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## A comparative study on the properties of chromium coatings deposited by magnetron sputtering with hot and cooled target

D.V. Sidelev<sup>a,\*</sup>, G.A. Bleykher<sup>a</sup>, M. Bestetti<sup>a,b,\*\*</sup>, V.P. Krivobokov<sup>b</sup>, A. Vicenzo<sup>a</sup>, S. Franz<sup>a</sup>, M.F. Brunella<sup>a</sup>

<sup>a</sup> Tomsk Polytechnic University, 30, Lenin Avenue, Tomsk, 634050, Russian Federation

<sup>b</sup> Politecnico di Milano, Department of Chemistry, Materials and Chemical Engineering "Giulio Natta", Milano Italy

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### ABSTRACT

This article reports on the analysis of energy flux density to the substrate and results of the detailed study of properties of Cr coatings deposited by magnetron sputtering with hot and cooled Cr targets. It was demonstrated that deposition rates of Cr films increase and particle flow density on the substrate changes by target sublimation. Heat radiation of the hot target leads to more rapid heating of the substrate and this energy flux has a main contribution in enhancement of total energy flux density on the substrate (from 0.06 to 0.43 W/cm<sup>2</sup>). As consequence, energy per deposited atom increases in 5.8 times for the hot Cr target sputtering even taking into account higher deposition rates. Cr films deposited by the cooled target sputtering have a (110) crystal texture, columnar microstructure, low surface roughness (~2.66 nm) and hardness from 13.8 to 14.2 GPa. In the case of the hot target sputtering, the competitive crystal growth of (110) and (200) directions is observed, microstructure of the Cr films is denser and homogeneous, grain size increases up to 200–300 nm and film surface becomes coarse ( $R_a$  ~11.75 nm), hardness of the Cr films drops by a factor of about 2.

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### 1. Introduction

Magnetron sputtering systems are extensively used techniques for conventional deposition of nanostructured coatings due to high quantity of adjusting work parameters (pressure, discharge power, ion current density on substrate, etc.). Nowadays, the extra interest in magnetron sputtering is directed to extend the range of operation parameters, which are required to meet new challenges in coatings deposition. One of these directions is the increase of deposition rates without degradation of functional properties of deposited films.

Hot target magnetron sputtering is a high-rate method of surface modification in comparison with conventional sputtering [1–4]. In the case of the hot target sputtering, the target can be heated up to elevated temperatures by the energy of bombarding ions, when the target has a particular heat insulation from a water-

cooled magnetron body. In this way, the target material additionally sublimates coupled with sputtering. The combination of these erosion processes results in the increase of deposition rates by several times, or more significantly, dependent on material properties and target temperature. For instance, the hot Ti target sputtering revealed the enhancement of deposition rates by up to 1.9 times [1]. The mathematical simulation of the hot target sputtering showed the technological possibilities to increase deposition rates by 5 and 20 times for Ti and Cr film deposition, respectively [5]. Besides, this sputtering technique has another strengths, it is effective to reduce a hysteresis effect in a reactive sputtering processes, even in an oxygen atmosphere [2,3]. In Ref. [6], the hot target sputtering was used to improve adhesion of metal films to the substrate, but the main feature of the hot target sputtering is that the heated (hot) target is a source of additional heat flux and flow of sublimated particles onto the substrate [5,7,8]. This causes significant changes of energy flux and particles flow to substrate during a pulse period, which are critical to structural and functional properties of deposited coatings.

In this study chromium films were deposited using cooled and hot target magnetron sputtering at typical power density of the conventional sputtering (20 ... 40 W/cm<sup>2</sup>). The interest in the

\* Corresponding author.

\*\* Corresponding author. Tomsk Polytechnic University, 30, Lenin Avenue, Tomsk, 634050, Russian Federation.

E-mail addresses: [sidelevdv@tpu.ru](mailto:sidelevdv@tpu.ru) (D.V. Sidelev), [massimiliano.bestetti@polimi.it](mailto:massimiliano.bestetti@polimi.it) (M. Bestetti).

chromium film deposition is based on the satisfactory sublimation properties of Cr [9] and high demand of metallic Cr coatings in decorative, mechanical and corrosion resistance applications [10,11]. The influence of the state of the sputtered target on the structure, morphology, topography and mechanical properties was investigated under the equal deposition conditions for both cases (hot and cooled target sputtering). Furthermore, the mathematical modelling was used to calculate the energy density and particle flow on the substrate over a wide range of deposition conditions. The analysis of these results by comparison with microstructure and mechanical properties of the Cr films reveals key factors of the hot target magnetron sputtering.

## 2. Experimental

### 2.1. Film deposition

Cr films were deposited in an Ar atmosphere (at 0.2 Pa) by sputtering of cooled and hot Cr targets (99.95%, 90 mm in dia, 8 mm thick) using a pulsed DC power supply APEL-M Series (operation frequency - 15 kHz, duty cycle - 0.25) with increased capacitance up to 2 mF. The schematic diagram of the deposition system is shown in Fig. 1a. The base pressure was  $10^{-3}$  Pa. The magnetron sputtering system has an indirect cooling of the Cr target by dint of a copper diaphragm (2 mm). In the case of the hot target sputtering, another construction of the Cr target with a radial cavity was used. The schematic diagram of the hot Cr target magnetron is presented in Fig. 1b. Additional description and construction details of the hot Cr target magnetron are reported in Ref. [4]. AISI 321 stainless steel plates and Si (100) wafers were used as substrates. The target to substrate distance was 80 mm.

Before the Cr film deposition, the substrates were set to face a wide-aperture ion source and treated by  $\text{Ar}^+$  for 20 min (2.5 kV, 50 mA). Then, the samples were rotated to the magnetron sputtering system and the Cr films were deposited at constant discharge power of 1.75 kW (target power density over pulse period  $W_{av} = 27.5 \text{ W/cm}^2$ ). Deposition conditions are presented in Table 1. In the case of the hot target sputtering, the Cr target was previously sputtered for 8 min to heat and to stabilize the target temperature which was determined by the attainment of the discharge current maximum.

Three different positions of substrates were fixed on the substrate holder in one deposition process (Fig. 1c). The substrates were thermally and electrically insulated from the substrate holder by using alumina ceramic interlayers. The substrates were separately grounded from the vacuum chamber. The scheme to measure the substrate current during films deposition is shown in Fig. 1a. The data were recorded using a digital oscilloscope Tektronix TDS 2022B. The substrate temperature was measured by using a chromel-copel thermocouple, which is connected to the backside of the central substrate.

### 2.2. Calculation of energy balance on the substrate

Previously, we used the mathematical model of heat and erosion processes for pulsed magnetron sputtering of a solid-state target taking into account sublimation and local evaporation. The previously obtained data of [5] is the basis of the current calculations.

For the high-rate magnetron sputtering with a hot target, the energy density on the substrate ( $F_{SUM}$ ) comprises heat flux density by target radiation ( $F_{RAD}$ ), heat flux density of condensation of deposited particles ( $F_{CS}$  - sputtered particles;  $F_{CE}$  - sublimated particles) and energy density by kinetic energy of sputtered ( $F_{KS}$ ) and sublimated ( $F_{KE}$ ) particles deposited on the substrate:

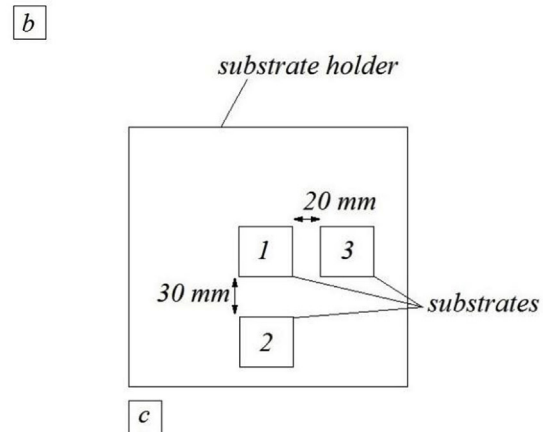
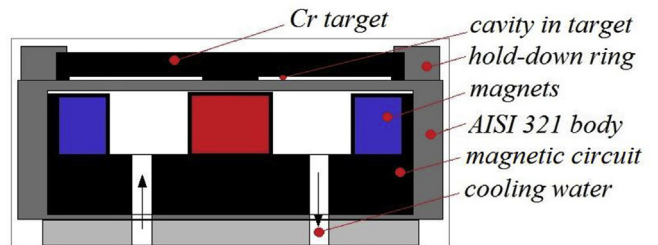
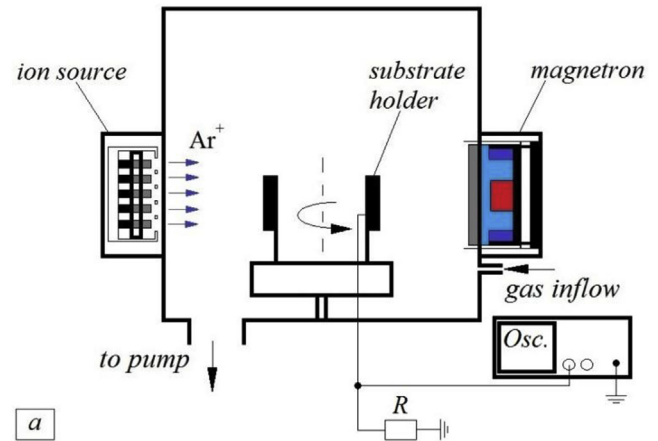


Fig. 1. (a) Schematic diagram of the deposition system, (b) the magnetron sputtering system with the Cr hot target and (c) the arrangement of samples on the substrate holder.

$$F_{SUM} = F_{RAD} + F_{CS} + F_{CE} + F_{KS} + F_{KE}. \quad (1)$$

At the stabilized deposition mode,  $F_{SUM}$  is a superposition of continuous and pulsed components of the energy density on the substrate. The energy flux density due to target radiation, condensation and kinetic energy of sublimated particles is continuous as is the target temperature ( $T_{tag}$ ) and these components do not change during a pulse period [5]. However, the temperature field of the target surface is non-uniform. So,  $F_{RAD}$  on the elemental area ( $1 \text{ cm}^2$ ) with  $(X, Y)$  coordinates was calculated based on the Lambert and Stefan-Boltzmann laws:

$$F_{rad}(X, Y) = \frac{L^2}{\pi} \int_{S_{tag}} \frac{\epsilon_p \sigma_{SB} (T_{tag}^4(x_{tag}, y_{tag}) - T_{sub}^4)}{(L^2 + (X - x_{tag})^2 + (Y - y_{tag})^2)^2} dx_{tag} dy_{tag}, \quad (2)$$

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