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Determining the sputter yields of molybdenum in low-index crystal planes via electron backscattered diffraction, focused ion beam and atomic force microscope

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ABSTRACT

Previous literature has used several monocrystalline sputtering targets with various crystalline planes, respectively, to investigate the variations of the sputter yield of materials in different crystalline orientations. This study presents a method to measure the sputtered yields of Mo for the three low-index planes (100), (110), and (111), through using an easily made polycrystalline target. The procedure was firstly to use electron backscattered diffraction to identify the grain positions of the three crystalline planes, and then use a focused ion beam to perform the micro-milling of each identified grain, and finally the sputter yields were calculated from the removed volumes, which were measured by atomic force microscope. Experimental results showed that the sputter yield of the primary orientations for Mo varied as $Y_{(110)} > Y_{(100)} > Y_{(111)}$, coincidental with the ranking of their planar atomic packing densities. The concept of transparency of ion in the crystalline substance was applied to elucidate these results. In addition, the result of (110) orientation exhibiting higher sputter yield is helpful for us to develop a Mo target with a higher deposition rate for use in industry. By changing the deformation process from straight rolling to cross rolling, the (110) texture intensity of the Mo target was significantly improved, and thus enhanced the deposition rate.

1. Introduction

Sputter deposition is a physical process that uses energetic ions to bombard a solid target material, thereby causing the ejection of surface atoms from the target, and further the condensation of the ejected atoms on a substrate as a thin film. Since sputter deposition has many advantages, such as uniform large-area coating, good adhesion between films and substrates, and easy control of film thickness, the process nowadays is widely used in semiconductors, flat panel displays, magnetic recording media, wear-resistance, decorative coating industries, etc. [1,2]. For these coating industries, the sputter yield (Y), which is defined as the average number of atoms removed from the sputtering target per incident ion, is an important indicator because it directly affects the deposition rate and the speed of production line. In general, the sputter yield depends on the energy of the ion, the ion incident angle, the masses of the ion and the target atom, and the surface binding energy of the target atoms [3]. In addition, for different polycrystalline targets but the same material, the sputter yields are also not quite the same due to differences in grain size or grain orientation, which practically affect the coating rate and the thickness uniformity of the thin film in coating industries [4,5].

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Since the variation in the sputter yield is sensitive to change in both grain size and grain orientation, to purely understand specifically the effect of crystal orientation on the sputter yield $Y_{(uvw)}$ previous literature has mainly prepared monocrystalline targets with various low-index crystallographic planes for sputtering study. For instance, Magnuson and Carlston [6] explored the sputtering yields of Cu and Ag single crystals for the three main low-index planes in 1963, and found that (111) plane exhibits the highest sputter yield, followed by (100) plane, with (110) plane showing the lowest yield. Moreover, Robinson and Southern [7] reported in 1967 the same sputtering results for Au and Al single crystals, namely, $Y_{(111)} > Y_{(100)} > Y_{(110)}$ for face centered cubic (fcc) metals.

Unlike previous studies to determine the sputter yields of different crystal planes by several single crystal targets, this work presents a method that can measure the sputter yields of different crystal planes merely on a polycrystalline material. The method is especially suitable for refractory body centered cubic (bcc) metals, which are more difficult to prepare as single crystals. In this work, electron backscattered diffraction (EBSD), focus ion beam (FIB), and atomic force microscope (AFM) techniques were combined to measure and compare the sputter yields of the three main low-index planes of bcc Mo by using a common polycrystalline Mo target. The results are useful for metallurgical engineers to know how to control the crystallographic texture of the polycrystalline Mo target, and thus improve the target's deposition rate.

2. Experimental

A schematic procedure for measuring the sputter yields of low-index planes of Mo by using EBSD, FIB, and AFM is shown in Fig. 1. A field-emission scanning electron microscope (FE-SEM, JSM-7401F, JEOL) equipped with an EBSD system (OIM™ 5.22, EDAX) was firstly employed to identify the crystal orientation for individual grains of a polycrystalline Mo specimen, so that the positions of grains with exactly (100), (110), and (111) planes could be located individually. Next, an FIB (Nova 200 NanoLab, FEI) was used to perform the micro-milling to form a pattern of square shaped holes on these identified low-index grains, using identical ion milling conditions. After FIB patterning, an AFM (SPA 300HV, SII NanoTechnology) was used to accurately measure the depth of erosion for each grain. Finally, the sputter yields of the three low-index planes could be estimated based on the measured depths plus other sputtering parameters.

The polycrystalline Mo sample used in this study was fabricated through the process of powder metallurgy and thermo-mechanical treatment. Mo powders were compacted into a rectangular bar by cold isostatic pressing, and sintered in hydrogen to obtain a Mo compact with a density of 97%. The compact was then hot rolled and annealed to obtain a 100% dense Mo plate. The Mo plate was cut, ground, and polished to prepare it as a micro-sputtering specimen with a dimension of 10 × 10 × 3 mm. For EBSD analysis, the specimen was inclined at 70° to a scanned electron beam with an accelerating voltage of 20 kV to collect the Kikuchi diffraction patterns of individual grains for orientation identification. For FIB micro-sputtering, a gallium ion (Ga^+) source operating at energy of either 15 keV or 25 keV, and an ion beam current of 1 nA, were selected to mill the identified low-index grains, respectively. The lateral milling area was 10 × 10 μm and the total milling time for each grain was 10 min. For depth measurement by AFM, a commercial Si-based probe (PPP-NCH, NANOSENSORS™, 42 Nm^{-1}, 330 kHz) operating in tapping mode was employed to investigate the surface morphologies of the milled grains.

To simulate the sputtering of Ga ions with different incident energy in Mo target, the Monte Carlo simulation of the software of transport of ions in matter (TRIM) [8] was also used to estimate the sputter yield and compare with the experimental results in this study. The simulating conditions of TRIM were listed in Table 1.

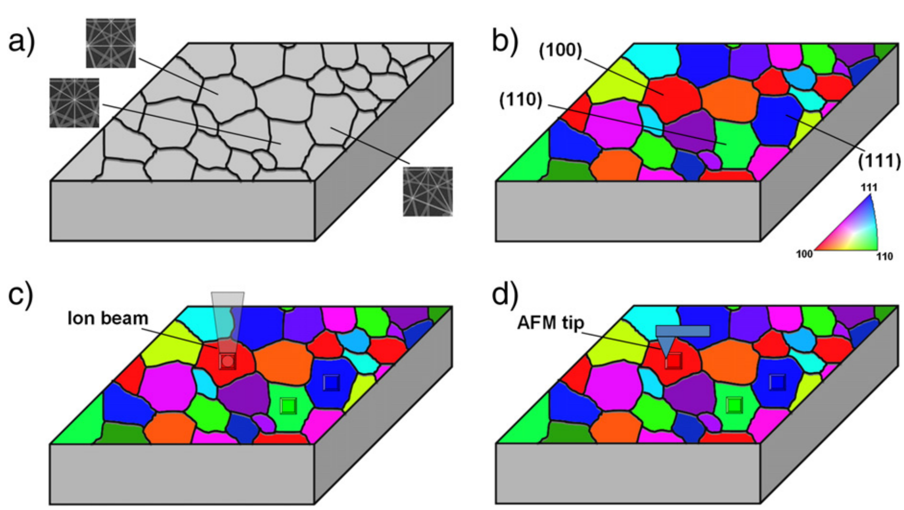

Fig. 1 – Schematics of the procedure for determining the sputter yields of low-index crystalline orientations of Mo in this study: (a) a polycrystalline Mo sample with different grain orientations, (b) orientation identification by EBSD, (c) micro-sputtering by FIB, (d) measurement of milled volume by AFM.