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Relation of hardness and oxygen flow of Al_2O_3 coatings deposited by reactive bipolar pulsed magnetron sputtering

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Abstract

Aluminum oxide thin films are widely used because of their excellent properties, especially in terms of chemical, thermal, abrasive and corrosive resistance. But many properties of alumina films are significantly deposition parameters dependent. Since different applications and environments demand different kind of properties in thin films, it is important to determine the influence of the deposition parameters on the alumina film properties. In this work, different alumina structures were deposited by means of reactive, bipolar, pulsed, magnetron sputtering. In order to find the appropriate parameter combination to synthesize crystalline alumina (for this investigation γ-Al_2O_3), substrate temperature, power density at the target and oxygen flow were varied. The γ-Al_2O_3 films were synthesized at 650 °C, 0.2 Pa, 800 W, 1:4 duty cycle, 19.2 kHz, and 11–12% oxygen flow. The structure and morphology of the deposited Al_2O_3 films were characterized by X-ray diffractometry (XRD) and scanning electron microscopy (SEM). Since the coating hardness is a decisive factor for many applications, the aim of this paper was to investigate the influence of the oxygen flow on the alumina hardness. It was observed that the hardness and the structure of the PVD-deposited alumina coatings are significantly oxygen flow dependent. The hardness of the alumina films was determined by nanoindentation. It varied between 1 and 25.8 GPa. The hardness increased by increasing oxygen flow until the target reached the poisoned state, where a hardness reduction was clearly observed.

Keywords: Gamma alumina; PVD coatings; Pulsed magnetron sputtering; Machining inconel 718; Tool coatings

1. Introduction

Amorphous and crystalline alumina thin films are widely used. Particularly, the synthesis of crystalline alumina coatings by means of PVD technology has aroused a keen interest of the scientist in the last years [1]. Motivations for this interest are the useful properties of the crystalline alumina and the important advantages offered by PVD technology [1]. Crystalline alumina offers higher density, hardness and also chemical and thermal resistance than the amorphous form. Furthermore, crystalline alumina is cost-efficient, electrically insulating and possesses a good heat conductivity in comparison to other ceramic materials [1–4]. Especially its high hot hardness and stable form at elevated temperatures provide an exceptional good wear protection and allow high cutting speeds [5]. In comparison with other deposition techniques such as CVD processes, PVD technology offers the possibility to produce high compressive stresses in the coatings (i.e. high alternating thermal stress resistance), keep sharp cutting edges, synthesize pure structures (without chlorides) and deposit various coating combinations such as multilayer TiAlN+γ-Al_2O_3. Furthermore, the combination of magnetron sputtering processes with pulsed power supplies allows the deposition of insulating coatings such as crystalline alumina at reduced temperatures, which implies a lower substrate thermal load [1]. Several investigations about the synthesis of crystalline by means of PVD-technology have been reported before [6–10]. But, although many properties of alumina films are significantly deposition parameters dependent, the relation between the deposition parameters and the alumina properties has not been exhaustively reported. Since different applications and environments demand different kind of properties in thin films, it is important to determine the influence of the deposition parameters on the alumina film properties.

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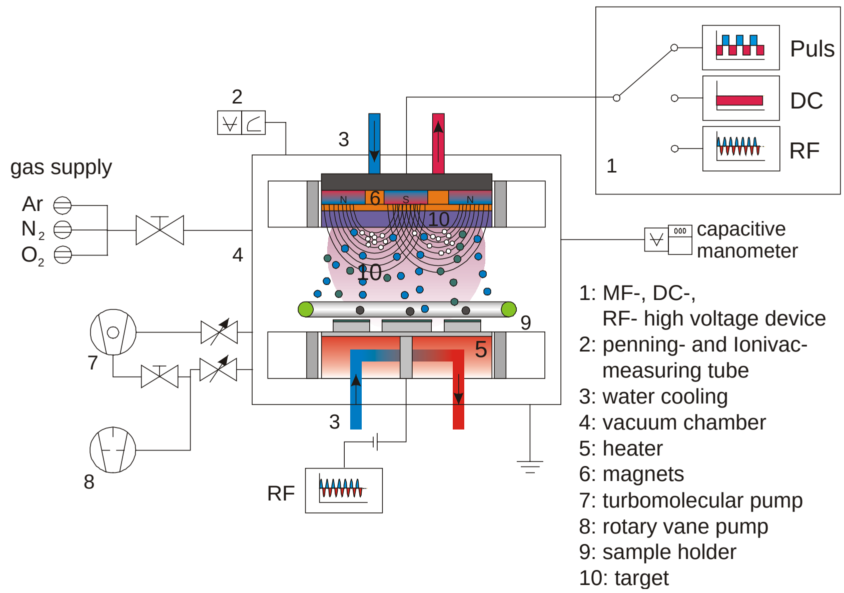

Fig. 1. Schematic drawing of the setup used for the deposition of the alumina coatings.

In this work, different alumina structures were deposited by means of reactive bipolar pulsed magnetron sputtering. In order to find the appropriate parameter combination to synthesize crystalline alumina (for this investigation γ-Al_2O_3), substrate temperature, power density at the target and oxygen flow were varied. Since the coating hardness is a decisive factor for many applications, the aim of this paper was to investigate the influence of the oxygen flow on the alumina hardness.

2. Experimental details

2.1. Samples preparation and deposition process

Tungsten carbide cutting inserts THM-11 were used as substrates. The samples were mirror polished ($R_a < 0.01$ μm) with a 6-μm diamond suspension and cleaned in a multi-stage ultrasonic cleaning bath with alkaline solutions of different concentrations and finally rinsed with de-ionized water and dried with nitrogen.

The Al_2O_3 films were deposited in a laboratory-sputtering device (Z400, Leybold-Heraeus) equipped with a bipolar pulser (SPIK™ 2000A, Melec). The pulser was used to increase the target power density without drastically affecting the substrate temperature. A 99.9 at.% aluminum target with a diameter of 75 mm was used as material source. TiAlN was deposited in some experiments as bonding layer. Oxygen was used as a reactive gas. All alumina coatings were deposited at 0.2 Pa in an Ar–O_2 atmosphere. Fig. 1 shows a schematic drawing of the used setup. Vacuum quality prior to deposition was $7.8 \cdot 10^{-3}$ Pa. The samples are heated up to 480 °C and ion etched during 30 min at an argon pressure of 1 Pa and 100 W RF. After etching, the reactive gas is introduced into the deposition chamber at a total pressure of 0.2 Pa. This low pressure was chosen to achieve a big mean free path and thereby a higher particle energy during deposition. In order to find the appropriate parameter combination to synthesize γ-Al_2O_3, substrate temperature, power density at the target and oxygen flow were varied as displayed in Table 1. Three different experiment series were performed. In the first experiment series, the oxygen flow was varied between 2% and 16% while the substrate temperature and the power at the target stayed constant at 550 °C and 600 W. In the second experiment series, the oxygen flow was varied between 3% and 16% while the substrate temperature and the power at the target stayed constant at 650 °C and 600 W. In the third experiment series, the oxygen flow was varied between 3% and 16%

Table 1
Deposition parameters of alumina coatings

Deposition parameter	Experiment series I	Experiment series II	Experiment series III
Pressure [Pa]	0.2	0.2	0.2
Oxygen flow [%]	2, 4, 6, 8, 10, 12, 14, 16	3, 5, 7, 8, 10, 12, 14, 16	3, 5, 6, 8, 11, 12, 14, 17
Substrate temperature during deposition [°C]	550	650	650
Power [W]	600	600	800
Frequency [kHz]	19.2	19.2	19.2
Duty cycle=$t_{on-}:(t_{on-}+t_{off-}+t_{on+}+t_{off+})$	1:4	1:4	1:4