The reaction interface between MgO and Al₂O₃

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The reaction interface between MgO and Al₂O₃ is a good prototype to understand the mechanism of solid reaction. MgO films were deposited on R-cut sapphire (1-102) substrates at different temperatures by pulsed laser ablation technique. It was found that the MgO films keep one orientation relation with sapphire substrates if the growth temperature is below 920°C. The films of good quality can be obtained at the intermediate substrate temperatures, between 700°C and 920°C [1]. The films were then annealed at 1100°C from a few minutes to 30 minutes to form a reaction layer. TEM observation was performed to investigate the interface in order to understand the solid reaction mechanism between MgO and Al₂O₃.

Fig. 1a presents the typical low-mag cross-section image of a MgO film grown on an R-cut sapphire. The MgO film consists of columnar grains, most of them epitaxially grow on R-plane of sapphire substrate by the same orientation [1]. A additional layer between MgO film and Al₂O₃ substrate evidently exist. The thickness of this layer increases with the annealing time. It is assumed that this layer is spinel formed by the solid reaction of MgO+Al₂O₃=MgAl₂O₄. The reaction layer is a single crystal layer. Fig. 1b is the corresponding selected area diffraction pattern of sapphire substrate, spinel layer and MgO film. The orientation relationship of the reactive layers are [11-20]₁₁₂₀ // [100]₁₁₂₀ // [010]₁₁₂₀ //≈ (001)₁₁₂₀ // (001)₁₁₂₀ (about 4° off). It is obvious that MgAl₂O₄ spinel keeps the cubic-cubic relation with MgO or inherits the relation between MgO and Al₂O₃, indicating the reaction is determined by the orientation of MgO film grown on Al₂O₃ substrate.

Fig. 2 presents a HREM image of the interface between layers. A thin layer of spinel is evidently developed due to solid reaction between MgO (upper layer) and Al₂O₃ substrate (bottom layer). It is obvious that the interface between MgAl₂O₄ spinel and r-cut sapphire is flat although some interfacial steps present. The coincided site lattice, or four (1-104)₁₁₂₀ planes matching five (010)₁₁₂₀ planes, is evident, as marked with white arrows. However, the interface between spinel and MgO film is curve and interfacial dislocations of various distances decorate at the interface. Hence, the reactive fronts are different, or depend on the reactive components.

Fig. 3 is a series of electron energy loss spectra of O K-edge probed crossing the interface. Eventually, the first sharp O peak (marked with A) get broader, shows a small low-energy shoulder in the spinel, and became significantly strong in the MgO layer. The small peaks between main peaks (A and B) gradually emerge in the spinel layer: these peaks results from the interference effects of backscattered waves on the third or higher coordination shell of O atoms or cations [2]. The eventual variation of these peaks indicates the off-stiochiometric spinel layer, or the gradual interdiffusion between MgO and Al₂O₃. Obviously, the reaction is controlled by the ionic diffusion.

In summary, TEM study of the reaction interface between MgO and Al₂O₃ shows that the orientation relation of MgO films grown on Al₂O₃ will determine reactive front of solid reactions. The evolution of O K-edge indicates that the solid reaction is controlled by ionic diffusion.
References

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Fig. 1. (a) A cross-section micrograph of MgO columnar grains epitaxially grown on R-cut sapphire substrate. (b) The selected area electron diffraction reveals that the orientation between MgO and Al₂O₃ determines the orientation of reactive product spinel.

Fig. 2. A HREM image of the interfaces of reaction layers. The bottom interface is the stepped interface between spinel and sapphire, while the upper part is the curved interface between spinel and MgO.

Fig. 3. A series of EELS O K-edge spectra shows the evolutions of O peaks.