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Analysis of ion energy distribution at the substrate during a HPPMS (Cr,Al)N process using retarding field energy analyzer and energy resolved mass spectrometer

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ABSTRACT

The ion energy is known to have a strong influence on the properties of coatings deposited by physical vapor deposition (PVD). Therefore, the ion energy distribution (IEDF) especially measured at the substrate side is of great interest for understanding the coating growth. In PVD coating processes the ion energy at the substrate can be adjusted by applying a negative voltage (bias) to the substrate table. In the present work, mass integrated measurements of the IEDF were carried out during a high power pulsed magnetron sputtering (HPPMS) (Cr,Al)N process using a Cr target with 20 plugs of Al within an industrial scale PVD coating unit. The HPPMS cathode was operated with different average powers (1 kW, 3 kW and 5 kW) and pulse lengths (t_{on}) of 40 µs, 80 µs and 200 µs at constant frequency of 500 Hz. In a first step, measurements of the IEDF using retarding field energy analyzer (RFEA) were carried out on grounded substrate table and later on using −100 V substrate bias. Additionally, measurements of the IEDF were carried out using mass spectrometer (MS) exemplary to compare with the results from RFEA. By comparing the measured IEDFs some limitations of these two different measurement methods were found. The MS underestimates that the amount of high energetic ions and the RFEA measurement are influenced by the incidence angle of the ions. Besides those limitations the influence of cathode power and HPPMS pulse length on the IEDF was studied. At high peak power density, i. e. short pulse length and high average cathode power, a much greater high energetic fraction in the IEDF between 10 and 50 eV was found in the results of the RFEA compared to MS results presented here. Also the effective mean ion temperature in the high energetic region of the IEDF was found to increase with increasing peak power density. Furthermore, the effective mean ion temperature was not strongly influenced using substrate bias.

1. Introduction

In PVD (physical vapor deposition) coating processes the energy of the ions reaching the substrate has a crucial influence on the coating properties [1,2]. For controlling the energy of those ions commonly a substrate bias is used, i.e. a negative voltage is applied to the substrate table [3]. Thereby an electrical field is established and positive ions are accelerated to the substrate. In conventional magnetron sputtering plasmas the ionization is about 1% and thereby relatively low, but using the HPPMS (high power pulse magnetron sputtering) technology a much higher ionization in the order of 40% can be reached [3–8] and coating properties can be improved. The HPPMS technology is reviewed in detail by Lundin [9] and Sarakinos [10]. The much higher ionization of the plasma is desirable for controlling the ion energy at the substrate using a substrate bias because the energy of neutral atoms cannot be influenced. The ion energy distribution (IEDF) is often measured using energy resolved mass spectrometer [11,12]. Measurements comparing dcMS (direct current Magnetron Sputtering) and HPPMS show a much higher density of ions in the HPPMS plasma due to the higher ionization [5,6]. Also these measurements show the presence of high energetic ions in the HPPMS plasma. The high peak power density due to a high peak current during the HPPMS pulses can be considered the reason for this [13–15]. However, mass spectrometers have some limitations regarding their difficult positioning in the coating chamber. Furthermore, mass spectrometers have small acceptance angles in the order of 10° for an ion energy of 1 eV which decreases with increasing ion energy and the orifices are typically connected to the grounded chamber walls of the coating chamber [16]. Therefore, at the mass spectrometer orifice a voltage cannot be applied like at a substrate table. A solution for this problem is e.g. reported by Kadlec [17] where the mass spectrometer orifice was surrounded by an extraction electrode. In this work an industrial scale PVD coating unit is used and therefore an approach as reported in [17] is not possible without changing the geometry of the coating unit. Thus, another method was chosen to measure the time averaged ion energy distribution (IEDF) on the biased substrate table by a retarding field energy analyzer (RFEA). Its sensor can measure mass integrated IEDF on grounded as well as on surfaces with voltage

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applied [18]. Despite the possibility of using an RFEA to easily measure the IEDF on biased surfaces, retarding field energy analyzers are used only rarely to analyze HPPMS coating processes [19,20] especially in an industrial scale PVD coating unit. To ensure that the IEDF is measured on a biased surface correctly, in this work IEDF measurements were performed by RFEA with grounded and −100 V biased substrate table. The IEDF was measured for different average cathode powers and pulse lengths. The results from the RFEA measurements are compared with exemplary mass spectrometry measurements. For the measurements an HPPMS (Cr,Al)N process was chosen.

2. Experimental details

The investigations were carried out using an industrial scale PVD coating unit, CC800/9 Custom, CemeCon AG, Würselen, Germany. The coating chamber has a dimension of 100 cm × 100 cm × 100 cm. The coating unit has two cathodes, one equipped with a dcMS power supply GX 100/600 and one with a HPPMS power supply GX 150/1000, both by ADL GmbH, Darmstadt, Germany. The angle between the cathodes and the wall is approx. 45°. For the presented investigations only the HPPMS cathode was used. Onto the HPPMS cathode a chromium target with 20 aluminum plugs was mounted equivalent to approx. 60% chromium to 40% aluminum in the racetrack, chromium purity 99.9% and aluminum purity 99.5%, respectively. The target size was 500 mm × 88 mm. Measurements were carried out during a HPPMS-(Cr,Al)N process with argon and krypton as process gases (120/80 sccm.) The krypton was used for stability reasons [21]. Nitrogen was used as reactive gas. Its flow was determined by the pressure control to achieve $p = 450$ mPa. The nitrogen flow therefore was approx. $F(N_2) = 40$ sccm. Measurements were done using average cathode powers of $P = 1$ kW, 3 kW and 5 kW with $t_{on} = 200$ µs pulse length as well as $t_{on} = 80$ µs and $t_{on} = 40$ µs pulse length for $P = 5$ kW average cathode power. Pulse frequency as well as argon and krypton gas flows were not varied. Process temperature was approx. $T = 70$ °C. Heaters were not used because of temperature sensitivity of the plasma diagnostic equipment placed in the coating chamber. The process parameters are given in Table 1.

The cathode current and voltage during HPPMS pulse were measured using internal current and voltage probes of the PVD coating unit and an oscilloscope 3024A, Agilent Technologies, Santa Clara, USA. Within the coating unit an energy dispersive mass spectrometer (MS) and a RFEA were used for plasma diagnostics. The MS Hiden EQP 300, Hiden Analytical, Warrington, United Kingdom, was positioned at the substrate side using a flange in the wall opposite to the cathodes of the vacuum chamber, so that the orifice was in one axis perpendicular to the cathode, Fig. 1. In a second axis there was an angle of approx. 45° between the orifice of the mass spectrometer and the target. The flange position leads to a mismatch between the position of the mass spectrometer and the center of the target of approx. 2 cm. The distance between the MS orifice and the HPPMS cathode was 24 cm. The orifice had a diameter of 150 µm. It was electrically connected to the vacuum chamber wall and therefore grounded. A detailed description of the mass spectrometer is given by Budtz-Jørgensen [22].

The sensor of the RFEA, Semion pDC, Impedans, Dublin, Ireland, was placed on the substrate table next to the mass spectrometer (Fig. 1). Using a sensor holder, it was also oriented in one axis perpendicular to the HPPMS cathode and at the same height of the mass spectrometer orifice. The sensor of the RFEA was electrically connected to the substrate table, so that it can be either grounded or biased using a substrate bias. The potentials within the sensor of the RFEA are automatically related to the substrate's table potential. The acquisition time of the RFEA as well as of the MS was in the order of minutes and therefore the measurements were time integrated over many HPPMS pulses. Furthermore a three-point-smoothening was used. A detailed description of the RFEA can be found in [18].

Table 1
Process parameter of the HPPMS (Cr,Al)N process.

Process parameter	Value
Pressure p	450 mPa
Ar flow F(Ar)	120 sccm
Kr flow F(Kr)	80 sccm
N_2 flow $F(N_2)$	Pressure controlled
Substrate bias U_B	−100 V
Cathode power P	1/3/5 kW
Pulse duration t_{on}	40/80/200 µs
Pulse frequency f	500 Hz

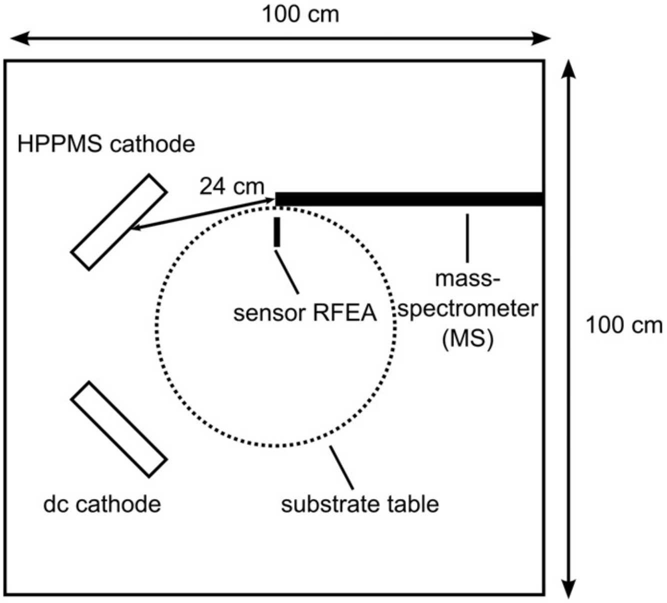

Fig. 1. Set up of the RFEA and MS within the vacuum chamber of the industrial scale PVD coating unit.

3. Results and discussion

3.1. Ion energy distribution without bias measured by RFEA and MS

At the beginning measurements of the IEDF using the RFEA and the MS with a grounded substrate table were carried out. IEDF measurements by the RFEA for several average cathode powers and pulse parameters are shown in Fig. 2. In the measured IEDF two different regions are visible: a symmetric peak centered at about 3 eV corresponding to thermalized ions which are accelerated in the plasma

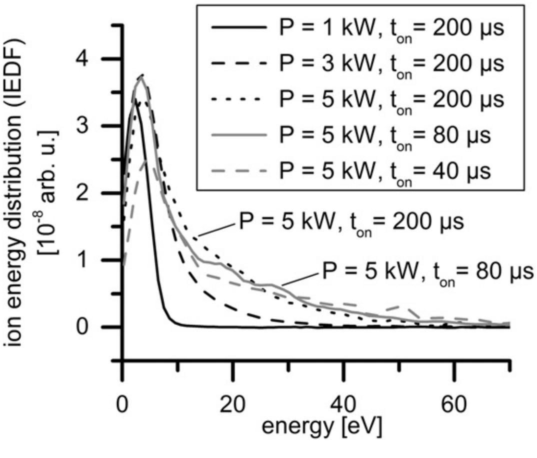

Fig. 2. Ion energy distribution measured using RFEA with grounded sensor for average cathode powers of $P = 1$ kW, 3 kW and 5 kW as well as for pulse lengths of $t_{on} = 40$ µs, 80 µs and 200 µs.