling conditions for the entire sample. In a previous work, Levi-Setti et al; noted that for channeling widths of 5-9 degrees using 60 keV Ga-ion beam on brass samples, channeling results in 20-70% drop in secondary electron signal compared to amorphous material [23]. The author also notes that while lattice orientation dependence and secondary electron and ion emission is generally comparable, several examples are noted where there is anti-correlation between secondary electron and ion emission intensities. These results confirm our observations related to SEM-SIM correlation from tables 2-4.

CONCLUSIONS

The data discussed in this article provides complimentary information to a recent article [24]. Surface modifications through energetic ion solid interactions at low doses is a sub-discipline in itself where many applications related to biological and chemical surface functionalization are already being investigated [25, 26]. Current study attempts to establish a correlation between high resolution imaging, applied Ga-ion dose and orientation dependent Ga-ion beam channeling effects. It was observed that for Ni-alloy, the critical dose ion region is in the vicinity of 10^{15} ions/cm² where a transition from grain surface imaging to sputtering dominated Ga-ion channeling occurred. Above this dose, strong orientation dependent channeling effects were observed. Analysis of selected are normalized grain transparency factor revealed that despite low sampling volume for a random polycrystalline orientations and variation in Ga-ion dose, the range of sputter depth variation during Ga-ion channeling remains above 80% indicating dose independency in the sputter dominated dose region. Because channeling induced contrast effects are stronger in SIM, the imaging contrasts generated due to lattice orientation can be exploited to understand lattice orientation effects. Since the channeling yields are dependent upon local crystal transparency along a particular direction, calculating the difference between two partially channeled crystals during secondary ion induced electron imaging will also be dependent upon changes in secondary electron emission coefficients. Therefore, it is generally believed that axial ion channeling along primary crystal axis is stronger than off-axis crystal channeling directions. Outcomes of such studies could be leveraged to modify surfaces with specific orientations resulting control over orientation dependent surface activity for a variety of disciplines such as catalysis and fuel cell development technologies.

ACKNOWLEDGEMENTS

All experimental portion of this research was performed using electron microscopy resources at the Environmental Molecular Sciences Laboratory (EMSL), a national scientific user facility from DOE's Office of Biological and Environmental Research located at Pacific Northwest National Laboratory (PNNL). PNNL is operated by Battelle for the US DOE. The funding support is partially provided by BER through the grant DE-AC06-76RL01830.

REFERENCES

- [1] R. Levi-Setti, P.H. La Marche, K. Lam, T.H. Shields, Y.L. Wang, Nucl. Instr. and Meth. Phys. Res., 218 (1983) 368-374.
- [2] M.B.H. Breese, E.J. Teo, L. Huang, Nucl. Instr. and Meth. Phys. Res. B, 260 (2007) 288-292.
- [3] L.V. Saraf, Microsc. Microanal., 18 (2012) 371–378.
- [4] L.V. Saraf, J. Micromech. Microeng., 20 (2010) 045031-6.
- [5] L.V. Saraf and D.W. Britt, J. Vac. Sci. and Technol., B, 29 (2011) 051603.
- [6] L.V. Saraf, Praktische Metallographie 48(2011)184-187.
- [7] L.V. Saraf, Nucl. Instr. and Meth. Phys. Res. B 269 (2011) 1540–1547
- [8] A. Medevielle, I. Hugon and O. Dugne, Electron backscattering diffraction (EBSD) application for nuclear materials, in Analysis of In Service Failures and Advances in Microstructural Characterization: Microstructural Science, Vol 26 Materials Park, OH: ASM International, 1999, pp. 265–268.
- [9] P. Vonlanthen and B. Grobety, Ceram. Int., 34 (2008) 1459–1472.
- [10] M. Humbert, N. Gey, C. Patapy, E. Joussein, M. Huger, R. Guinebretiere, T. Chotard and A. Hazotte, Scripta. Mater., 63 (2010) 411–414.
- [11] U. Wendt and G. Nolze, Praktische Metallographie, 44 (2007) 236-238.
- [12] P. K. Rol, J. M. Fluit, F. P. Viehbock and M. de Jong in Proce.4th Int. Conf. on Ionization Phenomena in Gases, N. R. Nilsson, (Ed.) North-Holland Pub. Co., Amsterdam, 1959, p. 257.
- [13] K. Yasuda, M. Nastasi, K.E. Sickafus, C.J. Maggiore, N. Yu, Nucl. Instr. and Meth. Phys. Res. B, 136 (1998) 499-504.
- [14] E.H.D. Vanvoorthuysen, P.J.M. Smulders, R.D. Werkman, J.L. Deboer, S. Vansmaalen, Phy. Rev. B, 45 (1992) 9667-9670.

- [15] H. E. Roosendaal, *Sputtering by Particle Bombardment*, vol. 1, ed. R. Behrisch, Springer, Berlin, 1981 p. 219.
- [16] (a) J. Lindhard, *Mat. Fys. Medd. Dan. Vid. Selsk.*, 14 (1965) 34. (b) J. Lindhard and A.H. Sorensen, *Phy. Rev. A*, 53 (1996) 2443-2456. (c) E.S. Mashkova, V.A. Molchanov, Chapter 1: Interactions of atomic particles with solids in *Medium-Energy Ion Reflection from Solids*, North Holland Physics Publishing Elsevier, 1985. (d) P. Sigmund, Chapter 11, *Channeling and Blocking of Energetic Particles in Crystals in Particle Penetration and Radiation Effects*, Volume 2, *Penetration of Atomic and Molecular Ions*, Springer, 2014, 549-585.
- [17] A.A. Bettiol, I. Rajta, E.J. Teo, J.A. van Kan, F. Watt, *Nucl. Instr. and Meth. Phys. Res. B*, 190 (2002) 154-159.
- [18] J.R. Silk, R.J. Dashwood, R.J. Chater, *Nucl. Instr. and Meth. Phys. Res. B*, 268 (2010) 2064-2068.
- [19] B.W. Kempshall, S.M. Schwarz, B.I. Prenitzer, L.A. Giannuzzi, R.B. Irwin, F.A. Stevie, *J. Vac. Sci. and Technol., B*, 19 (2001) 749-754.
- [20] S. I. Wright, B. L. Adams, *Metal Trans.*, 23 (1992) 759–767.
- [21] S. I. Wright, M.M. Nowell, *Microsc. Microanal.* 12 (2006) 72–84.
- [22] D.P. Field, *Ultramicroscopy*, 67 (1997) 1–9.
- [23] R. Levi-Setti, T.R. Fox, K. Lam, *Nucl. Instr. and Meth. Phys. Res. B*, 205 (1983) 299-309.
- [24] L.A. Giannuzzi and J.R. Michael, *Microscopy and Microanalysis*, 19 (2013) 344-349.
- [25] M.M.M. Bilek, *Appl. Surf. Sci.*, 310 (2014) 3-10.
- [26] L.A. Giannuzzi, B.I. Prenitzer, B.W. Kempshall, Chapter 2: Ion-Solid Interactions, in *Introduction to Focused Ion Beams; Instrumentation, Theory, Techniques and Practice*, by L.A. Giannuzzi (ed.), Springer, 2005, pages 13-52.

FIGURE CAPTIONS

Figure 1: The set of images in three vertical columns show time lapsed SIM images after Ga-ion beam exposure to the sample surface within first few milliseconds when sample surface was exposed to ion beam, when channeling effects are first observed and when channeling effects became clearly distinguishable. Ga-ion beam current for four sets of images in Fig. 1 (a-d) are 30 pA, 50 pA, 100 pA and 300 pA, respectively. Locations marked by arrows from the middle column images show appearance of Ga-ion channeling contrasts near grain boundary regions.

Figure 2: Total ion dose and time as a function of Ga-ion beam current where a transition to SIM imaging with grain channeling is observed.

Figure 3: AFM images represent craters created in Ni-alloy after Ga-ion beam sputtering at (a) 4.18×10^{16} , (b) 2.09×10^{17} and (c) 4.18×10^{17} ions/cm². Minimum and maximum depths for all three dose values were measured at 2 nm, 11 nm and 7 nm, respectively & 23 nm, 66 nm and 146 nm, respectively.

Figure 4: A set of images in (a-e) relate to results for the craters created using dose, 4.18×10^{16} ions/cm². The micrographs are (a) SIM, (b) Inverse pole figure (IPF) map, (c) top view SEM, (d) AFM and (e) angular SEM view.

Figure 5: A set of images in (a-e) relate to results for the craters created using dose, 2.09×10^{17} ions/cm². The micrographs are (a) SIM, (b) Inverse pole figure (IPF) map, (c) top view SEM, (d) AFM and (e) angular SEM view.

Figure 6: A set of images in (a-e) relate to results for the craters created using dose, 4.18×10^{17} ions/cm². The micrographs are (a) SIM, (b) Inverse pole figure (IPF) map, (c) top view SEM, (d) AFM and (e) angular SEM view.

Figure 7: To understand the extent of grain channeling within selected grains and compare them with orientation and Ga-ion dose dependency, the data for normalized orientation dependent grain channeling is shown for doses (a) 4.18×10^{16} ions/cm² (b) 2.09×10^{17} ions/cm² and (c) 4.18×10^{17} ions/cm².

TABLE CAPTIONS

Table 1: Details of experimental parameters used during FIB sputtering experiments.

Table 2: For ion dose of 4.18×10^{16} ions/cm², grain orientations, crystal misorientation angles with respect to direction of Ga-ion beam and measured sputter depths using AFM analysis. Normalized depth percentage is shown in the bracket.

Table 3: For ion dose of 2.09×10^{17} ions/cm², grain orientations, crystal misorientation angles with respect to direction of Ga-ion beam and measured sputter depths using AFM analysis. Normalized depth percentage is shown in the bracket.

Table 4: For ion dose of 4.18×10^{17} ions/cm², grain orientations, crystal misorientation angles with respect to direction of Ga-ion beam and measured sputter depths using AFM analysis. Normalized depth percentage is shown in the bracket.

Figures

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Tables

Table 1

FIB parameters: 30 kV @ 7 nA,

Dwell Time : 1 μ S

Volume per Dose: 0.15 $\mu\text{m}^3/\text{nC}$

Total beam diameter: 102 nm

Saturated current density: 20 nA/nm²

Total sputter rate: 1.05 $\mu\text{m}^3/\text{s}$

Outer pattern diameter: 50 μm

Inner beam diameter: 10 μm

Total Sputtered Area: 1880 μm^2

Max Sputter depth (nm)	Time to complete (s)	No. of Passes	Ion beam dose (ions/cm²)
23	18	25	4.18×10^{16}
65	90	124	2.09×10^{17}
146	180	248	4.18×10^{17}

Table 2

Grain ID	Orientation (hkl)(uvw)	Misorientation angle (deg)	Depth (nm)	SEM- SIM correlation	AFM (color)	AFM- SIM correlation
1	(-23, -22, -10) (6, 1, -16)	148.14	22.93nm (100%)	(dark-light) N	Dominant Sputtering	(dark-light) Y
2	(-5, 1, 22) (-1, -5, 0)	101.91	7.72nm (33.66%)	(light-dark) N	Dominant Channeling	(light-dark)Y
3	(16, 8, 9) (-16, 23, 8)	141.52	1.61nm (7.02%)	(dark-dark) Y	Dominant Channeling	(light-dark)Y
4	(-3, 10, -1) (-3, 2, 29)	116.88	9.09nm (39.64%)	(dark-light) N	Dominant Sputtering	(dark-light) Y
5	(4, 3, 1) (10, -7, -19)	78.87	7.56nm (32.96%)	(dark-dark) Y	Dominant Channeling	(light-dark)Y
6	(-2, -3, 4) (-10, 16, 7)	119.80	12.87nm (56.12%)	(dark-light) N	Dominant Sputtering	(dark-light) Y
7	(-2, 23, 2) (-3, -2, 20)	119.64	3.38nm (14.74%)	(light-dark) N	Dominant Sputtering	(light-dark)Y
8	(10, 22, 5) (1, -5, 20)	124.46	11.51nm (50.19%)	(dark-light) N	Dominant Sputtering	(dark-light) Y
9	(18, 8, 17) (11, -29, 2)	84.43	14.4nm (62.79%)	(dark-light) N	Dominant Sputtering	(dark-light) Y
10	(-9, -3, -10) (-15, 25, 6)	150.03	3.38nm (14.74%)	(light-dark) N	Dominant Channeling	(light-dark)Y
11	(7, -11, 2) (27, 11, -34)	82.85	6.52nm (28.43%)	(light-dark) N	Dominant Channeling	(light-dark)Y

Table 3

Grain ID	Orientation (hkl)(uvw)	Misorientation angle (deg)	Depth (nm)	SEM-SIM correlation	AFM (color)	AFM-SIM correlation
1	(11, 5, 8) (-16, 24, 7)	137.03	11.42nm (17.41%)	(light-dark) N	dominant channeling	(light-dark) Y
2	(-7, 43, -3) (-5, 1, 26)	122.32	31.3nm (47.72%)	(dark-light) N	dominant sputtering	(dark-light) Y
3	(24, 16, -5) (9, -16, -8)	103.60	24.95nm (38.03%)	(light-dark) N	dominant channeling	(light-dark) Y
4	(-11, -4, -5) (-8, 7, 12)	121.63	51.91nm (79.14%)	(light-light) Y	dominant sputtering	(dark-light) Y
5	(11, 4, -6) (2, -4, 1)	134.63	28.97nm (44.16%)	(light-dark) N	dominant channeling	(light-dark) Y
6	(-13, -5, -6) (-13, 11, 19)	122.18	45.79nm (69.81%)	(light-light) Y	dominant sputtering	(dark-light) Y
7	(-6, 23, 8) (-19, 2, -20)	151.64	12.79nm (19.5%)	(light-light) Y	dominant sputtering	(dark-light) Y
8	(-7, -19, -5) (1, 2, -9)	137.87	39.76nm (60.61%)	(light-light) Y	dominant sputtering	(dark-light) y
9	(-3, -1, -5) (-13, 29, 2)	159.76	55.13nm (84.05%)	(light-light) Y	dominant sputtering	(dark-light) Y
10	(4, -2, -29) (-1, 27, -2)	171.62	21.08nm (32.13%)	(light-dark) N	dominant channeling	(light-dark) Y
11	(-9, 26, -1) (-19, -6, 15)	141.90	15.45nm (23.55)	(light-dark) N	dominant channeling	(light-dark) Y
12	(-13, -7, 12) (-7, 13, 0)	124.83	65.59nm (100%)	(dark-light) N	dominant sputtering	(dark-light) Y

Table 4

Grain ID	Orientation (hkl)(uvw)	Misorientation angle (deg)	Depth (nm)	SEM-SIM correlation	AFM (color)	AFM-SIM correlation
1	(-14, 24, -5) (-3, 2, 18)	114.37	130.49nm (89.36%)	(light-light) Y	dominant sputtering	(dark-light) Y
2	(-4, -2, 5) (-9, 8, -4)	147.17	146.02nm (100%)	(light-light) Y	dominant sputtering	(dark-light) Y
3	(-5, -3, -33) (0, 11, -1)	177.60	41.42nm (28.36%)	(dark-dark) Y	dominant channeling	(light-dark) Y
4	(1, -7, 1) (8, 5, 27)	109.67	19.42nm (13.29%)	(dark-dark) Y	dominant channeling	(light-dark) Y
5	(-2, 7, -8) (-2, -4, -3)	165.85	6.8nm (4.65%)	(dark-dark) Y	dominant channeling	(light-dark) Y
6	(5, 1, 6) (2, -4, -1)	70.44	15.93nm (10.9%)	(dark-dark) Y	dominant channeling	(light-dark) Y
7	(-15, -17, -15) (-18, 15, 1)	159.41	83.5nm (57.18%)	(light-light) Y	dominant sputtering	(dark-light) Y
8	(-2, 5, 0) (-20, -8, 7)	152.14	77.61nm (53.15%)	(dark-dark) Y	dominant channeling	(light-dark) Y
9	(2, -13, 1) (8, 3, 23)	110.96	19.75nm (13.52%)	(dark-dark) Y	dominant channeling	(light-dark) Y