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Highly insulating alumina films by a bipolar reactive MF sputtering process with special arc handling

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ABSTRACT

Aluminum oxide (Al_2O_3) thin films are used in microelectronics and sensor applications due to their good insulating properties. Usually, RF sputtering processes are used to produce insulating coatings for sensors, but this process has major drawbacks, mainly, a very low deposition rate which leads to higher production costs. AC reactive sputtering processes present higher deposition rates, but issues regarding arcing and creation of defects in the films need to be addressed. In this work, an AC power supply with a new concept for fast arc handling and limited current output was investigated. Alumina films were produced by means of a bipolar reactive sputtering process, using large area double rotatable cathodes. The process was very stable and arc counts are as low as 3–10 arcs/h in the best deposition conditions. High deposition rates were also observed, reaching 2–3 μm/h. The films obtained excellent insulating properties with breakdown voltages higher than 3 kV for films with thicknesses between 2 and 3 μm and a corresponding breakdown strength of 1.5 kV/μm (15 MV/cm).

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1. Introduction

Aluminum oxide, commonly referred to as alumina, is a material of great technological importance with unique properties such as: high hardness, high resistivity and good chemical and thermal stability [1,2]. Due to these interesting properties, alumina thin films encounter many applications as an insulating layer for sensors and semiconductor devices [3–6], as hard-coating for tools or as wear-protective coatings [7–9]. In spite of its many applications, deposition of alumina thin films is not straightforward as the material possesses many different phases with differing properties, for instance, the α-phase which is the hardest and most stable phase, but industrially, only achievable with high temperatures (~1000 °C) and costly deposition processes [10–12], although lower deposition temperatures can be achieved by pulsed processes, highly ionized processes or substrate manipulation [7,13,14]. Nevertheless, for applications as insulating layers in microelectronics and sensors, low temperature deposition can produce amorphous films that already meet the requirements [15,16]. In these cases, film properties depend mainly on the type of deposition process. Among Physical-Vapor-Deposition (PVD) processes, the most commonly used for fabrication of alumina films is RF sputtering [9,16–18], since in this process, a ceramic target can be used with arc events being greatly suppressed. Furthermore, no sophisticated feedback control for reactive processes is required. Therefore, films do not usually present defects related to deposition of macro particles or droplets, although a major drawback of RF deposition is its very low deposition rate and high equipment cost. Mid-frequency AC pulsed processes present much higher deposition rates, are cheaper than RF systems and by means of a bipolar process with dual magnetrons, reactive sputtering can be realized with long-term stability by avoiding the disappearing anode problem [7,19]. In theory, it can also suppress arcs by using short negative pulse lengths, usually shorter than 20 μs, but in practice arcs may still be present. Especially reactive processes deal with non-ideal target surfaces due to redeposition of insulating material on the edges of the target, as well as other surface irregularities (roughness, scratches) which may lead to points of charge concentration with high charging rate. Therefore, power supplies must present an efficient arc handling mechanism.

In this work, a mid-frequency AC power supply with a specific concept for arc handling by limited current output was investigated. Alumina films were produced and characterized focusing on their insulating properties.

2. Experimental

Alumina films were deposited at a FHR SV470 in-line coater at Fraunhofer IST. The machine is equipped with dual rotatable cylindrical aluminum targets with a height of 550 mm and a diameter of 158 mm. Thus, the system is suited to study processes that can be scaled up from lab size to industrial application. Reactive processes were carried out with a working gas mixture of Ar/O_2. Oxygen partial pressure was controlled by using the optical emission signals from aluminum peaks as feedback

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and fast acting piezoelectric valves to adjust the oxygen flow. The plasma emission intensity (PEI) in a pure argon discharge was calibrated to 100%, with addition of oxygen in the chamber leading to different degrees of target poisoning and lower emission intensities. Depositions were performed in the transition region between the metallic and oxide modes, where measured plasma emission intensities were 20%, 30% and 40% in relation to a pure argon discharge. Argon flow was kept constant at 200 sccm and total working pressure was kept constant at 1 Pa by controlling the pump valve aperture. For the depositions, a PlasmaTec AC power supply was used, which is a mid-frequency AC power supply by J. Schneider Elektrotechnik GmbH, Germany. It delivers symmetric bipolar pulses at a fixed frequency of 38.5 kHz. Depositions were performed with average power of 4, 5 and 6 kW. Fig. 1 shows the experimental set-up including the power supply connected to the dual rotatable targets, voltage and current measurement, and the moving substrate. In order to improve the film uniformity, the substrate holder was set to an oscillating motion, moving continuously from left to right and vice-versa, in front of the target, with a speed of 20 mm/s and amplitude of 250 mm in relation to the center of the target. Films were deposited onto unheated glass substrates, and substrate temperature at different average power was monitored by temperature strips. Silicon substrates were used for SEM and EPMA characterization and stainless steel samples used for breakdown voltage determination. The experimental conditions for alumina deposition are listed in Table 1.

Discharge voltage was measured by a Tektronix P5200 high voltage differential probe. Discharge current was measured with a current monitor by Pearson, model 110. Time-dependent voltage and current profiles were recorded by a Yokogawa DL1640 digital oscilloscope. Surface morphology was studied with a Leo 1530 Gemini SEM microscope from Zeiss. Film thickness was measured with a Dektak 3-Profilometer. EPMA analysis for chemical composition and stoichiometry of the films was performed with a SX100 electron beam microprobe. Breakdown voltage was measured using an Elabo programmable high voltage tester according to the DIN EN 60243-1 standard. For these measurements, alumina films were deposited onto stainless steel substrates and then metallized with a copper film following a grid pattern, with grids of differing areas ranging from 15 to 100 mm^2.

3. Results and discussion

3.1. Pulse generation and arc handling

A typical pulse pattern for the PlasmaTec AC power supply is shown in Fig. 2, for a pure argon discharge with working pressure of 1 Pa and average power of 4 kW. The power supply features a voltage overshoot, which is used in the beginning of each pulse in order to get a fast current rise which results in almost rectangular current pulse shape, acting more as a current source rather than a voltage source. The controlled current flow is an important feature of the power supply and plays an important role in its arc handling system.

Fig. 3 shows the arc handling behavior of the power supply. This is possibly the most important feature with respect to obtaining films with low defect density and high insulating properties. When an arc event happens, the power supply quickly reacts and switches off. In this example, the power was switched off after 4 μs, but it is possible to set a response time as low as 1 μs. The time for switching back on can also be adjusted to give enough time for the arc to quench. It is important to notice that after there is a voltage drop associated to the arc event, no current rise is observed. This is due to the internal circuitry of the equipment and low residual energy, which results in a limited current output. This is a very interesting approach for arc handling since, if there is no current rise, less energy is provided to the arc spot, especially if the response time for switching the power off is very short. This will lead to a lower heating at the arc spot and lower probability of emission of macro particles due to melting of material, especially for materials with high melting point such as aluminum oxide. Therefore, it is expected that defects in the films are reduced. After reignition, the current and voltage curves behave the same way as before the arc event without long running-in behavior.

Table 1
Deposition conditions for alumina films.

Process	Reactive bipolar mid-frequency sputtering J. Schneider PlasmaTec AC power supply	
System parameters	Target material	Aluminum
	Distance target-substrate	10 cm
	Argon flow	200 sccm
	Oxygen flow	0–35 sccm
Process parameters	Substrate temperature	77–150 °C
	Total pressure	1 Pa
	Average power	4–6 kW
	Negative pulse length	12.7 μs
	Plasma emission intensity	20–40%
	Deposition time	60 min
	Duty cycle	100%
	Substrate material	Glass, silicon, stainless steel

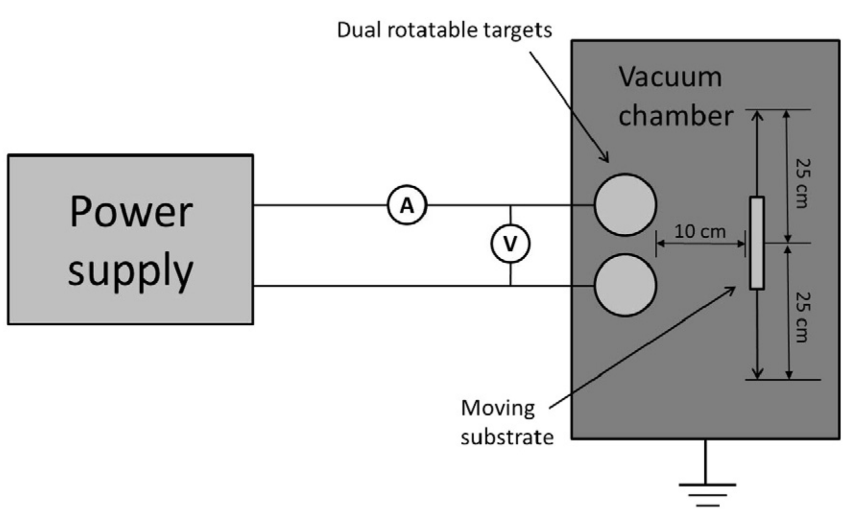

Fig. 1. Experimental set-up for the bipolar process. Each pole of the power supply is connected to one of the dual rotatable targets. Vacuum chamber is grounded. Voltage and current were measured and monitored with an oscilloscope. The substrate moves from left to right and vice-versa in front of the target in an oscillation motion of amplitude 25 cm. Target to substrate distance was 10 cm.

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