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Influence of the irradiation doze and temperature on the flux of ejecting atoms during relief formation on Mg surface by Ar^+ sputtering

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The time dependences of the evaporation and sputtering rate of magnesium under irradiation by 20 keV argon ions have been determined at the temperatures sufficient for considerable sublimation of the material from the target surface. It has been shown that ion beam action can activate the evaporation process continuing even after the irradiation is over. During continuous ion bombardment after the activation, the linear increase of the sputtering rate is observed, perhaps connected with the formation of an irradiation temperature dependent relief on the target surface.

Определены временные зависимости выхода распыления и испарения магния при облучении его ионами аргона с энергией 20 кэВ при температурах, достаточных для сильной сублимации материала с поверхности образца. Показано, что воздействие ионного пучка может приводить к активации процесса испарения, которое не прекращается после окончания облучения. При постоянном воздействии ионного пучка после активации наблюдается линейное возрастание скорости удаления атомов с поверхности, что, возможно, связано с формированием на ней рельефа, зависимого от температуры облучения.

1. Introduction

The microrelief formation on the surface using ion irradiation is a perspective area in the investigation of ion-surface interactions. Possible mechanisms of the surface relief have been considered [1–3], but in most works, the material sputtering is assumed to occur according to cascade mechanism. The development of nanotechnologiescalls interest to new kinds of surface structures, thus stimulating the search for and studying of new mechanisms resulting in formation thereof. For example, we observed a high rate and efficiency of relief formation during irradiation of materials with low sublimation energy at elevated temperatures [4], that is, at dominating non-cascade sputtering mechanisms. The aim of the present work is to investigate the sputtering features of Mg exposed to ion irradiation at elevated temperature, which, in similarity to [5], may result in thermal sputtering domination, as well as the main features of the relief formed on its surface under such conditions.

2. Experimental

The samples of polycrystalline magnesium were used during the experiments due to its low sublimation energy that, in our opinion, provides appropriate conditions for thermal sputtering to bring a considerable contribution into the overall sputtering rate. The mechanically polished and finished samples were placed into vacuum chamber with residual pressure about

$5 \cdot 10^{-7}$ Torr. The surface was irradiated by 20 keV Ar^+ ions, the current density was 10–200 μ/cm^2. The quartz resonator method was used to measure the sputtering yield and the evaporation rate. The AT cut quartz plates with 4000–5000 kHz resonance frequencies were used as sensors. The frequency shift was recorded using an electronic frequency counter connected to the computer, providing the registration rate of about 50 measurements per minute. Using the numeric differentiation of the measured dependences, the time dependences of $\Delta f/\Delta t$ value were found, that value being in proportion to the material deposition rate on the crystal sensor which is, in turn, proportional to the ejection rate V of the atoms leaving the target surface, providing that the relative orientation of the sensor and material ejection source remains constant [6]; at low temperatures, when material is ejected only by sputtering, this value is in proportion to the sputtering yield Y. The temperature was controlled by copper-constantan thermocouple and was stabilized by the regulation program realizing the PID regulation law.

3. Results and discussion

To determine the temperature at which thermal sputtering would contribute considerably to surface relief formation, the temperature dependences of the ejection and sputtering rates were measured.

The normalized dependences $V(T)$ and $Y(T)$ are shown in Fig. 1. Near the temperatures of about 400–425°C, a considerably increased amount of material leaving surface is observed. This is true both for sputtering as well as for the total ejection rate. It is to note that the measurement procedure of the $Y(T)$ dependence included periodical switching-on/off the flux of incident ions and the sputtering rates were determined with subtraction of the evaporated particle fluxes. The amount of the evaporated material at specified temperature was determined from the time dependences of the frequency $f(t)$ recorded prior to and after irradiation. So as it results from Fig. 1, not only evaporation rate, but also the sputtering yield increases at the temperatures about 400°C, thus indicating a sufficient contribution from the thermal sputtering mechanism.

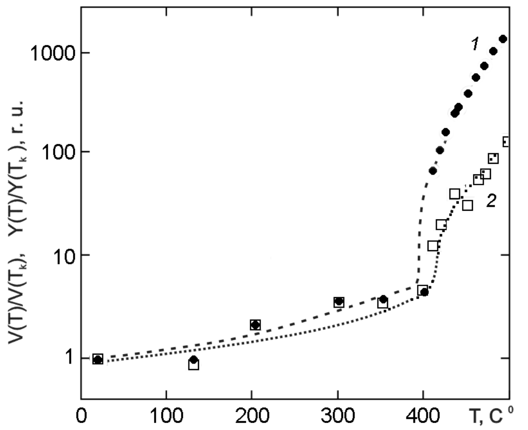

Fig. 1. Temperature dependences of the material ejection rate from the magnesium surface during ion irradiation: *1*, total evaporation and sputtering rate; *2*, the sputtering rate only. T_k = 20°C.

The time dependences of the sputtering rate at temperatures above 430°C differ considerably from those measured at room temperatures, but dependences measured below 400°C are qualitatively similar to the mentioned ones. In Fig. 2, the time dependences are shown recorded at 200°C (a) and 500°C. At low temperatures, a the sensor

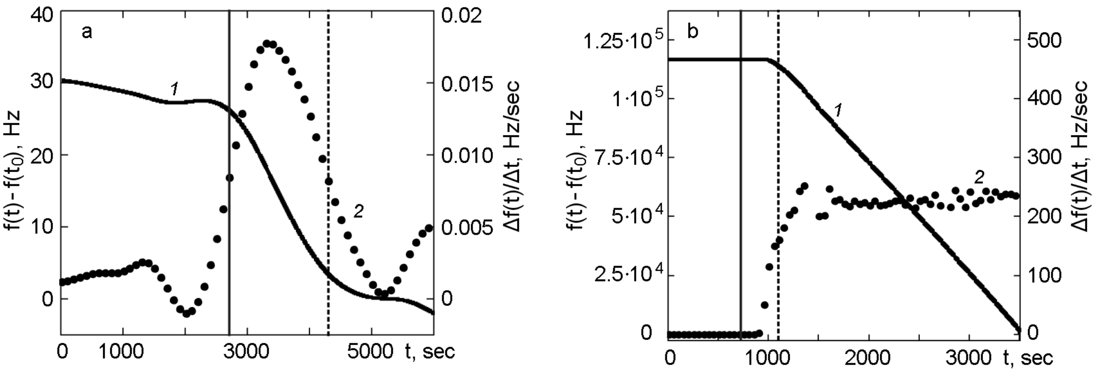

Fig. 2. Time dependences of the crystal sensor frequency shift (*1*) and its time derivative (*2*) during ion irradiation of magnesium at 200°C (a) and 500°C (b). Solid vertical lines show the time moments of the ion beam opening; dotted ones, those of the shutting.

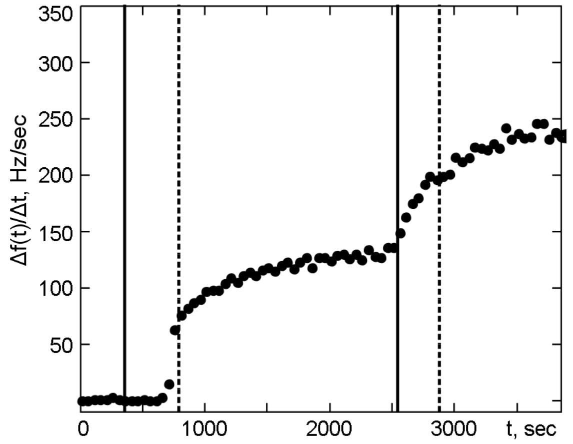

Fig. 3. Time dependence of the sensor frequency shift derivative during repetitive ion bombardment after activation of the intensive evaporation. Solid vertical lines show the time moments of the ion beam opening; dotted ones, those of the shutting.

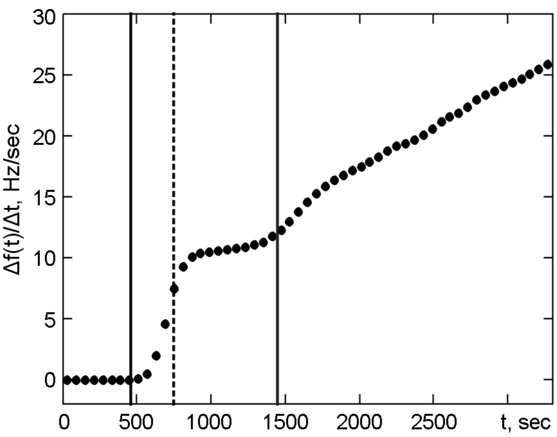

Fig. 4. Time dependence of the sensor frequency shift derivative during continuous irradiation after activation. Solid vertical lines show the time moments of the ion beam opening; dotted ones, those of the shutting.

frequency considerable shift appears only during the irradiation, when the material is sputtered with the ion beam. It is to note that during the sample heating in absence of irradiation, there is no increase in the ejection rate before it was exposed to irradiation. The increase in ejection rate is observed some later after the irradiation onset, as it is shown in Fig. 2b. That increase does not stop even after the ion beam is switched off. The evaporation rate in this case keeps growing for some time, attains saturation, and keeps constant for a long time under absence of any excitations.

Under repeated irradiation, the sputtering rate increases additionally and does not return to the prior value (Fig. 3). Under continuous irradiation, the sputtering rate increases monotonously almost linearly (Fig. 4). Thus, we can say that at elevated temperatures, ion irradiation activates the process of evaporation from target surface. The evaporation in this case occurs only from the pre-irradiated region. The recess formed due to bombardment has sharp edges matching the beam profile.

The mechanism of the observed activation is not clear. Absence of the evaporation at elevated temperatures before irradiation is neither the result of mechanical harden-

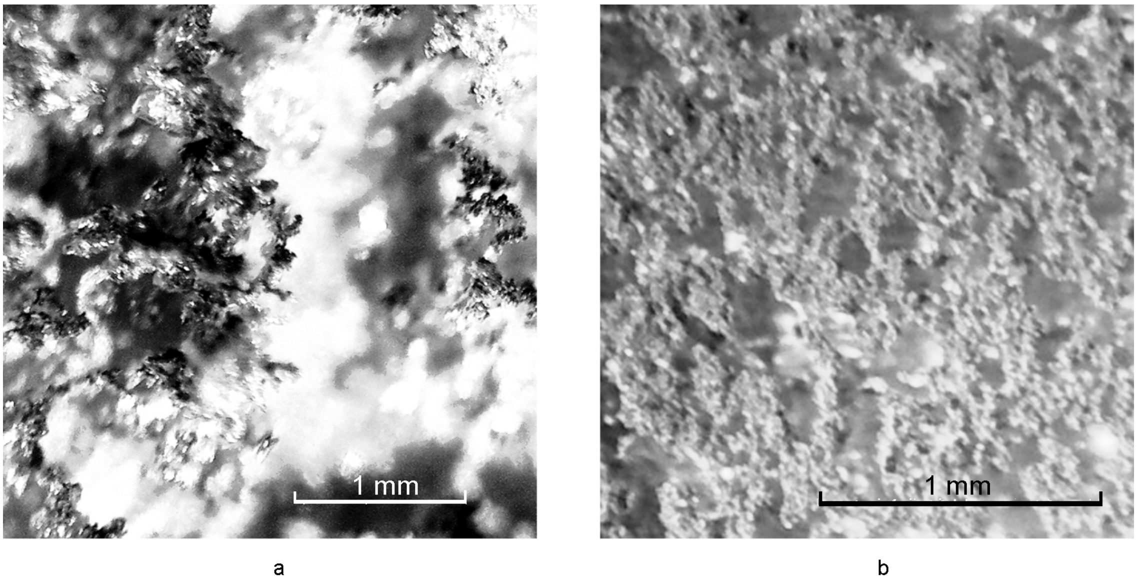

Fig. 5. Relief formed on the sample surface at about 500°C (a) and at 430°C (b).

ing which might occur during the polishing, nor the result of oxide film presence covering the material surface. To confirm that facts, the sample cooled to room temperature after measuring the time dependence at 430°C was exposed to repeated heating; there was no evaporation prior to irradiation, and thereafter, the above-described ejection activation occurred. Perhaps the oxide film was partly restored during cooling in the residual gas atmosphere. Also it had been clarified that this activation was not caused by the macroscopic overheating by the ion beam. To that end, we varied the ion beam current keeping the constant beam power density (by changing its incidence energy). The average quantity of sputtered material had not virtually shown any dependence on the ion beam current density while changing it by a factor of ten.

Perhaps the increase in the evaporation rate during continuous irradiation results from the activated area increase caused by relief formation on the sample surface. In the region of beam impact, a recess is formed filled with barnacles of metallic magnesium. The barnacle size and morphology differ depending on the irradiation temperature, Fig. 5.

4. Conclusions

Thus, the activation of the material evaporation from the target surface is possible during the ion irradiation of magnesium at elevated temperatures. The sputtering process after that activation shows a number of characteristic features: the amount of ejected material in such regime is much larger than during the usual cascade sputtering, the sufficient amount of material leaves the surface in the absence of irradiation, and during continuous irradiation, the ejection rate increases linearly with time.

References

1. R.M.Bradley, J.M.E.Harper, *J.Vac.Sci.Technol.A*, **6**, 2390 (1988).
2. V.I.Emelyanov, *Izv.RAN, Ser.Fiz.*, **70**, 779 (2006).
3. A.A.Gubarev, *Izv.RAN, Ser.Fiz.*, **72**, 632 (2008).
4. I.A.Melnichuk, A.A.Bogunets, I.N.Starshinov, *Izv.RAN, Ser.Fiz.*, **70**, 825 (2006).
5. I.A.Melnichuk, A.A.Bogunets, I.N.Starshinov, ISA 2005. Abstr. of XVII All-Union Conf., Moscow (2005), v.1, p.140.
6. V.V.Milov, Piezoresonance Sensors, Moscow State University, Moscow (1989) [in Russian].

Вплив дози опромінення та температури на потік атомів, що видаляються при формуванні рельєфу на поверхні Mg розпиленням Ar^+

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Визначено часові залежності виходу розпилення та випаровування магнію при його опроміненні іонами аргону з енергією 20 кеВ при температурах, достатніх для інтенсивної сублімації матеріалу з поверхні зразка. Показано, що вплив іонного пучка може викликати активацію процесу випаровування, яке не припиняється після закінчення опромінення. При постійному впливі іонного пучка після активації спостерігається лінійне збільшення швидкості видалення атомів з поверхні, що, можливо, зв'язано з формуванням на ній рельєфу, залежного від температури опромінення.