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High-rate magnetron sputtering with hot target

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Chromium films were deposited by cooled and hot target magnetron sputtering techniques with unbalanced magnetic field configuration at equal target power density. The dependence of deposition rates of Cr films by the hot target magnetron sputtering on the power density was a non-linear at 27.5–31.5 W/cm^2. The optical emission studies indicated the enhancement of deposition rates due to the evaporation of the hot Cr target. The chromium film structure was strongly depended on the factor of «hot target» and the target power density. Cr films were structured to (110) direction in the case of the hot target sputtering, for cooled target configuration – the preferred orientation was changed from (110) to (200) with the power density. Cr^+ to Cr ratio and heat flow from the cathode to the substrate influenced on the coating hardness and adhesion. The Cr films with 9.84–12.79 GPa and the non-cracking behavior (up to 15 N) were obtained.

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1. Introduction

Magnetron sputtering systems are widely used for thin film deposition in a medicine, semiconductor devices, space materials, machinery and tools [1–3]. Today, there are some new tendencies in the development of magnetron technologies. The high power impulse magnetron sputtering techniques (HiPIMS) is an enhanced physical vapor deposition device with high pulse power (up to 1…10 kW/cm^2) and increased ionization degree of the erosion material [4,5]. HiPIMS techniques are used to obtain new materials with unique or advanced properties (an adhesion [6], a film density [7], etc.). However, the generation of HiPIMS discharge is an inherent to effects of a return, a yield, ion species and a film thickness [5]. It adversely impacts on deposition rates, which in 2 and more times lower than conventional direct current (DC) magnetron sputtering at equal power. So, the HiPIMS technology is extremely slow implemented. For technologists, static and normalized static deposition rates are key parameters, which determine the price of the film deposition and a technology marketability. In this case, the coating technologies and techniques with enhanced deposition rates are extremely required.

In the magnetron sputtering, deposition rates are limited by the ion current on the cathode due to the existence only a sputtering mechanism of the target erosion. Thus, the addition of the target evaporation as the second mechanism of the cathode erosion doesn't have alternatives to the increase of the deposition rates. It can be realized in liquid phase or hot target magnetrons. We demonstrated the technology of liquid target sputtering for aluminum and copper depositions in a uncooled melting pot [8,9]. Although these magnetron techniques have the enhanced deposition rates (up to 100 times), significant limitations such as the stability and reproducibility of deposition process, the necessity of melting pots are existed. At the same time, the hot target sputtering system is an intensive investigated for the film deposition of titanium and its compounds [10–15]. There are three potential mechanisms of enhanced deposition rates: a particle-induced erosion [16], a sputtering yield increase [17] and the target evaporation (or sublimation). The input of the each mechanism isn't full understood and future investigations are required. Besides, technological possibilities of high-rate deposition systems and film properties deposited by such techniques are also important.

The main disadvantages of hot target magnetron sputtering are an irregularity of target geometry and a phase transition in target material [10,15], a target poisoning at high pressures [12–14] and a substrate heating [10]. Moreover, the low deposition temperature is required for majority technologies of the material modification. There are needed an additional substrate cooling systems.

In this study, the comparing of magnetron sputtering systems with cooled and hot chromium targets and properties of Cr deposited films will be presented. Chromium as the target material was selected due to a wide applications for hard and tribological, corrosion resistance coatings [18–20], the process of metallization of steel and other materials. Moreover, Cr has a high vapor pressure [21], it is the material with good sublimation properties. Thereby, we can investigate the evaluation of input of each mechanism (sputtering or evaporation) in deposition rates at lower temperature range than Cr melting point. It is a great importance in the view of the possible Cr target cracking at high temperatures. Besides, the numerical modelling and plasma analysis were strongly needed in our study.

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2. Experimental

2.1. Coating deposition

Cr films were deposited in an ion-plasma installation in Ar (99.95%) atmosphere at 0.2 Pa. The base pressure was 3×10^{-3} Pa. The strong unbalanced magnetron sputtering system with AISI 321 main body was equipped by commercial 90 mm-disk Cr targets (99.95%). For target heating, the magnetron sputtering system with indirect cooling and the special geometry of Cr target with voids were used (Fig. 1). The inner and outer diameters of voids were 16 and 70 mm, the depth was 1 mm. This configuration of the hot target sputtering system provided a low damage to the target and didn't practically impact on the stability of the deposition process. The using of Cr cathode with equal thickness (8 mm) for cooled and hot target magnetrons resulted in the identical magnetic fields over cathode surface and it was made possible to objectively compare deposition rates for different magnetron configurations.

In this study, a pulsed 5 kW-power supply (APEL-M series, Applied Electronics) with limitation of output voltage (1 kV) and increased capacitor (up to 2 mF) was used. The current pulse was a triangular shape. The pulse repetition frequency was 15 kHz, duty cycle 0.25 (the pulse-on and pulse-off times were 16 and 52 μs, respectively). The glass (25 × 50 mm^2) and stainless steel (25 × 25 mm^2) substrates were placed facing to Cr target. The substrate holder was grounded. The distance from target to substrate was 80 mm. Before coating deposition, all substrates were cleaned in an acetone during 1 h and treated by a wide-aperture ion source during 20 min in Ar atmosphere at 0.1 Pa. The deposition modes of Cr films are presented in Table 1. In our study we will use a *power density* (in W/cm^2), which is defined as an averaged discharge power per target area.

2.2. Plasma and film characterizations

The optical emission spectra of plasma discharge were measured by a double-channel (225–920 nm) spectrometer AvaSpec-ULS2048L-2 with 1800 and 1200 groove mm^{-1} gratings and 0.09 nm wavelength resolution. The integration time was set 1.05 ms, the number of measurements for averaging was 20. The collimating lens was set at 300 mm from the target center and 3 mm from Cr target plane. We used NIST atomic spectra database (ver. 5.3) for the identification of spectral lines. For analysis dynamics of plasma and erosion processes, intensities of Ar^+, Cr and Cr^+ were used.

The film thickness was measured by spherical microsection method (Calotest CAT-S0000, CSEM). XRD measurements of Cr films were performed using a Shimadzu XRD-7000S with accelerating tube Cu-Kα (40 kV and 30 mA) in 2θ from 35° to 85° with exposure step 0.03° and scanning angle 1°. The film hardness was determined by Nano Hardness Tester (NHT series, CSEM) with Vickers 100 μm-indenter (Rockwell C type). We used a depth sensing method from the load displacement. The indentation depth was equal to 10% of the film thickness. To minimize an experimental error, the hardness measurements were carried out in 10 points for each sample. The adhesion of Cr films to substrate was estimated by scratching the film surface (the diamond 100 μm-indenter, Micro-Scratch Tester, CSEM). The load was from 0 to 15 N with a maximal indenter rate (7 mm/min). Then, adhesion tracks were investigated by optical microscope.

Table 1
Deposition parameters.

No	Target state	U [V]	I_{av} [A]	I_p [A]	W_{av} [kW]	W_p [kW]	Q [W/cm^2]	t [s]
C1	Cooled	591	2.11	17.4	1.25	10.3	19.7	533
C2		614	2.44	20.1	1.5	12.3	23.6	476
C3		635	2.76	22.7	1.75	14.4	27.5	414
C4		655	3.05	25.1	2	16.4	31.5	387
H1	Hot	561	2.22	18.3	1.25	10.3	19.7	505
H2		574	2.66	21.9	1.5	12.6	23.6	426
H3		592	2.91	25.4	1.65	15.0	25.9	356
H4		617	3.01	26.4	1.75	16.3	27.5	218
H5		633	3.21	28.2	2	17.9	31.5	181

Note: I_{av} – averaged current in pulse; I_p – peak target current; W_{av} – averaged power in pulse; W_p – peak discharge power; Q – target power density; t – deposition time.

2.3. Numerical modelling

The sputtering and evaporation of solid-state target have a different physics and aren't depended on each other. Thus, the target erosion was proposed to separation of two independent components at elevated temperature. We believe that the sputtering rate is a proportional to the sputtering yield and the ion current density on the target and practically is independent of temperature. The calculation approach of sputtering rates was described in [8]. On the contrary, the evaporation rate is determined by surface temperature. There are needed a calculations of evolution of surface temperature in plasma discharge, which is defined by the equation of heat conductivity. The boundary conditions were determined by a spatial-temporal distribution of ion current on target, energy losses on the heat radiation, the cathode sputtering, the evaporation and limited by a heat sink in the cooled magnetron body. More details of calculations are presented in [7]. The dependence of evaporation rate is a non-linear and equal to exponent [22]. So, the summary erosion rate is also non-linear with average pulse power comparing to the sputtering process.

3. Results and discussion

3.1. Deposition rates

Fig. 2 shows the experimental (points) and calculated (solid lines) deposition rates of Cr films and calculated temperature on the cathode surface (dashed line) as a function of the target power density. Experiments of deposition Cr films were provided in the case of the temperature stabilization on the cathode and are in a good agreement with calculation results. For the cooled target magnetron sputtering, Cr cathode erosion is performed only by sputtering. Thus, the deposition rate is proportional to the power density on the target and didn't exceed 5 nm/s.

For the hot target magnetron sputtering, the non-linear increase of deposition rates is observed. The results of numerical modelling indicate

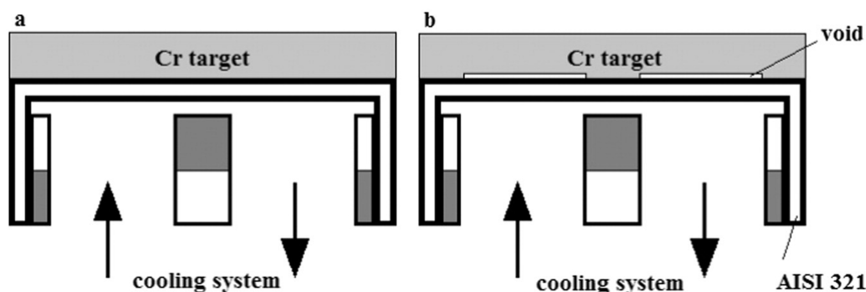

Fig. 1. The scheme of cathode devices with cooled (a) and hot (b) Cr targets.

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