FABRICATION OF FINE-DISPERSED COATINGS AT DEPOSITION WITH SIMULTANEOUS SPUTTERING

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New low temperature method of homogeneous Mo nanocrystallite coating formation was developed. The coating was formed at magnetron Mo deposition on polished Mo polycrystalline substrate with simultaneous ion sputtering. Deposition and sputtering were performed in combined discharge of magnetron – hole cathode [1]. X-ray difractometry and SEM were used for coating structure investigation. A theoretical model was developed for coating formation by proposed method. At low excess of deposition rate over sputtering rate uncontrolled fast growth of some Mo crystallites is effectively suppressed, being fine dispersed coating is formed. Such coatings can be used as a radiation stable reflecting coatings for high temperature plasma diagnostic mirrors [2, 3]. The proposed method can be used also for other materials modification, semiconductors included.

For ion sputtering cylindrical hole cathode was used made from stainless steel with length 65 mm. Substrate was placed on stainless holder at bottom of cylindrical hole cathode. All elements of hole cathode were initially coated with Mo to reach chemical homogeneity of received coating. Magnetron with isolated anode was placed at the open part of hole cathode. Polished Mo mirrors 3 mm thick with diameter 25 mm were used as substrates. Magnetron cathode with diameter 25 mm was made also from Mo. Two independent power sources were used for magnetron and hole cathode with maximal output 750 V. Magnetron anode was used also as anode for hole cathode. Experimental scheme is shown in figure 1.

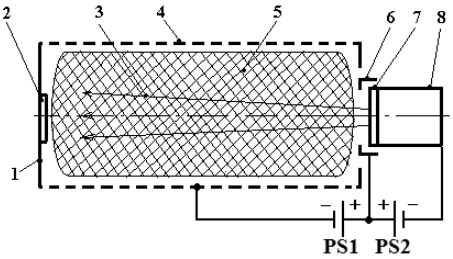

Figure 1. Experimental scheme: substrate holder (1), substrate (2), flow of sputtered in magnetron material (3), cylindrical hole cathode (4), region of hole cathode discharge (5), magnetron anode (6), magnetron cathode (7), magnetron body (8), power source of hole cathode (PS1), power source of magnetron (PS2)

Argon was used as working gas. Coating deposition was carried without addition heating at one gas pressure in all experiments. Two different deposition regimes were studied. In first case deposition lasts T_1 = 130 min, average magnetron discharge current was I_{1m} = 165 mA, average magnetron voltage was U_1 = 303 V, average current of hole cathode was I_{1hc} = 60 mA, average voltage of hole cathode was U_{1hc} = 417 V, coating thickness was h_1 = 1 μm and deposition rate 0.1 nm/s. In the second case T_2 = 600 min, I_{2m} = 257 mA, U_2 = 293 V, I_{2hc} = 80 mA, U_{2hc} = 289 V, coating thickness was h_2 = 1.2 μm and deposition rate 0.03 nm/s. In second experiment decrease of voltage in hole cathode was caused by charge particles exchange with magnetron. Surface reliefs on both samples are shown in the figures 2 and 3. Substrate surface relief after sputtering of top surface defects layer is shown in the figure 4.

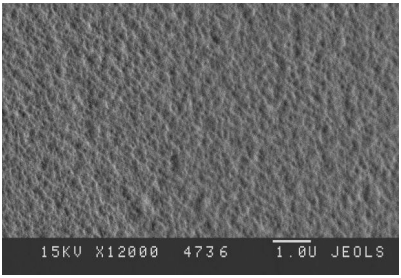

Figure 2. Deposition rate R_1 = 0.1 nm/min (marker 1 μm)

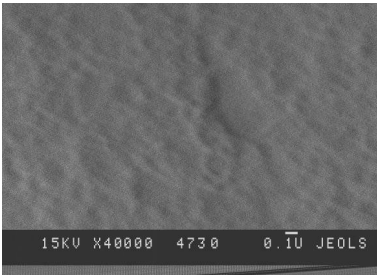

Figure 3. Deposition rate R_2 = 0.03 nm/min (marker 0.1 μm)

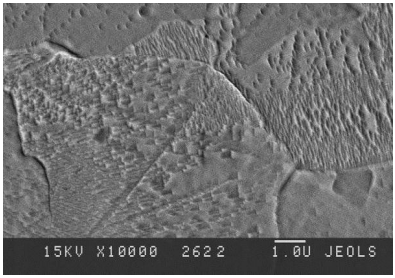

Figure 4. Grains on substrate surface after sputtering of top surface defects layer (marker 1 μm)

Typical grain size on substrate surface is 10 μm. Surfaces of coatings in both cases are smooth and homogeneous. Earlier in [1] surface smoothing was observed at deposition with simultaneous sputtering being polishing defects and scratches disappear and don't seen even after remove of all top surface layer.

X-ray diffractometry shows that coatings consist of BCC crystallites with direction [110] oriented normal to the surface. Crystallites transversal sizes are 6-7 nm for first case and 8-9 nm for second case.

A model of structure formation at deposition with simultaneous sputtering was developed. The model assumes that crystallites size is determined by size of nuclei, on which the crystallites grow. Size of atom cluster – nuclei is limited by cluster destruction under ion bombardment. At initial stage concentration of free diffusing adatoms on the surface is determined by their joining into clusters and equals $C = (q/(\pi \cdot D))^{1/2}$ ($q = q_0 - j \cdot Y$, q_0 is the atom deposition rate, $j \cdot Y$ is the sputtering rate, j is the ion current density, Y is the sputtering yield, D is the diffusion coefficient, which can be not only thermal activated, but also radiation activated). In this stage maximal atoms number in cluster is

$$n_{max} = (D \cdot q/\pi) \cdot a^{-4} \cdot (j \cdot Y_S)^{-2},$$

Y_S is the atoms number separated from a cluster when an ion hits the edge of cluster with width equal an interatomic space a ($d = a \cdot n^{1/2}$ is the cluster diameter). But at high cluster concentration N distance between adatoms becomes higher that distance between clusters ($N^{1/2} - a \cdot n_{max}^{1/2}$). Then new clusters formation deceases and all new atoms appearing on the substrate join to clusters. Thus the final cluster size is

$$d = N^{1/2} + a \cdot n_{max}^{1/2} = (\pi \cdot D/q)^{1/4} + (D \cdot q)^{1/2} \cdot a^{-1} \cdot (j \cdot Y_S)^{-1}.$$

Cluster size is minimal at $q_m = (j \cdot a/2)^{4/3} \cdot D^{-1/3}$. Calculated crystallites sizes are 5 nm for first case and 6.7 nm for second case.

Experiments and model show that coatings fabricated at deposition with simultaneous sputtering have nano size, mono dimension structure with mono oriented columns. Preliminary surface cleaning (by sputtering) and smooth transition to deposition regime result in good adhesion.

1. A.V. Rogov, M.Yu. Nagel, Yu.V. Martynenko. Problems of Atomic Science and Technology. Ser. Thermonuclear Fusion, 2013, V. 36, № 2, p. 19-24.
2. A.V. Rogov, K.Yu. Vukolov. Problems of Atomic Science and Technology. Ser. Thermonuclear Fusion, 2005, № 1, p. 9 – 25.
3. A.V. Rogov, K.Yu. Vukolov, A.V. Gorshkov, V.M. Gureev. Problems of Atomic Science and Technology. Ser. Thermonuclear Fusion, 2005, № 2, p. 39 – 55.