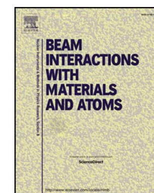
Nuclear Inst. and Methods in Physics Research B 418 (2018) 34–40

Contents lists available at ScienceDirect

Nuclear Inst. and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb



Crystallographic orientation dependence of the sputtering yields of nickel and copper for 4-keV argon ions determined using polycrystalline targets

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A R T I C L E I N F O

Keywords:
Sputtering yield
Crystallographic orientation
Channeling
Polycrystal
Electron backscatter diffraction

A B S T R A C T

A novel approach for revealing the crystallographic orientation dependences of sputtering yields was developed; this approach used 3D surface profiling and the crystallographic orientation imaging of polycrystalline targets. The sputtering yields of Ni and Cu for normally incident 4-keV Ar^+ ions were measured over practically all possible crystallographic orientations. They were found to be low around the directions parallel to the [0 0 1] and [1 0 1] axes and around the directions parallel to the $(1\,1\,\bar{1})$ and (0 1 0) planes. The Onderdelinden model for the crystallographic orientation dependences of sputtering yields was extended such that it could include planar channeling. The crystallographic orientation dependence determined experimentally was in semi-quantitative agreement with the dependences calculated using the extended Onderdelinden model; this demonstrated that the non-channeling probability dominantly controls the sputtering yield.

1. Introduction

Sputtering by energetic ions is a technique that is widely used for film deposition, surface modification, and surface analysis. It is also important as a process of plasma–surface interactions in nuclear fusion reactors.

Sputtering yield, which is a basic physical quantity used to characterize sputtering phenomena, is known to depend on a target's crystallographic orientation, *i.e.*, the angle between the incident direction of the ions and the crystal axes of the target. This crystallographic orientation dependence has been studied experimentally since the 1960's, [1–14]; for example, Onderdelinden et al. [7,11] measured the sputtering yields of monocrystalline targets by rotating them around low-indexed crystalline axes, and Southern et al. [2,9,12] measured the sputtering yields of monocrystalline targets having various crystallographic faces.

In addition, Onderdelinden [7,11] formulated the crystallographic orientation dependence of sputtering yields by assuming that axially channeled particles do not contribute to sputtering and by evaluating the channeling probability using Lindhard's theory [15] (Francken and Onderdelinden later discussed the transition from axial channeling to planar channeling [16]). The calculated dependence was in semi-quantitative agreement with the measured one, which suggested that axial channeling controlled the sputtering yield.

However, it is difficult to cover all possible crystallographic orientations by using such experimental methods as those of Onderdelinden et al. [7,11] and Southern et al. [2,9,12]. As a result, the experimental data for crystallographic orientation dependences are incomplete even for copper, which is the material that has been studied most intensively. It should also be noted that in experiments where monocrystalline targets were rotated [1,3,5–7,10,11,14], not only the crystallographic orientation of the target but also the incident angle of ions relative to the target surface changed with the rotation and thereby affected the sputtering yields. This is unfavorable for quantitative discussions about the effects of crystallographic orientation.

Therefore, we devised a novel experimental approach that could be used to reveal the crystallographic orientation dependence of sputtering yields. It involved the bombardment of a *polycrystalline* target with normally incident ions followed by 3D surface profiling using a white-light interferometer and crystallographic orientation imaging based on electron backscatter diffraction (EBSD); that is, we measured the sputtering depth and crystallographic orientation of individual grains within the polycrystalline target. This approach made it possible to measure the sputtering yields for practically all possible crystallographic orientations over a relatively short period of time. In this paper, we present the crystallographic orientation dependences of the sputtering yields of Ni and Cu for 4-keV Ar^+ ions determined using this novel approach. In addition, we extended the Onderdelinden model for the crystallographic orientation dependences such that it could include planar channeling. By comparing the experimentally determined dependences with those calculated using the extended Onderdelinden model, we discuss the origins of the crystallographic orientation dependences of the sputtering yields.

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https://doi.org/10.1016/j.nimb.2017.12.023
Received 20 September 2017; Received in revised form 16 December 2017; Accepted 19 December 2017
Available online 02 January 2018
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2. Experimental

The target specimens used were polycrystalline sheets of Ni (99 + % purity, Nilaco Corp.) and Cu (99.994% purity, Nilaco Corp.). They were polished with emery papers and 3 μm diamond slurry. They were then annealed in a vacuum at 600 °C for 2 and 4 h, respectively, to randomize the crystallographic orientations of the grains *via* recrystallization and remove the deterioration layer created by the polishing. Monocrystalline Si(1 1 1) wafers were also used as targets to measure flux density distribution of the sputter ion beam. These wafers were etched using a tetramethylammonium hydroxide aqueous solution (Semico Clean, Furuuchi Chemical Corp.) to remove any surface oxides prior to the sputtering. In a single sputtering run, one or two polycrystalline samples and a monocrystalline Si(1 1 1) wafer were loaded together in a vacuum system and sputtered successively without turning off the ion gun.

Sputtering was performed using an ion gun (FIG-5, ULVAC PHI) that was installed in the vacuum system 50 mm from the target; its axis was normal to the target surface. A 4-keV Ar^+ beam from the ion gun was focused to a diameter of 1 mm on the target surface and scanned over an area of 3 mm × 3 mm. Although the incident angle had a small distribution around the surface normal as a result of the focusing and scanning, its effect on the sputtering yield was considered negligible because the sputtering yield was reported to be insensitive to the incident angle in the vicinity of the surface normal [17]. The targets were biased to 27 V to suppress the emission of any secondary electrons. The ion current was ~5 μA, which resulted in an average flux density of $3 \times 10^{18}\,m^{-2}\,s^{-1}$. The sputtered depth was typically several micrometers. The base pressure of the vacuum system was 10^{-5} Pa, and it was increased to 10^{-4} Pa during sputtering mainly because of the argon gas that was fed into the ion gun.

The sputtering crater profiles in the specimens were measured using a white-light interferometer (New View 6200, Zygo Corp.). Although the specimen surfaces after the polishing were slightly curved, they were found to be well-fitted by fourth- or fifth-degree bivariate polynomials. This made it possible to locate the surfaces before sputtering by interpolation of the specimens that had been sputtered. The uncertainties in the sputtering depths resulting from the interpolation were estimated to be a few tens nanometers at most. The overall sputtering yield was directly calculated from the crater volume and the integrated beam current. The obtained sputtering depth distribution was converted into a sputtering yield distribution by using the sputtering depth distribution of the Si(1 1 1) wafer together with the overall sputtering yield. Crystallographic orientation imaging was conducted using an EBSD system (OIM, EDAX, AMETEK, Inc.) that was attached to a focused ion beam system (MI4000L, Hitachi High-Technologies Corp.). The sputtering yield map and crystallographic orientation map were combined such that the properties of the individual grains of the specimens could be obtained.

3. Results and discussion

3.1. Experimentally determined crystallographic orientation dependence of the sputtering yields

Fig. 1 is a pole figure of a Ni polycrystalline specimen. It shows that the specimen consisted of grains with nearly random crystallographic orientations due to the recrystallization that occurred during annealing. Similar results were obtained for the other specimens examined, including Cu.

The overall sputtering yields were 2.91 for Ni and 4.46 for Cu. These were well within the range of published data for polycrystals [17]. The scatter of the published data may have been partly due to the difference in the crystallographic orientation distributions of the samples. In the end of this subsection, we will evaluate the sputtering yield of a polycrystal that has completely random orientations. (Random orientations here mean that the points which represent crystallographic orientations

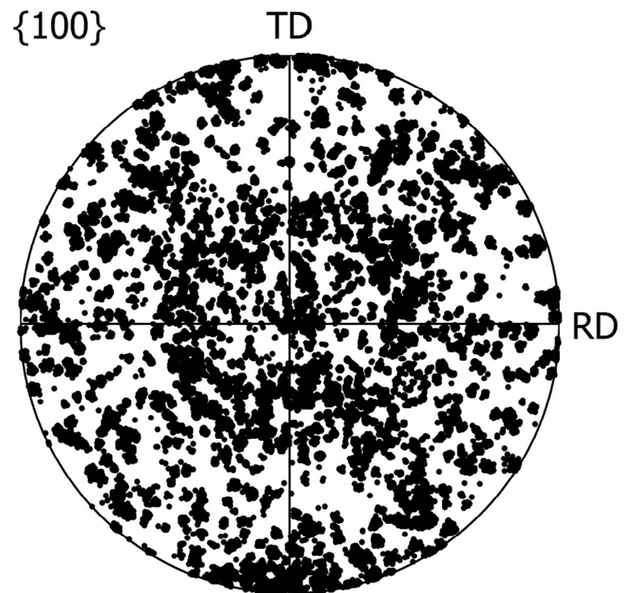

Fig. 1. The pole figure of a Ni polycrystalline specimen. The grains in the specimen have nearly random orientations.

of individual grains are randomly distributed not in the projection plane but on the surface of the unit sphere projected onto the plane.)

Fig. 2(a) is a sputtering depth map for a Ni polycrystalline target, and Fig. 2(b) is a sputtering depth map for a Si(1 1 1) monocrystalline target. The depth distribution of the latter is considered proportional to the flux density distribution of the ion beam. Fig. 2(c) is a sputtering yield map for the Ni polycrystalline target; it was calculated pixel by pixel using the data shown in Fig. 2(a) and (b). It shows that the sputtering yield varied from position to position. Fig. 3(a) is a crystallographic orientation image of the Ni polycrystalline target, and Fig. 3(b) is the corresponding sputtering yield map, which is a part of Fig. 2(c). The boundary shapes found in Fig. 3(a) can also be found in Fig. 3(b), thus indicating that the difference in the sputtering yields was due to the difference in the crystallographic orientation.

Fig. 4(a) shows the obtained sputtering yields of Ni plotted on an inverse pole figure (IPF); Fig. 4(b) shows a contour map of the same data generated by locally weighted smoothing (LOESS) using a third-degree bivariate polynomial together with tri-cube weight function. Fig. 5(a) shows the relative residuals of the LOESS fit; the standard deviation of the relative residuals was 0.080. The sputtering yield of a Ni polycrystal with completely random crystallographic orientations was evaluated to be 3.06 by averaging the yield shown in Fig. 4(b) over all possible orientations. Actually, a weighted average of the values on the IPF was calculated considering the relation between the area element $dXdY$ of the projection plane (IPF) and the area element dA of the unit sphere: $dA = 4(1 + X^2 + Y^2)^{-2}dXdY$, where X and Y are the coordinates in the projection plane.

It should be noted that the sputtering yield was low around the directions parallel to the [0 0 1] and [1 0 1] axes and around the directions parallel to the $(1 1 \bar{1})$ and (0 1 0) planes. The minima in the sputtering yield also appear in the three lines in Fig. 6(a), which lines show the sputtering yields for the directions parallel to the (0 1 0), $(1 \bar{1} 0)$, and $(1 1 \bar{1})$ planes as functions of the angles from the [0 0 1], [0 0 1], and [1 0 1] axes, respectively.

Figs. 4(d), (e), 5(b), and 6(b) show the corresponding results for Cu. The standard deviation of the relative residuals of the LOESS fit was 0.10. The sputtering yield of a Cu polycrystal with completely random crystallographic orientations was found to be 4.70. Although the sputtering yield of Cu was considerably higher than that of Ni, the crystallographic orientation dependence of the sputtering yield was almost the same for the two metals.

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