

Transmission Electron Microscopy Studies of Epitaxial Superconducting MgB₂ Thin Film

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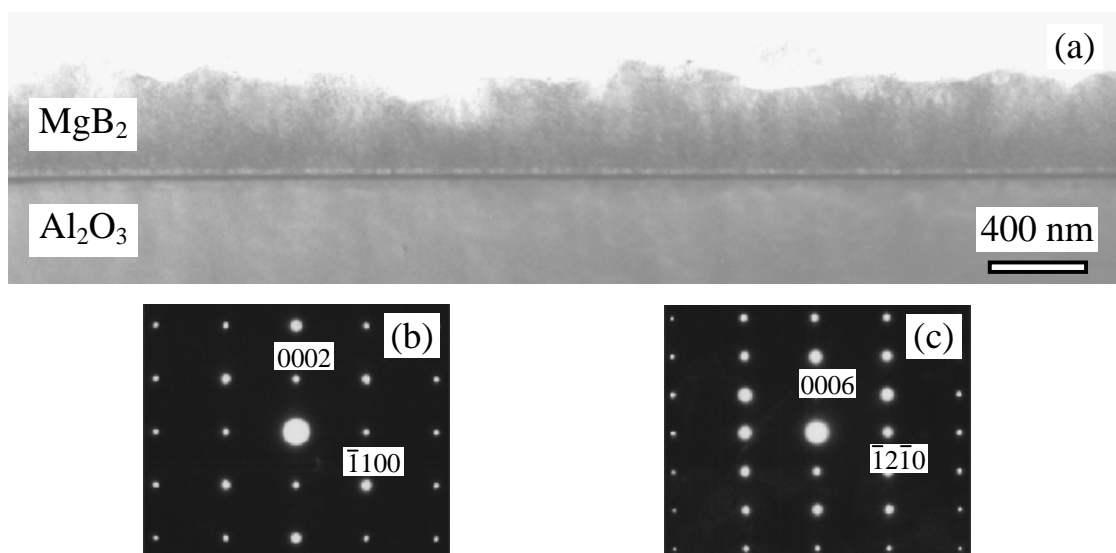
The recent discovery of superconductivity at 39 K in MgB₂ has resparked worldwide interest in non-oxide superconductors.[1] Among the prominent properties of MgB₂ are the record-breaking critical temperature in metallic superconductors and the ability to carry a strong-linked current flow.[2] Of the special interest to researchers are the fabrications of epitaxial MgB₂ thin films, which is one of the keys to facilitating the exploration of fundamental physics of MgB₂ and the applications in microelectronic devices.

Despite recent intensive efforts, the growth of epitaxial MgB₂ thin films remains challenging due to the high volatility of Mg in a broad temperature window and the tendency to form MgO in the presence of oxygen. Most recently, epitaxial MgB₂ thin films have been successfully fabricated by annealing boron precursor thin film in Mg vapor at high temperature. In this paper we report a transmission electron microscopy (TEM) study of such epitaxial thin films, with a goal to develop structure-property relationships and growth mechanisms.

An epitaxial MgB₂ thin film was fabricated by annealing a boron precursor thin film pre-deposited on the (0001) Al₂O₃ substrate in Mg vapor at 850 °C. Figure 1(a) is a bright-field low magnification cross-section TEM image of a MgB₂ film. Figure 1(b) and (c) are the selected-area electron diffraction pattern taken from the film and the substrate. The patterns were identified to be the [11 $\bar{2}$ 0] zone axis diffraction pattern of hexagonal MgB₂ and the [10 $\bar{1}$ 0] zone axis diffraction pattern of Al₂O₃. Detailed studies indicated that a phase-pure MgB₂ thin film grows on the (0001)Al₂O₃ substrate, with an epitaxial orientation relationship of (0001)MgB₂//(0001)Al₂O₃ and [11 $\bar{2}$ 0]MgB₂//[10 $\bar{1}$ 0]Al₂O₃, respectively. Interfacial structure between the epitaxial MgB₂ film and the Al₂O₃ substrate was analyzed using high resolution TEM (HRTEM). Figure 2(a) shows an HRTEM image of the interface with the electron beam incident along the [10 $\bar{1}$ 0] direction of the Al₂O₃ substrate. Between the MgB₂ film and the substrate, two intermediate layers exist. Fourier transformation, computer image simulations, and x-ray energy dispersive spectroscopy analysis revealed that two intermediate phases correspond to MgO (upper) and MgAl₂O₄ (lower). Additionally, these intermediate layers were epitaxially grown on the (0001) Al₂O₃. Figure 2(b) is an HRTEM image of the MgB₂/MgO interface taken with the electron beam incident along the [11 $\bar{2}$ 0] direction of MgB₂. A simulated image that optimally fits the experimental one is inserted in the middle of Fig. 2(b). The atomic structure of the interface determined is schematically shown in Fig. 2(c). The formation of the intermediate layers is ascribed to the presence of oxygen during the cause of annealing. The epitaxial orientation relationships between the Al₂O₃ substrate and the intermediate phases result from the favorable lattice matching between these phases. The existence of MgO layer provides a small lattice-mismatched template to facilitate the epitaxial growth of MgB₂.

[1] J. Nagamatsu *et al.*, Nature (London) **410**, 63 (2001).

[2] D. C. Larbalestier *et al.*, Nature (London) **410**, 186 (2001).



(a) Low magnification cross-sectional bright-field TEM image of a MgB_2 film grown on the (0001) Al_2O_3 substrate. (b) and (c) selected area diffraction patterns taken from the film and the substrate with the electron beam incident along the same direction.

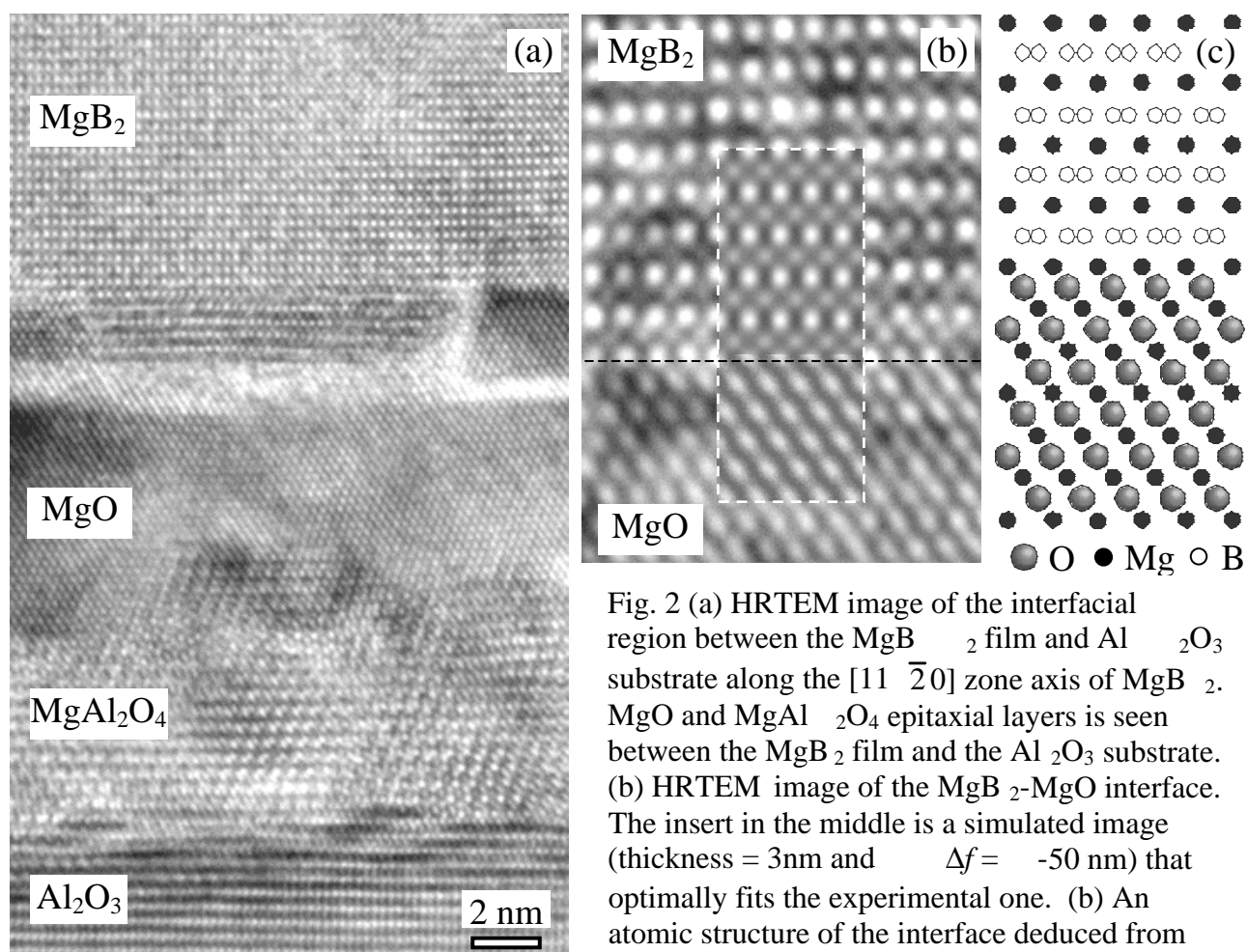


Fig. 2 (a) HRTEM image of the interfacial region between the MgB_2 film and Al_2O_3 substrate along the $[11\bar{2}0]$ zone axis of MgB_2 . MgO and MgAl_2O_4 epitaxial layers is seen between the MgB_2 film and the Al_2O_3 substrate. (b) HRTEM image of the MgB_2 - MgO interface. The insert in the middle is a simulated image (thickness = 3 nm and $\Delta f = -50$ nm) that optimally fits the experimental one. (c) An atomic structure of the interface deduced from the HRTEM and computer simulations.