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Title

Growth Mechanism and Properties of Alumina Prepared by Atmospheric Pressure Chemical Vapor Deposition (AP-CVD)

(800 words)

The work presented in this thesis deals with experimental and theoretical studies related to the growth mechanism and properties of alumina (Al_2O_3) by grown as a thin film and powder in atmospheric pressure chemical vapor deposition (AP-CVD) system using aluminum trichloride $(AlCl_3)/Ar/O_2$. Alumina (Al_2O_3) thin films are used in a wide variety of applications, ranging from microelectronics to catalysts and wear-resistant coatings. In order to achieve desired properties good control of the deposition conditions is required. In certain applications, e.g., as catalysts, metastable alumina is desired, while in many (high-temperature) applications the thermodynamically stable phase is needed. Consequently, phase control of alumina thin films. In particularly, low-temperature growth of Al_2O_3 has been studied intensely during the last decade. Deposition of Alumina thin film is through chemical bonding between the species incident onto the substrate. Aluminum oxide (Al_2O_3) is a polymorphic material utilized in a variety of applications, e.g., in the form of thin films. Many of the possibilities of alumina, and the problems associated with thin film synthesis of the material, are due to the existence of a range of different crystalline phases such as $(\gamma, \delta, \theta, \alpha, \kappa, \chi, \eta)$. Aluminum oxide (Al_2O_3) films were deposited by atmospheric pressure chemical vapor deposition (AP-CVD) system from aluminum trichloride $(AlCl_3)$, argon, and oxygen gas mixtures at temperatures ranging from 250 to 450 °C and 800 to 1000 °C. Alumina films with crystalline phases of γ - θ -, and α -alumina were obtained starting at 800 °C. Increase in the relative amount of the α -phase as well as improvement in crystallinity was observed as temperature is increased to 1000 °C. The films have low chlorine content, which continued to decrease with increasing temperature. Analysis of the film growth rate on tubular substrates of varying diameters revealed a diffusion-limited growth from 800 to 950 °C and gas-phase reaction-

The film mechanical properties also studied in detailed such as nanohardness, elastic modulus, film rougness, and Al/O ratio. Depending on the process parameters used, crystalline aluminum oxide thin films deposited on substrate had nanohardness between 8.25 and 15.09 GPa, elastic modulus between 104.62 and 279.03 GPa, and Al/O ratio between 0.32 and 0.78. The roughness of the aluminum oxide thin film on quartz substrate was between 15.2 and 30.3 nm. The nanohardness and elastic modulus of the aluminum oxide thin film on substrate are considerably same with others researchers.

Aluminum oxide powder formation by atmospheric pressure chemical vapor deposition (AP-CVD) using AlCl3/Ar/O2 system at 800 to 1000 °C was studied. The result showed that the particle size was decreased with an increase of reaction temperature in the range from 0.045 µm at 800 °C to 0.023 µm at 1000 °C.

We also tried to improve the sintering characteristics of microstructured γ -Al₂O₃ ceramics. The nanosized γ -Al₂O₃ (9.7 ± 3.2 nm) powders were added to microstructured γ -Al₂O₃ (1.9 ± 0.6 μ m) powders and they were well mixed. Its sintering behavior is studied in the temperature range of 1000 °C to 1300 °C and in holding time from 1 hour to 10 hours. Compacted samples with a different mixed ratio of nanosized and microstructured Al₂O₃ (N/M ratio) powders were prepared and then pressured on 1 GPa by a uniaxial pressing die. The phase transformation from γ -Al₂O₃ to α -Al₂O₃ took place at 1100°C for 1 hour in all compacted samples. The transformation rate is also increased with increasing N/M ratio. The relative density varied from 70% to 95% as a function of temperature and N/M ratio. With increasing sintering temperature from 1000°C to 1300°C, it was changed from 70% to 93%.

Finally, the mechanical properties, including Meyer hardness (H_M) and elastic modulus (E'), for compacted Al_2O_3 samples were investigated with variation of sintering temperatures and nanosized γ -alumina additives in microstructured γ -alumina powders (N/M ratio). The microstructured γ -alumina powders with $1.9 \pm 0.6 \, \mu m$ and nanosized powders with $9.7 \pm 3.2 \, nm$ were used as starting materials. The mechanical characteristic for sintered Al_2O_3 samples was done using the indentation test system, which consisted of a load cell, displacement sensor and indenter. The Berkovich and Rockwell indenters were used to measure the Meyer hardness (H_M) and elastic modulus (E') of samples, respectively. The Meyer hardness (H_M) for sample composed of nanosized Al_2O_3 only (N/M = 100 wt%) was rapidly increased from $0.3 \pm 0.03 \, GPa$ to $6.98 \pm 3.7 \, GPa$ in the temperature range from $1000 \, ^{\circ}C$ to $1300 \, ^{\circ}C$, while that of another samples were slowly increased in the same temperature range. At $1300 \, ^{\circ}C$, the Meyer hardness (H_M) of only nanosized Al_2O_3 sample obtained about $6.98 \, GPa$ and it was approximately $100 \, times$ larger than only microsized Al_2O_3 sample. For the elastic modulus (E') for nanosized Al_2O_3 samples, it increased from $90 \pm 10 \, GPa$ to $152 \pm 87 \, GPa$ in temperature range from $1000 \, ^{\circ}C$ to $1300 \, ^{\circ}C$. The Meyer hardness value ($6.98 \, GPa$) of only nanosized Al_2O_3 sample sintered at $1300 \, ^{\circ}C$ was lower than that for a fully dense alumina ($9.5 \, GPa$). This reason is that the hardness of samples decreases with an increase of porosity in samples.