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Thick HS-PVD $(Al,Cr)_2O_3$ coatings for challenging cutting and die casting applications

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ABSTRACT

In the last decades crystalline physical vapor deposition (PVD) Al_2O_3 coatings offered their great potential due to outstanding properties such as high hot hardness and wear resistance, especially concerning cutting applications. However, the properties of alumina strongly depend on the crystallographic phase. Thereby, the thermodynamically stable α-Al_2O_3 phase is the technical most interesting. The deposition of α-Al_2O_3 by chemical vapor deposition (CVD) is well-established, but requires high process temperatures, which reduce the selection of substrates. Another promising candidate concerning cutting and die casting applications is γ-Al_2O_3. The formation of γ-Al_2O_3 phase by PVD starts already at $T \geq 450$ °C, allowing lower deposition temperatures. Regarding the wear protection of turning tools, a higher coating thickness ($s \geq 10$ µm) is beneficial. A possibility to deposit thick PVD alumina coatings is the high-speed PVD (HS-PVD) technology. In the present work thick, $s \geq 20$ µm, $(Al,Cr)_2O_3$ films are deposited on cemented carbides at substrate temperatures $T \geq 500$ °C, by means of HS-PVD. A metallic (Cr,Al) bond coat was employed to improve the adhesion of $(Al,Cr)_2O_3$ coatings. In order to analyze the influence of the bond coat regarding the compound adhesion, scratch tests were conducted. For determining the coating morphology and thickness, scanning electron microscopy (SEM) was used. Phase analysis was carried out by X-ray diffraction spectroscopy (XRD). The mechanical properties were determined by means of nanoindentation. Regarding the use in high temperature applications, especially the formation of $(Al,Cr)_2O_3$ at substrate temperatures of $T \approx 850$ °C indicates that the use of the coatings is possible at equal high temperatures without phase transformation.

1. Introduction

The demand of high thermal stability, adhesion resistance, high hot hardness and wear resistance in a variety of applications lead to a widespread use of thin alumina films, due to their outstanding properties. In particular, typical applications are cutting operations [1–4]. In this context the deposition of thin alumina coatings is either carried out by chemical vapor deposition (CVD) or physical vapor deposition (PVD) technologies [5–7]. Alumina exists in different crystallographic phases, whereby the formed phase significantly influences the property profile of the coating. Depending on the process parameters, the deposition of amorphous Al_2O_3, the metastable crystalline γ-, δ-, θ- or κ-phases or the thermodynamically stable α-phase is possible [5, 8, 9]. The industrial deposition of the thermodynamically stable α-Al_2O_3 is mainly conducted by CVD, whereby the formation of α-phase requires high deposition temperatures, $T \geq 1000$ °C [10, 11]. Thus, the choice of substrate materials is limited due to undesired property changes as a result of the heat influence during the deposition process. In comparison several research projects revealed that the metastable γ-Al_2O_3 phase can be deposited at temperatures below $T \leq 700$ °C by PVD technology [2, 12–16]. Besides the lower deposition temperatures, the PVD γ-Al_2O_3 coatings exhibit the advantages of higher fracture toughness and strength due to smaller grain sizes compared to α-Al_2O_3 [17, 18]. Concerning die casting applications, smaller grain sizes of γ-Al_2O_3 coatings promise a smoother surface of the die, allowing increased surface quality of the finished product. Furthermore, investigations of γ-Al_2O_3 coatings with regard to cutting applications revealed a reduced friction coefficient µ and adhesion during interaction of oxide coating and workpiece material while turning operation [19]. Due to reduced friction and adhesion an increase of service lifetime was determined [19]. Besides, the comparison of γ-Al_2O_3 PVD and CVD coatings verified an increased abrasion resistance of PVD coatings, due to higher brittleness of CVD coatings [19]. Nevertheless, the metastable nature of γ-Al_2O_3 must be taken into consideration due to the transformation into α-Al_2O_3 at elevated temperatures [8]. However, examinations regarding the thermal stability of γ-Al_2O_3 coatings deposited by means of

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magnetron sputtering (MS) PVD revealed a phase stability of γ-Al_2O_3 up to T_{at} = 900 °C at atmosphere and T_{vak} = 1000 °C in vacuum [18, 20]. Furthermore, investigations regarding the behavior of γ-Al_2O_3 PVD coatings in cutting applications and die casting of alumina alloys showed promising results concerning wear behavior, adhesion resistance towards aluminum melt and corrosion protection as well as increased lifetime [21–25]. Despite the benefits, the deposition of aluminum oxide coatings by PVD technology also offers disadvantages in particular with regard to the economic deposition of thick ($s \geq 10$ µm) wear protecting coatings, as required in turning operations for example. The economic deposition of thick PVD coatings is influenced by deposition rate ds/dt, stability of the reactive gas supported process and thickness depending residual stresses of the coatings. A PVD technology combining high deposition rates ds/dt and a high tolerance towards target poisoning is the high-speed physical vapor deposition (HS-PVD) technology. Due to promising characteristics of the γ-Al_2O_3 coating system as well as the HS-PVD technology, investigations concerning the deposition of Al_2O_3 coatings by means of HS-PVD technology were performed. The alumina phase formation during PVD process depends on sundry influence factors such as stoichiometry, sputtering power P_s and bias voltage U_B. Nevertheless, one of the most decisive factors influencing the phase formation of alumina is the substrate temperature T_{Sub}. In order to enable a reduction of the phase formation temperature of crystalline alumina during deposition process, Cr was added to the Al, which promises the intended reduction of formation temperature [6, 7]. The $(Al,Cr)_2O_3$ films were deposited by means of HS-PVD in a substrate temperature range between T_{Sub} = 500 °C and T_{Sub} = 850 °C to investigate the influence of temperature regarding $(Al,Cr)_2O_3$ phase formation. The coatings were analyzed by scanning electron microscopy (SEM), X-ray diffraction spectroscopy (XRD), nanoindentation (NI) and scratch test in order to analyze the microstructure, phase composition and mechanical properties of the coatings as well as the adhesion between the substrate and the coating. The coating development and analyses were performed considering the long term goal of using the coatings in cutting and die casting applications.

2. Experimental details

2.1. Deposition setup

The investigated coatings were deposited on cemented carbides WC-Co, THM 12, Kennametal, Friedrichsdorf, Germany, with a rectangular geometry of 12 mm × 12 mm × 4.8 mm (width x depth x height) for coating development and characterization. The WC-Co substrates were polished with a d = 3 µm water based diamond suspension to an arithmetic mean roughness Ra < 0.02 µm. After thorough specimen cleaning in ultrasonic baths and drying, the specimens were loaded into the coating chamber that was subsequently evacuated to a base pressure of approximately p = 3 Pa. The specimens were coated by means of HS-PVD technology, basing on a hollow cathode glow discharge (HCD) [26, 27]. The schematic setup of HS-PVD is shown in Fig. 1.

Fig. 2 represents the target configuration A, which was used for the coating deposition of all specimens concerning the analysis of the correlation between substrate temperature T_{Sub} and phase formation. The sputter target is composed of 60 individual target elements with two different widths, a = 30 mm and b = 20 mm, consisting of Al or Cr (purity: Al 99.5%,

Cr 99.95%), Fig. 2. The Cr targets were added in order to reduce the enthalpy of formation, enabling a decrease of process temperature, as described in [6, 7].

The entire coating process was divided into four process steps: heating, etching, coating and cooling. First, the specimens were heated up. Regarding the substrate temperature variation, the heater temperature was varied in the range of T_H = 550 °C to T_H = 950 °C, in order to investigate whether amorphous or crystalline coatings are deposited at these process temperatures. The influence of the process temperature was determined by two series. Therefore, the temperature after each coating process was raised by ΔT = 50 °C. During the first series four coating processes with heater temperatures in the range of T_H = 550 °C to T_H = 700 °C were performed. The second series also comprised four coating processes with heater temperatures in the range of T_H = 800 °C to T_H = 950 °C. The heater temperature T_H was kept constant during the whole coating process to avoid influences on phase formation by temperature fluctuations. When evaluating the results, it must be taken into consideration that the heater temperatures T_H do not correspond directly to the substrate temperature T_{Sub}. Table 1 illustrates that the substrate temperature T_{Sub} is well below the set heater temperature T_H. The substrate temperature T_{Sub} was measured by a thermocouple close to the sample.

Second, the samples were etched in an argon atmosphere with a direct current (dc) bias supply $U_B = -600$ V for t = 30 min in order to clean the surface and remove contaminations. In the third step, the coating of the specimens was performed. The power supply of the sputtering unit was turned on, leading to the formation of a dense plasma due to the ionization of the argon process gas, so that the target elements were sputtered, Fig. 1. The distance between both target surfaces forming the hollow cathode is not variable and fixed to a distance of d_{target} = 20 mm. The atoms, ions and clusters sputtered within the hollow cathode were transported by the argon flow Q_{Ar} to the substrate, enabling the coating of the samples at the entire surface without specimen rotation during deposition. The coating process was subdivided into two phases. First phase was performed without reactive gas flow supply depositing a metallic interlayer consisting of Al and Cr for about t_{INT} = 15 min, to enhance the adhesion between substrate and ceramic $(Al,Cr)_2O_3$ coating. During second phase, oxygen was supplied to the vacuum chamber starting the deposition of the $(Al,Cr)_2O_3$ top coating. The oxygen flow was set to Q_{O2} = 300 sccm for each process. All deposition parameters of the investigated samples are summarized in Table 1.

Depending on the heater temperature T_H, the intensity of the reactions inside the coating chamber varies due to an increased reactivity of oxygen with rising process temperature. Therefore, slight parameter adaptions were required to reach stable and comparable processes with increasing process temperature. In this context the current of the sputtering unit I_S was slightly increased with rising temperature. Furthermore, the coating time was adapted. First, the process time was increased to compensate process instabilities, T_{Sub} = 597 °C, A650, and T_{Sub} = 635 °C, A700. In this context, instabilities mean short sputtering interruptions of a few microseconds due to the intervention of HS-PVD arc management. At higher process temperatures, $T \geq 728$ °C, A800, the process time was decreased, because instabilities prevent deposition time higher $t \geq 135$ min. Therefore, the coating time was reduced twice at T_{Sub} = 728 °C, A800, and T_{Sub} = 811 °C, A900, to t = 105 min or rather t = 75 min. However, allowing a direct comparison of the different coating processes regarding the coating thickness s and process efficiency, the deposition rate ds/dt is used. The distance between the target and the substrate was set to d = 120 mm, Fig. 1. After coating deposition, the cooling step started, ending at a substrate temperature below T_{Sub} = 70 °C.

2.2. Coating characterization

Coating morphology, architecture and thickness were determined by means of cross sectional micrographs using scanning electron microscopy (SEM), DSM982 Gemini, Carl Zeiss GmbH, Jena, Germany, at the Central Facility for Electron Microscopy (GfE) at RWTH Aachen University, Aachen, Germany. Furthermore, the Cr:Al ratio of the coatings was measured by energy dispersive X-ray analysis (EDX). Surface roughness of the prepared samples and coatings was determined by means of confocal laser scanning microscopy (CLSM), VK-X210, Keyence, Neu-Isenburg, Germany. Thereby, the mean roughness Ra and the mean arithmetic height Sa were calculated. In this context

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