Crystallization and thermal stability of nanocrystalline gamma-Al2O3 thin films prepared by reactive magnetron sputtering

Jakub Blazek¹, Radomir Cerstvy¹, Petr Zeman¹, Jindrich Musil¹

¹Dep. of Physics, Univ. of West Bohemia, Plzen, Czech Republic

blazekj@kfy.zcu.cz

The paper reports on the formation of transparent nanocrystalline (nc-) γ-Al2O3 thin films, thermal stability of γ-phase and its conversion to the thermodynamically stable α-Al2O3 phase during a post-deposition thermal annealing. Thin films were prepared by reactive magnetron sputtering in Ar+O2 discharge using a DC pulse dual magnetron with Al targets of diameter 50mm operated in the oxide mode at the repetition frequency of pulses f_r =40 kHz, duty cycle τ/T=0.5 and target power density W_t=25 W/cm² averaged over pulse period T on Si(100) substrates held at the floating potential U_s=U_fl with substrate temperature T_s=500°C. The total sputtering gas pressure was p_T =p_Ar+p_O2=1.5 Pa with the partial pressure of oxygen p_O2=0.2 Pa. The film structure was characterized by an X-ray diffraction. The post-deposition annealing was performed in a Setaram thermogravimetric system TAG 2400 with the heating rate 10°C/min and cooling rate 30°C/min. The effect of annealing temperature T_a, time of annealing at selected value of T_a and the film thickness h on film crystallization was investigated in detail. Mechanical properties of nc-γ-Al2O3 films were evaluated from load vs displacement curves measured using a computer controlled microhardness tester Fischerscope H 100. It was found that (1) the as-deposited films prepared under conditions given above are nc-γ-Al2O3 thin films, (2) the nc-γ-Al2O3 phase is thermally stable up to 1100°C; the γ phase is converted to α phase at T_a=1100°C, (3) the conversion of γ-Al2O3 phase to α-Al2O3 phase was achieved already after 1 hour of isothermal heating at T_a=1100°C, (4) γ-Al2O3 phase is converted to α-Al2O3 phase and it is accompanied by the film cracking due to different densities of γ- and α- phase; ρ_γ-Al2O3 =3.66 g/cm³ and ρ_α-Al2O3 =3.97 g/cm³ and (5) both the as-deposited and the thermally annealed nc-γ-Al2O3 thin films exhibit the hardness H≈12 GPa, the effective Young’s modulus E’≈140 GPa and elastic recovery W_e≈65 %.

The main result of our investigation is the finding that sputtered nc-γ-Al2O3 thin films are thermally stable above 1000°C and exhibit excellent elastic properties and the hardness sufficient for many applications.

Keywords
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reactive magnetron sputtering
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mechanical properties