Preparation of α-Al_2O_3/TiN multilayer coating on Ti(C,N)-based cermet by laser CVD

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Abstract. α-Al_2O_3 and α-Al_2O_3/TiN multilayer films were prepared on Ti(C,N)-based cermet substrate by laser chemical vapor deposition.α-Al_2O_3 and NaCl-type TiN films were prepared at T_{dep} = 1148 K. α-Al_2O_3/TiN multilayer film showed dense structure of cross section, and its surface morphology consisted of aggregated spherical grains. The adhesion of α-Al_2O_3/TiN multilayer film prepared on Ti(C,N)-based cermet was higher as compared with α-Al_2O_3 film directly prepared on the cermet.

Introduction

Hard protective coatings, such as TiC, TiN and Al_2O_3, are widely deposited on WC–Co cemented carbide cutting tool inserts by chemical vapor deposition (CVD) in order to increase cutting performance and extend the life-time [1–3]. However, there was no report has yet appeared addressing the successful deposition of hard coatings on Ti(C,N)-based cermet cutting tool. Ti(C,N)-based cermets, typically Ti(C,N)-Ni cermets, are increasingly popular due to their enhanced wear resistance and low cost as compared with WC-Co cutting tool inserts [4, 5]. The hard coatings on Ti(C,N)-based cermets commonly display low adhesion because of oxidation of substrate and the formation of brittle phases at interface between coating and cermet [6]. The presence of Ni often causes a degraded mechanical performance of the coating because of diffusion of Ni forming micropores in both coated layer and the interface [7]. Oxide coating of α-Al_2O on cermet would be further difficult due to higher deposition temperature and more oxidized O-containing atmosphere, causing serious oxidation of Ti(C, N)-based cermet substrate. By using CO_2–H_2 atmosphere, the oxidation of Ti(C,N)-based cermet substrate would be significantly suppressed. On the other hand, TiN film can be employed as an intermediate layer to enhance the adhesion of the coating [8–10].

So far, we prepared α-Al_2O_3 films by laser CVD at a low-deposition temperature [11–14] and CO_2-H_2 atmosphere [15]. In the present study, we prepare α-Al_2O_3/TiN multilayer films were prepared on Ti(C,N)-based cermet by laser CVD.

Experimental

Ti(C,N)-based cermets (NS530, Tungaloy corp., 13 mm × 13 mm × 3 mm) were used as substrates. A cold-wall type CVD apparatus was used for deposition [11]. An InGaAlAs diode laser (wavelength = 808 nm) beam was introduced into the chamber through a quartz-glass window and was slightly expanded by lenses to about 20 mm in diameter at the substrate surface. The laser was operated in the continuous mode with laser power (P_L) fixed at 140 W. A thermocouple was inserted into a slot engraved from the back of the substrate to measure the deposition temperature (T_{dep}). α-Al_2O_3 film was prepared using $Al(acac)_3$ as precursor under CO_2–H_2 atmosphere [15]. TiN intermediate layer was deposited using $Ti(OiPr)_2(dpm)_2$ precursor under NH_3 atmosphere [16]. Evaporation temperatures of $Al(acac)_3$ and $Ti(OiPr)_2(dpm)_2$ precursor were 433 K, and their vapor were transported into the chamber using Ar gas at flow rates of 1.65×10^{-6} m^3 s^{-1}. NH_3 gas and CO_2–H_2 mixture gas (1:1 molar ratio) was introduced separately into the chamber through a double-tube nozzle. The flow rates of NH_3 and CO_2-H_2 mixture gases were fixed at 1.65×10^{-6} and 3.3×10^{-6} m^3

s^{-1}, respectively. The total pressure (P_{tot}) in the chamber was fixed at 0.2 kPa. The feed pipes and the nozzle were maintained at 523 K to prevent condensation of the precursor vapor.

The crystal phases of TiN films were examined by X-ray diffraction with Cu Kα radiation (XRD; Rigaku, RAD-2C). The surface and cross-sectional microstructure were characterized by using a scanning electron microscope (SEM; Hitachi, S-3100H). The adhesion between TiN film and substrate was evaluated based on ISO 26443:2008 using a Rockwell-type hardness tester (Future-Tech Corp, FR-1e).

Results and discussion

Figure 1 shows the XRD patterns ofα-Al_2O_3 and α-Al_2O_3/TiN multilayer films prepared at T_{dep} = 1115 K and 1148 K, respectively. In case of α-Al_2O_3 film directly prepared on Ti(C,N)-based cermet substrate (Fig. 1(a)), TiO_2 (rutile) and Ti_2O_3 phases were identified. Because a small amount of W and Co are contained in Ti(C,N)-based cermet substrate, the decarburization of Ti(C,N)-based cermet caused Co_6W_6C formation. In case of α-Al_2O_3/TiN multilayer film, α-Al_2O_3 and NaCl-type TiN phases were identified (Fig. 1(b)), implying that the formation of TiO_2 and Co_6W_6C was significantly suppressed.

Figure 2 shows the microstructure of α-Al_2O_3/TiN multilayer film prepared on Ti(C,N)-based cermet substrate at T_{dep} = 1148 K. A cauliflower-like structure consisting of aggregated spherical grains was observed on the surface (Fig. 2(a)). α-Al_2O_3 top-layer and TiN intermediate layer with respective thickness of 2.0 and 1.5 μm were observed in the cross section (Fig. 2(b)). Both α-Al_2O_3 top-layer and TiN intermediate layer showed dense microstructure.

According to ISO 26443, the adhesion of coatings can be evaluated by indentation with a Rockwell diamond indenter. The adhesion of coating can be rated on a scale of 0 to 3. Class 0 represents high adhesion, *i.e.*, no cracking nor adhesive delamination occurs after indentation. Class 1 permits some cracking without delamination. Class 2 and Class 3 represent low adhesion, in which the coating is partly or completely delaminated after

Figure 1. XRD patterns of α-Al_2O_3 (a) and α-Al_2O_3/TiN multilayer (b) films prepared on Ti(C, N)-based cermet substrate at T_{dep} = 1115 K and 1148 K, respectively (P_L = 140 W).

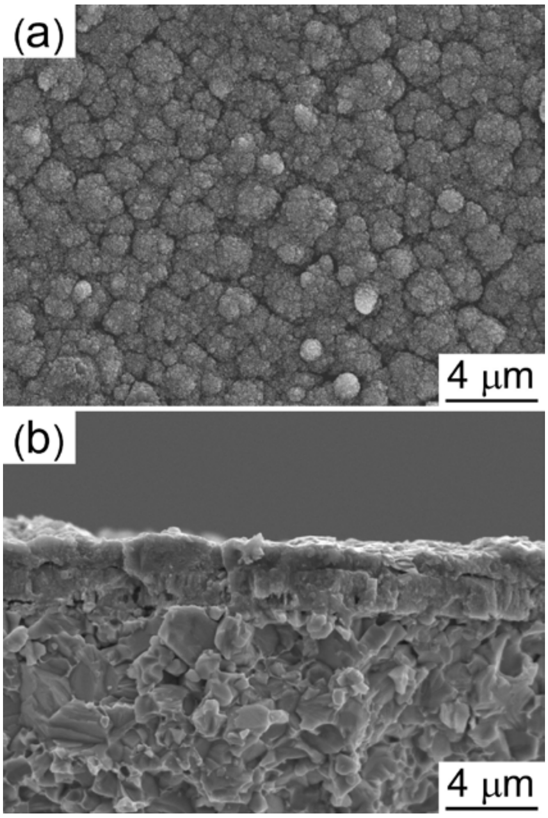

Figure 2. Surface (a) and cross-sectional (b) SEM image of α-Al_2O_3/TiN multilayer film prepared on Ti(C,N)-based cermet at T_{dep} = 1148 K (P_L = 140 W).

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indentation. Figure 3 shows the optical photographs of Rockwell indentation on α-Al_2O_3 and α-Al_2O_3/TiN multilayer films prepared at T_{dep} = 1115 K and 1148 K, respectively. After indentation, a large-area delamination was observed in the α-Al_2O_3 film (Fig. 3(a)), which can be classified as Class 2. The α-Al_2O_3/TiN multilayer films showed no evident delamination (Fig. 3(b)), which can be classified as Class 1. The adhesion of the coating is highly affected by interface morphology and material compatibility. TiN intermediate layer would exhibit high compatibility with the Ti(C,N)-based cermet substrate, leading to an improvement of adhesion between α-Al_2O_3/TiN multilayer and Ti(C,N)-based cermet substrate.

Summary

α-Al_2O_3 and α-Al_2O_3/TiN multilayer films were prepared on Ti(C,N)-based cermet substrate by laser CVD. The direct deposition of α-Al_2O_3 single layer on Ti(C,N)-based cermet substrate was accompanied by the oxidation of the cermet and a large-area delamination of α-Al_2O_3 layer after indentation. With introducing TiN intermediate layer, the formation of TiO_2 and Co_6W_6C was suppressed. The adhesion of α-Al_2O_3/TiN multilayer on Ti(C,N)-based cermet substrate was highly enhanced as compared with that of α-Al_2O_3 single layer.

Figure 3. Optical photographs of Rockwell indentation on α-Al_2O_3 film (a) α-Al_2O_3/TiN multilayer film (b) prepared on Ti(C,N)-based cermet at T_{dep} = 1148 K (P_{L} = 140 W).

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