Ion sputtering rate of nanostructured FCC, BCC and HCP metals processed by severe plastic deformation

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Abstract. The effect of nanostructuring on the ion sputtering rate of metals and the glow discharge current is studied. Nanostructured samples were processed by severe plastic deformation using high pressure torsion. For comparative measurements, coarse-grained samples were obtained by annealing of nanostructured samples. The sputtering rates of nanostructured and coarse-grained nickel, iron and zirconium were measured after ion sputtering in glow gas-discharge. It has been found that nanostructuring of metals leads to an increase in the ion sputtering rate and the glow discharge current. Possible causes of changes in the ion sputtering rate due to nanostructuring are analyzed.

1. Introduction

Functional elements of ion sources and similar devices, such as walls, cold cathodes etc. suffer from ion sputtering, which results in degradation of these elements with time. Therefore, the elements are fabricated of metals with a low value of the ion sputtering yield. From another point of view, ion sputtering can also play a positive role, for instance, in the case of sputtering targets, which are widely used in nano- and microelectronics. Target materials must meet certain requirements, such as high purity and a lack of pores, homogeneity of the grain size and crystallographic texture.

Nanostructured metals, which are polycrystals with an average grain size of the order of 100 nm, potentially can be used for a fabrication of elements of ion sources or sputtering targets. One of the attractive methods for obtaining nanostructured metals is deformation nanostructuring, often referred to as severe plastic deformation [1]. To date, various physical properties of nanostructured metals have been studied. It has been found that these materials have significantly modified values of magnetic, thermal, mechanical, electrical, emission properties as compared to their coarse-grained counterparts [1-5]. However, there are some fields that are poorly investigated, for example, properties associated with ion sputtering of nanostructured metals. In this regard, it is important to study the effect of nanostructuring on the rate of ion sputtering of metals.

2. Materials and experimental procedure

Pure nickel, iron and zirconium were chosen as the FCC, BCC and HCP metals for the investigation. To obtain a nanosized microstructure, the samples were processed by severe plastic deformation using high pressure torsion [6,7] in Bridgman anvils under pressure P = 6.5 GPa (10 anvil rotations at a rotation rate of 2 revolutions per minute). For comparative measurements, coarse-grained samples...
were obtained by annealing of the nanostructured samples at 873-1073 K in vacuum for 1 hour. All samples had the form of discs with a diameter of 10 mm and a thickness of 0.5 mm. The microstructure of the samples was investigated by a Mira 3 LHM scanning electron microscope (Tescan, Czech Republic).

Ion sputtering of the samples was carried out in a glow discharge plasma in a gas discharge setup. The gas discharge setup (similar to the one described in [8]) comprises a glass bulb with an anode and six sputtering targets placed inside. The distance between the anode and the targets was equal to d = 15 cm. The nanostructured and coarse-grained samples of nickel, iron and zirconium (six samples in total) were used as the sputtering targets. The simultaneous use of six targets provides the same conditions of sputtering for different samples. Before sputtering, all samples were subjected to mechanical grinding to obtain flat surfaces. The samples were attached to T-shaped metal holders. To measure the gas discharge current, the ammeters were connected to the metal holders. The gas discharge bulb was placed in a vacuum chamber. The experiment was performed at a vacuum level in a chamber of about 10^{-5} Torr. To achieve the glow discharge conditions, the bulb was filled with Ar at a pressure of P \sim 0.1-1 Torr. The discharge voltage was equal to U = 800 V. The discharge current varied for different samples. Ion sputtering was performed for 2 hours. The ion sputtering rate was determined by evaluating the difference in weight of the samples before and after ion sputtering on the precision analytical balance of MV 210-A (Sartogosm, Russia).

3. Results and discussion

Electron backscatter diffraction (EBSD) was used to study the microstructure of nanostructured nickel and iron. Corresponding EBSD maps are presented in figure 1. Analysis of these maps shows that the average grain size of nanostructured nickel and iron after high pressure torsion is almost the same and equal to 180 nm. Analysis of the spectrum of grain boundary misorientations shows that the majority of grain boundaries (up to 80%) are high angle. The microstructure of nanostructured zirconium is studied using the back-scattered electron (BSE) mode, which showed that the grain size was equal to 100-300 nm.

![Figure 1. EBSD maps of nanostructured Ni (a) and Fe (b).](image)

Figure 2 shows the BSE images of the microstructure of coarse-grained nickel, iron and zirconium, i.e. after annealing. It was observed that the grain size of coarse-grained samples is larger than 10 µm.

The values of the average glow discharge current for all investigated samples for U =800 V are collected in figure 3a. This figure shows that the glow discharge current J of nanostructured samples is higher than that of coarse-grained samples (J_{NS-Ni}>J_{CG-Ni}, J_{NS-Zr}>J_{CG-Zr}, J_{NS-Fe}>J_{CG-Fe}) and the nickel sample has the highest value (J_{Ni}>J_{Zr}>J_{Fe}). The glow discharge current depends on the ion-induced electron emission yield \gamma of metal as J \sim \gamma [9].

The ion sputtering rate V can be determined from the measurements of the sample mass before \(m_1\) and after \(m_2\) ion sputtering, as
where $x$ is the thickness of the sputtered layer, $t$ is the total sputtering time, $\Delta m$ is the mass change of the sample $\Delta m=m_1-m_2$, $\rho$ is the density of the metal, $S$ is the sample area. Ion sputtering rates for nanostructured and coarse-grained nickel, iron and zirconium for $J=1.0$ mA/cm$^2$ are presented in figure 3b. These data show that the ion sputtering rate of nanostructured samples is again higher than that of coarse-grained samples: $V_{\text{NS-Ni}}>V_{\text{CG-Ni}}$, $V_{\text{NS-Zr}}>V_{\text{CG-Zr}}$, $V_{\text{NS-Fe}}>V_{\text{CG-Fe}}$ and $V_{\text{CG-Ni}}>V_{\text{CG-Zr}}>V_{\text{CG-Fe}}$.

![Figure 2. BSE images of coarse-grained nickel (a), iron (b) and zirconium (c).](image)

![Figure 3. The average glow discharge current density (a) and the calculated ion sputtering rates at $J=1.0$ mA/cm$^2$ (b) for the nanostructured (NS) and coarse-grained (CG) FCC, BCC and HCP metals for discharge voltage $U=800$ V, $P\sim0.1-1$ Torr, $d=15$ cm.](image)

It is known that ion sputtering of a metal depends on its microstructure. For instance, the smaller the grain size, the higher the ion sputtering rate of metals [10-12]. The increase in the ion sputtering rate due to nanostructuring can be explained as follows. The ion sputtering yield $Y$ depends on the binding energy of the atom $E$ on the surface as $Y \sim 1/E$. The presence of defects reduces the binding energy. Therefore, the ion sputtering yield $Y$ of the grain boundary is higher than that of the grain [10]. Consequently, one can assume that grain boundaries are sputtered faster than grains. Nanostructured metals have a larger volume fraction of grain boundaries, which may explain the higher values of their ion sputtering rates as compared to coarse-grained metals. Thus, the increase in the ion sputtering rate due to nanostructuring is a result of the increase in the volume fraction of grain boundaries.
4. Summary
The effect of deformation nanostructuring on the rate of ion sputtering of nickel, iron and zirconium in a glow gas-discharge has been studied. It has been shown that the ion sputtering rate and the glow discharge current of nanostructured samples are higher than those of coarse-grained samples for all investigated metals having FCC, HCP and BCC lattices. The enhanced values of both quantities are the result of a decrease in the grain size and, correspondingly, an increase in the specific grain boundary area in the materials. Further studies are required for better elucidation of the effect of nanostructuring on the ion sputtering process.

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