

Effect of Precursor Supply on (100) and (001) Orientations of α -Al₂O₃ Film Prepared by Laser CVD

Kadokura Hokuto¹, Akihiko Ito^{1,a}, Teiichi Kimura^{1,b} and Takashi Goto^{1,c}

¹Institute for Materials Research, Tohoku University, Sendai 980-8577 Japan

^aitonoum@imr.tohoku.ac.jp, ^btei@imr.tohoku.ac.jp, ^cgoto@imr.tohoku.ac.jp

Keywords: α -Al₂O₃, Laser CVD, Orientation

Abstract. α -Al₂O₃ films were prepared by laser chemical vapor deposition and the effects of precursor evaporation temperature (T_{vap}) and oxygen gas flow rate (FR_{O}) on phase and orientation of Al₂O₃ films were investigated. At $T_{\text{vap}} = 413$ K, (100)-oriented α -Al₂O₃ and θ -Al₂O₃ were codeposited. The amount of θ -Al₂O₃ increased with increasing FR_{O} . At $T_{\text{vap}} = 433$ – 443 K, α -Al₂O₃ films showed a (001) orientation. (100)- and (001)-oriented α -Al₂O₃ films had a rectangular- and hexagonal-shaped grains, respectively, and showed a columnar in cross section. Grain size of (100)- and (001)-oriented α -Al₂O₃ films decreased from 10 to 2 μm with increasing FR_{O} from 0.085 to 0.85 $\text{Pa m}^3 \text{s}^{-1}$. Deposition rate increased from 100 to 300 $\mu\text{m h}^{-1}$ with increasing T_{vap} from 413 to 443 K.

Introduction

Since α -alumina (α -Al₂O₃) has high thermal and chemical stability, low thermal conductivity and excellent high-temperature hardness, α -Al₂O₃ coating has much attention as a protective coating for cemented carbide (WC-Co/Ni) cutting tools to improve its mechano-thermal properties and lifetime [1]. α -Al₂O₃ coating would enable Ti(C,N)-based cermet cutting tools to expand wide applications. Orientation and microstructural control of α -Al₂O₃ coating is highly demanded to develop high performance cutting tools [2]. α -Al₂O₃ coating has been commonly prepared by thermal chemical vapor deposition (CVD) [3]; however, the deposition temperature is usually more than 1300 K, which is too high to coat cutting tools, and orientation and microstructure is hardly controlled. We have developed a new CVD route using high-power lasers (laser CVD) for preparing various thick films with controlling nano- and macro-structures [4–6]. In laser CVD process, chemical reactions are significantly accelerated around a substrate, enabling low temperature and high-speed deposition with orientation control.

In the present study, α -Al₂O₃ films were prepared by laser chemical vapor deposition and the effects of precursor evaporation temperature (T_{vap}) and oxygen gas flow rate (FR_{O}) on the orientation of α -Al₂O₃ films was investigated.

Experimental procedure

A schematic of laser CVD apparatus has been reported elsewhere [7]. Aluminum acetylacetonate (Al(acac)₃) was used as a precursor. Al(acac)₃ was heated at $T_{\text{vap}} = 413$ – 443 K and its vapor was carried into a chamber with Ar gas (0.34 $\text{Pa m}^3 \text{s}^{-1}$). O₂ gas was separately introduced into the chamber through a double-tube nozzle. Flow rate of O₂ gas changed from $FR_{\text{O}} = 0.085$ to 0.85 $\text{Pa m}^3 \text{s}^{-1}$. The total pressure in the chamber was held at 0.9 kPa. Yttria-stabilized zirconia (YSZ) plate (12.5 mm \times 12.5 mm \times 1 mm) was used as a substrate. The substrate was heated on a hot stage at a preheating temperature 873 K, and a thermocouple was inserted backside of the substrate to measure the deposition temperature (T_{dep}). A continuous-wave mode Nd:YAG laser beam (wavelength: 1064 nm; diameter: 20 mm; output 192 W) was introduced through a quartz window to irradiate the whole substrate.

(001)-oriented α - Al_2O_3 film prepared at $T_{\text{vap}} = 433$ K and $FR_{\text{O}} = 0.17$ $\text{Pa m}^3 \text{s}^{-1}$ ($T_{\text{dep}} = 1199$ K) had a hexagonal terrace (Fig. 3(c)). Hexagonal terraces grew parallel to substrate surface can be associated with α - Al_2O_3 (001) plane. With increasing FR_{O} from 0.085 to 0.85 $\text{Pa m}^3 \text{s}^{-1}$, the size of hexagonal grain of (001)-oriented α - Al_2O_3 films decreased from 10 to 2 μm . (001)-oriented α - Al_2O_3 film also showed a columnar growth in cross section and the columnar grains were characterized by a feather-like structure (Fig. 3(c')). Deposition rate increased from 100 to 300 $\mu\text{m h}^{-1}$ with increasing T_{vap} from 413 to 443 K.

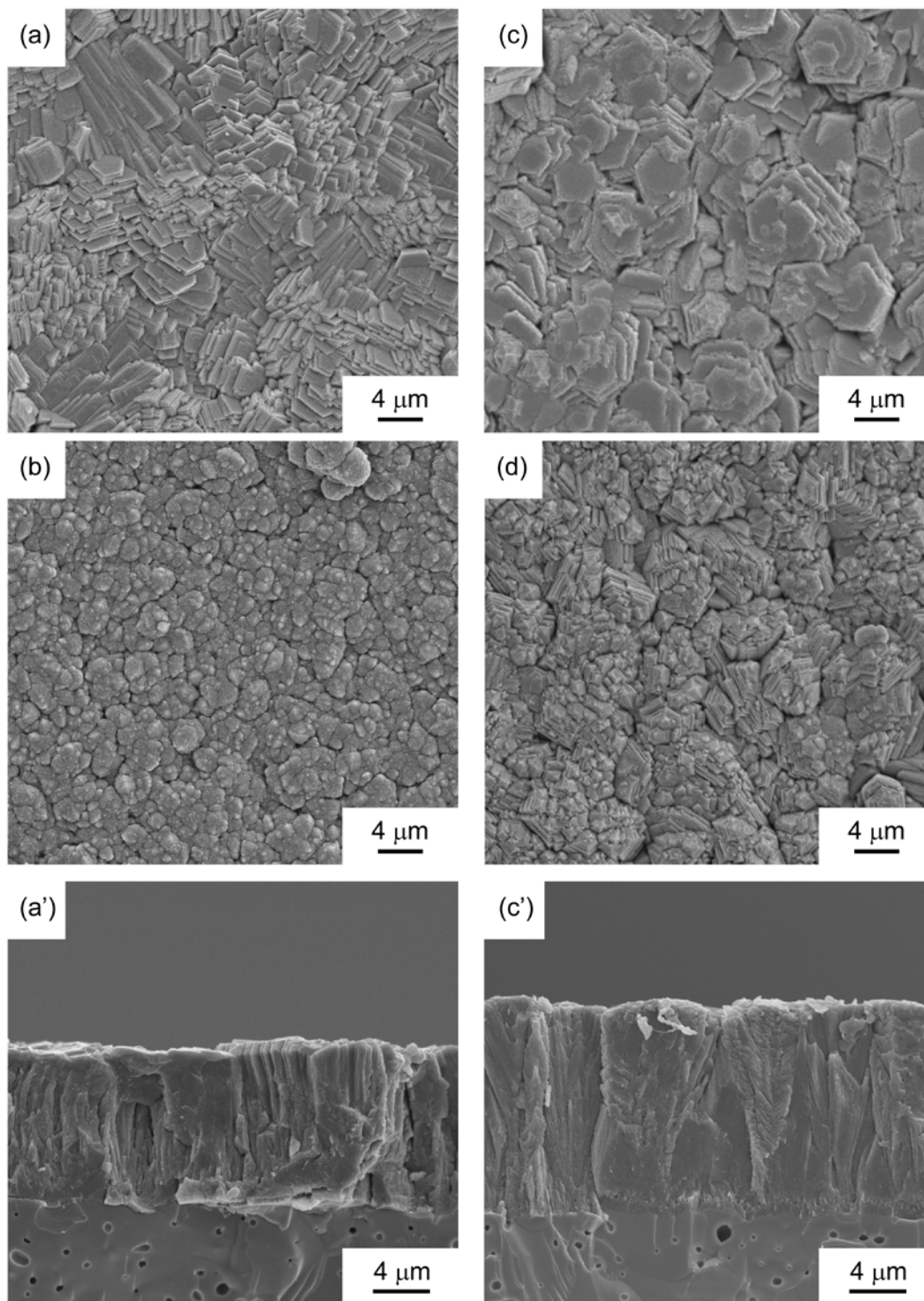


Fig. 3 Surface (a-d) and cross-sectional (a', c') SEM images of (100)-oriented α - Al_2O_3 films prepared at $T_{\text{vap}} = 413$ K and $FR_{\text{O}} = 0.17$ (a) and 0.85 (b) $\text{Pa m}^3 \text{s}^{-1}$, and (001)-oriented α - Al_2O_3 films prepared at $T_{\text{vap}} = 443$ K and $FR_{\text{O}} = 0.17$ (c) and 0.85 (d) $\text{Pa m}^3 \text{s}^{-1}$.

Summary

α -Al₂O₃ films were prepared on YSZ substrate at various T_{vap} and FR_{O} . (100)-oriented α -Al₂O₃ films were obtained at $T_{\text{vap}} = 413$ K. θ -Al₂O₃ was codeposited with (100)-oriented α -Al₂O₃ and the amount of θ -Al₂O₃ increased with increasing FR_{O} from 0.085 to 0.85 Pa m³ s⁻¹. α -Al₂O₃ films has no orientation at $T_{\text{vap}} = 423$ K. At $T_{\text{vap}} = 433$ –443 K, (001)-oriented α -Al₂O₃ films were prepared. (100)- and (001)-oriented α -Al₂O₃ films had a rectangular- and hexagonal-shaped grains, respectively, and showed a columnar growth in cross section. Grain size of (100)- and (001)-oriented α -Al₂O₃ films decreased from 10 to 2 μm with increasing FR_{O} from 0.085 to 0.85 Pa m³ s⁻¹. Deposition rate increased from 100 to 300 $\mu\text{m h}^{-1}$ with increasing T_{vap} from 413 to 443 K.

Acknowledgements

This work was supported in part by Global COE Program of the Materials Integration and the Rare Metal Substitute Materials Development Project, New Energy and Industrial Technology Development Organization (NEDO). This work was also supported in part by the Japan Society for the Promotion of Science, Grant-in-Aid for Young Scientists (B), No. 22760550, and by the Ministry of Education, Culture, Sports, Science and Technology, Grant-in-Aid for Scientific Research (A), No. 22246082.

References

- [1] M. Fallqvist, M. Olsson and S. Rупpi: *Surf. Coat. Technol.* Vol. 202 (2007), p. 837–843
- [2] S. Rупpi, A. Larsson and A. Flink: *Thin Solid Films* Vol. 516 (2008), p. 5959–5966
- [3] C. Park, J. Kim and J.S. Chun: *J. Vac. Sci. Technol. A* Vol. 1 (1983), p. 1820–1824
- [4] T. Kimura and T. Goto: *Mater. Trans.* Vol 44. (2003), p. 421–424
- [5] J. Endo, A. Ito, T. Kimura and T. Goto: *Mater. Sci. Eng. B* Vol. 166 (2010), p. 225–229
- [6] H. Kadokura, A. Ito, T. Kimura and T. Goto: *Surf. Coat. Technol.* Vol. 204 (2010), p. 2302–2306
- [7] Y. Yu, A. Ito, R. Tu and T. Goto: *J. Ceram. Soc. Japan* Vol. 118 (2010), p. 366–369
- [8] A. Ito, H. Kadokura, T. Kimura and T. Goto: *J. Alloys Compd.* Vol. 489 (2010), p. 469–474