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IR emission from the target during plasma magnetron sputter deposition

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ABSTRACT

In this article, energy flux measurements at the substrate location are reported. In particular, the energy flux related to IR radiation emanating from the titanium (10 cm in diam.) target surface is quantified during magnetron sputter deposition processes. In order to modulate the plasma–target surface interaction and the radiative energy flux thereof, the working conditions were varied systematically. The experiments were performed in balanced and unbalanced magnetic field configurations with direct current (DC), pulsed DC and high power impulse magnetron sputtering (HiPIMS) discharges. The power delivered to the plasma was varied too, typically from 100 to 800 W. Our data show that the IR contribution to the total energy flux at the substrate increases with the supplied sputter power and as the discharge is driven in a pulse regime. In the case of HiPIMS discharge generated with a balanced magnetic field, the energy flux associated to the IR radiation produced by the target becomes comparable to the energy flux originating from collisional processes (interaction of plasma particles such as ions, electron, sputtered atoms etc. with the substrate). From IR contribution, it was possible to estimate the rise of the target surface temperature during the sputtering process. Typical values found for a titanium target are in the range 210 °C to 870 °C.

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1. Introduction

Cold plasma based magnetron sputtering is one of the work horses of the coating industry. Magnetron sputtering offers various advantages such as low pollution level and precise control of the coating chemistry and microstructure which, in turn, determine the final properties of the coated surfaces [1]. It is well known that film features, and especially crystallinity and microstructure, widely depend on the energy delivered to the substrate and the thin film during the growth process [2]. During magnetron sputter deposition, the energy can be supplied through bombardment by ions, neutrals, electrons, photons, and/or through intentional heating of the substrate on its backside.

On the other hand, the sputter target's top most surface is always heated during the sputtering process, even if it is properly cooled, as it is bombarded by the plasma ions accelerated in the cathode sheath. In most cases, IR emission from the hot target surface is completely ignored as its interaction with the growing film. One of the main reasons lies in the difficulty to evidence such energetic contribution by conventional discharge diagnostics [3]. Nevertheless, IR photons can be absorbed by the growing film relatively efficiently depending on the optical properties of the latter. This was suggested by Mercs et al. in the

case of the deposition of Al_2O_3 thin films by reactive magnetron sputtering from a hot (i.e. not cooled) aluminum target [3], although no quantification of the IR radiation flux was reported.

In this work, the energy delivered to the growing film was directly measured during magnetron sputter deposition processes. An energy flux diagnostic tool specially designed for low-pressure plasma processes was used [4]. The sensitivity and the accuracy of this tool were previously demonstrated in several plasma systems [4–7]. It has been shown that, thanks to the fast time response of the sensor (thermopile), the energetic contribution of the plasma can be separated from thermal processes such as IR emission from the heated target. The aim of this work was to determine how the plasma–cathode surface interaction influences the radiative energy flux at the substrate position. Therefore, the evolution of the heat flux attributed to IR photons was studied as a function of the cathode magnetic field configuration, the discharge type, and the sputter power. The energetic contribution due to IR emission was quantified in terms of magnitude and kinetics. Finally the temperature of the sputter target was deduced for each working condition.

2. Experimental procedure

The diagnostic is based on a commercial heat flux microsensor (Vattell-HFM-7). The HFM is composed of a thermopile sensor (approx. 6 mm in diameter) which provides a voltage variation directly

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Table 1HiPIMS and pDCMS parameters used for the study of the influence of each discharge type at an argon pressure of 0.66 Pa and a mean power of 400 W.

Parameters	HiPIMS	pDCMS
Peak power (kW)	20	420
Pulse ON time (μs)	20	18.8
Pulse OFF time (μs)	480	1.2
Repetition rate (kHz)	1.950	50
Duty cycle (%)	4	95

proportional to the incoming energy flux density. It is calibrated according to a NIST protocol based on IR radiation emitted from a black body [4]. The HFM voltage was registered every 0.5 s using a nanovoltmeter (Keithley 2182). In order to avoid any radiation losses by IR emission from the sensor itself, the HFM was water-cooled down to 5 °C. Since the sensor temperature is not allowed to rise, a clear positive energy flux is always measured. A 6 mm diameter copper substrate was glued on the HFM active area using a thermally conductive paste. Previous works have shown that this copper plate does not disturb the measurements [6,8].

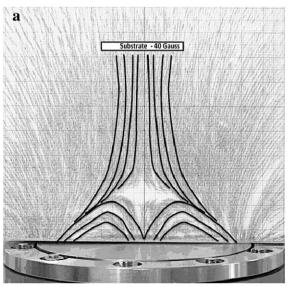
The energy flux diagnostic was mounted in a conventional magnetron sputtering experiment dedicated to the deposition of titanium films. Energy flux measurements were performed 8 cm away from a water-cooled, 10 cm in diameter, 6 mm-thick, titanium target. DC magnetron (DCMS), pulsed DC magnetron (pDCMS) and high power impulse magnetron sputtering (HiPIMS) discharges were ignited successively. More details about the experimental setup and the HiPIMS pulse can be found in Konstantinidis et al. [9] and Cormier et al. [10]. To study the influence of each discharge type, pressure and time-averaged power were set to 0.66 Pa (argon) and 400 W, respectively. Discharge parameters are given in Table 1 for HiPIMS and pDCMS. In each case the magnetic field configuration was either balanced (B) or unbalanced (UB). The field lines corresponding to these two magnetron cathodes are reported on Fig. 1. The strength of the parallel component of the magnetic field, ~1 cm from the target surface, equals ~300 G for the unbalanced magnetron cathode, while it equals ~400 G in the case of the balanced magnetron cathode. The magnetic field trap is therefore more efficient in the case of the balanced discharge. For a given target voltage, the discharge current and the ion bombardment are thus increased for this latter case. In Fig. 1, the substrate (or energy flux probe) position is also indicated. Measurements were also carried out in DCMS, 0.66 Pa and at various powers in the 100 W to 800 W range.

3. IR contribution and target temperature

As it was previously demonstrated, HFM measurements allow separating contributions exhibiting different time scales [7]. Fig. 2a shows a typical HFM signal which represents the evolution of the energy flux with respect to time in the case of a balanced pDCMS discharge. A rapid energy deposition (hundreds to thousands of mW·cm⁻² per s) is detected when the plasma is switched on. This rise lasts about 1 s, which is the time resolution of our data acquisition system. This first rise is mainly attributed to plasma energetic particles such as charged particles (electrons and ions), sputtered atoms, and fast neutrals. The quantification of each of these contributions and their relative importance was previously carried out in the case of cathodic plasma sputtering experiments [7].

This sharp increase of the heat transfer is followed by a slower one $(1.4 \text{ mW} \cdot \text{cm}^{-2} \text{ per s})$. A saturation of the heat signal is finally reached after approximately 10 s. This time scale is typical of a gradually heating body [5] that emits IR radiations. During plasma magnetron sputtering processes, it is expected that the target surface, located in front of the substrate (or the HFM), is heated by the intense ion bombardment. The grounded chamber walls may also be heated up; however the temperature rise of the chamber walls (and other pieces of the equipment) is significantly limited: i) as particle bombardment is dramatically limited on these parts and ii) due to geometrical considerations. The assumption that the slow increase monitored at the substrate level is due to a thermal process is corroborated by the slow decrease (2 mW·cm⁻² per s) of the heat flux that is observed as soon as the sputtering plasma is switched off. The intensity of the IR radiation emitted by the target surface should slowly decrease as soon as the later starts to cool down.

It is seen on Fig. 2a that the signal related to the IR emission from the target saturates. At this point, thermal equilibrium is reached at the target surface. This may take a relatively long time to appear. Rough estimations of the contact thermal resistance of the system which is constituted of the thermal paste (Jelt compound silicone paste: $0.009~\rm W\cdot cm^{-1}~K^{-1}$), the Cu plate $(0.5~\rm W\cdot cm^{-1}~K^{-1}~[11])$, and the growing layer $(0.219~\rm W\cdot cm^{-1}~K^{-1}~for~Ti~for~example~[12])$, have shown that the energy flux measurements remain valid even if a thick



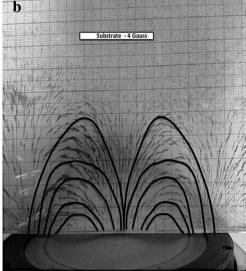


Fig. 1. Mapping of the magnetic field between the target surface and the substrate (8 cm) for a) the unbalanced magnetron cathode and b) the balanced one. The black lines underlining the field lines are added as a guide to the eye.

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