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Determining substrate temperature in an AC inverted cylindrical magnetron sputtering PVD system

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Abstract

The substrate temperature achieved during physical vapor deposition significantly affects the properties and phases of thin films. An Isoflux ICM-10 Dual Target Inverted Cylindrical Magnetron Sputtering system is used to deposit low temperature alpha-phase alumina, for which an experimentally determined substrate temperature is desired. Constraints inherent to the Isoflux system limit the use of a thermocouple. Instead, a pressure vessel containing numerous samples of temperature indicating liquids is placed in the chamber during operation. These indicator paints are observed upon completion of the deposition process to record the substrate temperature. Measurements indicate a temperature range of 343 °C to 399 °C for our alpha-phase alumina films deposited at a pressure of 0.27 Pa (2 mTorr), a substrate bias of −35 V, and a power of 5 kW. Theoretical heat transfer calculations support the experimental measurements. A parametric investigation showed that the removal of the −35 V substrate bias decreases the substrate temperatures by approximately 35 °C and that the substrate temperature increases about 50 °C for each additional kilowatt of power in the range of 1 kW to 6 kW.

Keywords: Substrate temperature measurement; Magnetron sputtering; Alumina films

1. Introduction

Alpha-phase alumina has always been a desirable coating material. It withstands temperatures of 1200 °C, possesses high hardness (24 GPa), and is resistant to corrosion and oxidization [1]. The commercial appeal of alpha-phase alumina films is based in large part on their potential application to high speed steels (HSS) and other cutting tools. However, the deposition of α-Al_2O_3 on steels is limited by the tempering temperature of these substrates.

Alpha alumina has been deposited by CVD [2] and PVD [3–9]. In CVD, the temperature of deposition is roughly 1000 °C, which generally ensures a good metallurgical bond between the substrate and coating. However, the thermal expansion mismatch between carbide substrates and the coating leads to residual tensile stresses that adversely affect adhesion by generating cracks. This process also limits the use of temperature sensitive substrates like HSS; it is therefore desirable to ensure that the substrate does not exceed 550 °C [3]. A suitable alternative to this process is PVD. Using this method, our laboratory has deposited alpha-alumina films at a rate of 330 nm/h, though at an unknown temperature.

While thermocouples are routinely used to monitor substrate temperatures during deposition processes [9–15], technical concerns inherent to the Isoflux ICM-10 Dual Target Inverted Cylindrical Magnetron Sputtering system restrict this method of measurement. Substrate biasing causes an electrical interference between the power supply and thermocouple. While this can be mitigated, the necessary steps add unnecessary cost and complexity to the system. Alternative procedures were investigated, and temperature indicating liquids were selected as the appropriate alternative. In this paper, we will confirm the validity of this method and present results for varying system parameters.

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2. Experimental

Tempilaq G temperature indicating liquids were acquired from Tempil, Inc. of South Plainfield, NJ [16]. The advantages of this product are its ease of application and its direct indication of temperature. The liquids are simply applied by brush to the desired surface where they quickly dry into an opaque film. Once the rated temperature of the paint is reached, it undergoes a distinct phase change that can be observed visually. The use of multiple paints enables one to further discern the temperature. The manufacturer claims an accuracy of ±5 °C.

Selected paints were tested on thin stainless steel test slides in a Lindberg/Blue furnace, model number BF51894C-1. The samples were exposed to temperatures near their rated melting point for 45 min, approximately four times greater than that considered necessary to reach equilibrium. This procedure was then repeated, but this time the paints were applied to the interior wall of the pressure vessel to be used in the Isoflux ICM-10.

Because the ICM-10 and other PVD systems operate at very low pressures on the order of 0.27 Pa (2 mTorr) [17–18], the melting paints and their gaseous emissions pose a potential danger to the internal components of the system. To protect these parts, the paints are contained in a small pressure vessel of AISI 316 stainless steel featuring copper sealant rings. This 13 cm tall pressure vessel is then placed inside the deposition chamber at locations where components to be coated would be placed (Fig. 1). Note that Fig. 1 shows the size of the pressure vessel to scale in relation to the deposition chamber. Multiple temperature indicating liquids, covering a suitable range of temperatures, are painted along the inside wall of the vessel. It is assumed that the presence of the pressure vessel within the deposition chamber has a negligible effect on the working gas and thus the measured temperature.

For the majority of experiments, the pressure vessel was removed and disassembled after 60 minutes of deposition. The temperature indicating paints were observed and assessed for their reaction. Additional repetitions of 15, 45, and 90 min were conducted to verify both steady-state at 60 min, and the accuracy of the theoretical heat transfer model.

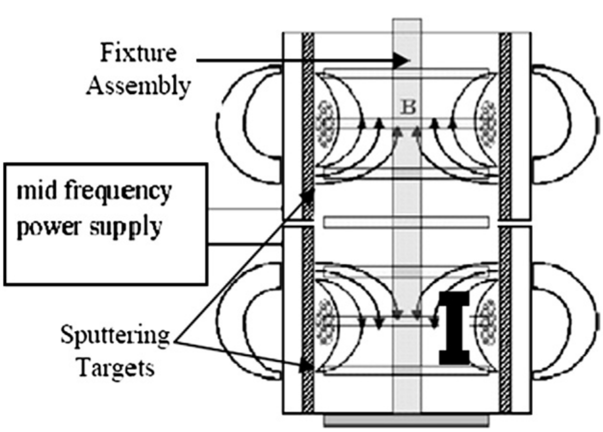

Fig. 1. Experimental schematic showing the pressure vessel inside the ICM-10 chamber (to scale).

Table 1
Properties of AISI 316 stainless steel chamber

Emmisivity	0.57
Density	8000 kg/m^3
Specific heat	500 J/(kg K)
Surface area	2.32×10^{-2} m^2
Volume	1.32×10^{-4} m^3

Multiple substrate biasing (0 V, −35 V, and −88 V) and power conditions (1 kW–6 kW) were imposed on the measurement apparatus. This range of bias and power is commonly used for plasma analysis and thin film deposition.

3. Numerical analysis

The heat transfer during deposition was modeled using numerical analysis methods. The total heat transfer is the sum of two components: radiation from the chamber walls ($\dot{q}_{\mathrm{rad}}$) and ion bombardment ($\dot{q}_{\mathrm{ions}}$). At our low deposition pressure of 0.27 Pa (2 mTorr), the contribution of thermal conduction to the working gas is found to be negligible.

The heat generated by the bombardment of argon ions is assumed to remain constant during the film deposition for a given substrate bias. The heat transfer due to radiation decreases over time as the substrate heats; it is given by Eq. (1)

$$\dot{q}_{\mathrm{rad}} = \varepsilon\sigma A\left(T_{\mathrm{S}}^{4}-T_{\infty}^{4}\right) \tag{1}$$

where e is the emissivity of the radiating surface, σ is the Stefan–Boltzmann constant, A is the surface area of the pressure vessel, T_{S} is the substrate temperature, and T_{∞} is the temperature of the chamber walls. It should be noted that T_{∞} is unknown; the targets of the ICM-10 are water-cooled and their temperature during the deposition process has not been recorded to date.

While the pressure vessel is expected to accumulate an alumina coating during the experiment, we still use the emissivity value for 316 stainless steel in Eq. (1). However, the typical value of both alumina and steel are comparable, and the resulting effect on our model is within the overall uncertainty of our experiment. Thus, our results and conclusions are unaffected by our choice.

The instantaneous change in substrate temperature with respect to time is given by the Eq. (2):

$$\frac{dT_s}{dt} = \frac{-\dot{q}_{\mathrm{ions}}-\dot{q}_{rad}}{\rho Vc}, T_{\mathrm{S}}(0) = 20°C = 293\mathrm{K} \tag{2}$$

where, t represents time, ρ represents the density of the steel vessel, V represents the volume of the steel, and c represents the steel's specific heat. Properties for the steel vessel are shown in Table 1 [19–20]. Explicit Euler time integration is used to solve Eq. (2) for substrate temperature as a function of time.

4. Results and discussion

To confirm the accuracy of the thermal indicating liquids, samples were first tested in the controlled conditions of a