Vol 11 No 12, December 2002
1009-1963/2002/11(12)/1300-03
Chinese Physics
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Superconductivity and x-ray photoemission study of MgB_2 thin films*

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(Received 21 August 2002; revised manuscript received 11 September 2002)

Highly c-axis oriented MgB_2 thin films with T_c^{onset} of 39.6K were fabricated by magnesium diffusing into pulsed-laser-deposited boron precursors. The estimation of critical current density J_c, using hysteresis loops and the Bean model, has given the value of 10^7 A/cm^2 (15 K, 0 T), which is one of the highest values ever reported. The x-ray photoemission study of the MgB_2 thin films has revealed that the binding energies of Mg 2p and B 1s are at 49.4 eV and 186.9 eV, which are close to those of metallic Mg and transition-metal diborides, respectively.

Keywords: superconductivity, x-ray photoemission, magnesium diboride
PACC: 7400, 7960, 7470

1. Introduction

The discovery of the MgB_2 superconductor with a T_c of 39K[1] has attracted considerable interest in both fundamental studies and practical applications.[2–10] MgB_2 was reported to be an anisotropic s-wave superconductor with a conventional BCS electron–phonon coupling[11] and is not compromised by the weak-link problem.[12] MgB_2 has a record superconducting transition temperature among the non-oxide and non-C60-based compounds. Its T_c is close to that of $La_{2-x}Ba_x(Sr_x)CuO_4$ and is two to four higher than those of conventional metallic superconductors, such as Nb_3Sn and Nb–Ti alloy. Besides, MgB_2 has a long coherence,[13] higher critical current density and upper critical field.[14,15] These properties make MgB_2 a good candidate for practical applications. To date, MgB_2 has been fabricated in various forms such as bulks, wires, tapes, single crystals and thin films. Among these, thin films are of particular importance because of their great potential in electronic applications and further basic studies.

In this paper, we report on the superconductivity and x-ray photoemission (XPS) of MgB_2 thin films. The films show c-axis orientation with a T_c^{onset} of 39.6 K. The critical current density of the films at 15 K in zero field is more than 10^7 A/cm^2. The XPS study has revealed that the binding energies of the Mg 2p and the B 1s in MgB_2 thin films are close to those of metallic Mg and transition-metal diborides, respectively.

2. Experiments

The MgB_2 thin films were fabricated by magnesium diffusing into boron precursors inside an evacuated quartz tube, as described previously.[16] The boron precursors were prepared on c-cut sapphire by pulsed-laser deposition at a substrate temperature in the range of 300–900°C. The resistance measurement was performed with the standard four-probe method. The magnetization hysteresis loops (MHL) were measured at 5 K, 10 K, 15 K, 20 K, 25 K, 30 K up to a magnetic field of 6T.

XPS spectra data were obtained with a VG ESCALAB5 spectrometer using Mg $K\alpha$ source radiation operating at an accelerating voltage of 11 kV and an emission current of 20 mA. The working pressure in the XPS chamber was approximately 2×10^{-7} Pa. To remove the oxide and other impurities on the surface

*Project supported by the State Key Programme for Basic Research of China (Grant No 1999064604).
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of MgB_2 thin films, the samples were bombarded by low-energy Ar^+ for about 5 min in the XPS chamber.

3. Results and discussion

Typical $\theta - 2\theta$ scans for MgB_2 thin films fabricated on Al_2O_3 (0001) substrates are presented in Fig.1. Only (00L) reflections of MgB_2 are observed in Fig.1, providing the evidence that the grown MgB_2 films are of single phase and in c-axis orientation. The full width at half maximum of the rocking curve of (002) reflection is only about 0.56°, suggesting that a high crystalline quality has been achieved for the films. A MgO (440) peak is also detected, which is introduced by the oxidation of the remnant Mg on the surface of the MgB_2 thin films in the diffusing process.

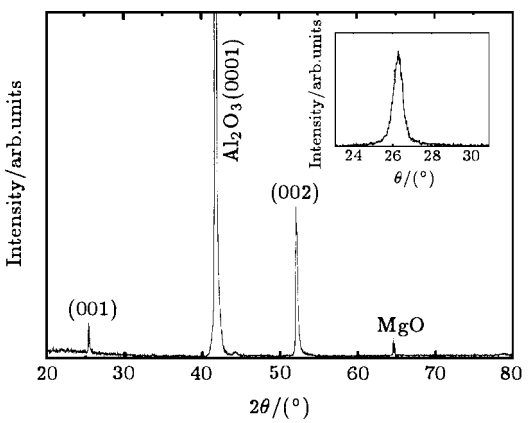


Fig.1. XRD pattern for MgB_2 thin films fabricated on AL2O3 substrates. The inset shows the rocking curve of MgB_2 (002).

The temperature dependence of the resistance of the MgB_2 films under a magnetic field is shown in Fig.2(a). The onset temperature of the superconducting transition is observed at about 39.6 K with a transition width ΔT_c=0.5 K. Measurements of the M–H loops at different temperatures were carried out on a $3\times4\times(3\times10^{-4})$mm^3 MgB_2 thin film with the applied field perpendicular to the sample surface. A typical flux-jumping pattern is present at temperatures below 15 K due to rapid flux penetration. The critical current density was obtained from M–H loops using the Bean critical model J_c=$20\Delta M/va(1-a/3b)(a<b)$. J_c versus magnetic field up to 6 T at 15 K, 20 K and 30 K is shown in Fig.2(b). J_c at 15 K and zero field is as high as 1.2×10^7 A/cm^2, which is one of the highest values ever reported,[15,17] as far as we know.

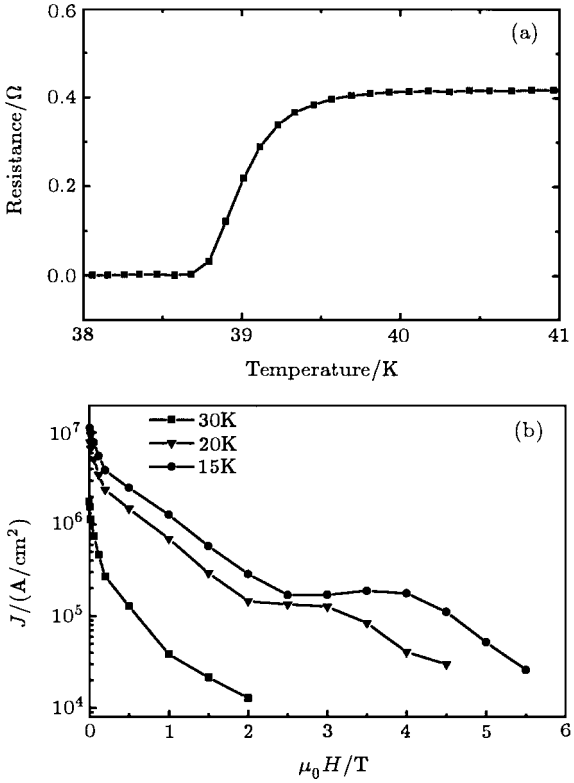


Fig.2. (a) Resistance versus temperature for MgB_2 thin films, and (b) transport critical current density (J_c) of MgB_2 thin films as a function of temperature.

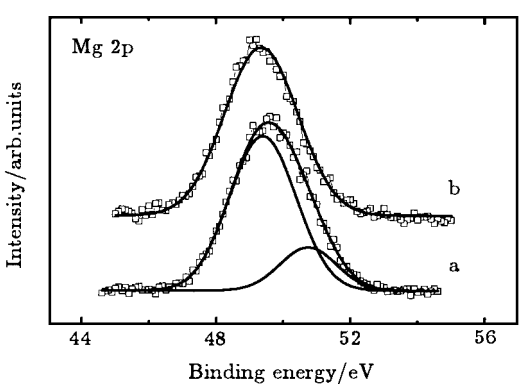


Fig.3. The Mg 2p core level spectra of MgB_2 thin films. The open square line represents the experimental spectrum and the solid line represents the Gauss-fitted spectrum: (a) before Ar^+ bombardment; (b) after Ar^+ bombardment.

Figure 3(a) and (b) show the Mg 2p core level spectra of MgB_2 thin films before and after Ar^+ bombardment, respectively. For both cases, a Gauss-fitted peak at 49.4 eV is observed, which can be assigned to the Mg signal in the MgB_2 phase.[18] Before the Ar^+ bombardment, an additional small peak is also observed at a higher energy, 50.5 eV, due to MgO. The MgO is mainly a surface contamination layer. We hardly detect any B 1s core level signal in MgB_2 thin

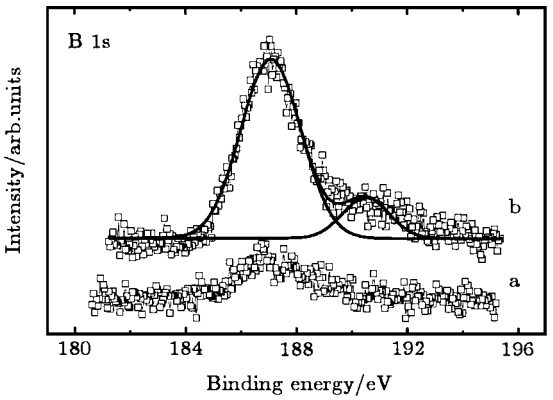


Fig.4. The B 1s core level spectra of MgB_2 thin films. The open square line represents the experimental spectrum and the solid line represents the Gauss-fitted spectrum: (a) before Ar^+ bombardment; (b) after Ar^+ bombardment.

films before Ar^+ bombardment, which suggests that the surface region of the MgB_2 thin films is oxidized and B-deficient. Figure 4(b) shows the B 1s core level spectra of MgB_2 thin films after Ar^+ bombardment. It appears to be composed of two contributions located at 186.9 eV and 190.3 eV. The higher binding energy peak corresponds to B_2O_3 [19] and the lower binding energy peak is somewhat close to B 1s in transition-metal diborides.[19,20] Therefore, we have assigned the peak at 186.9 eV to the B signal in MgB_2 phase.

4. Conclusion

Highly c-axis oriented MgB_2 thin films with T_c^{onset} of 39.6 K have been fabricated on a c-cut sapphire by magnesium diffusing into pulsed-laser-deposited boron precursors. An estimation of the critical current density J_c, using hysteresis loops and the Bean model, has given the value of 10^7 A/cm^2 (15 K, 0 T), which is one of the highest values ever reported. The XPS measurements revealed that the MgB_2 thin-film surface was oxidized and B-deficient. After Ar^+ bombardment, the Mg 2p and B1s core levels are at 49.4 eV and 186.9 eV, similar to those of the metallic Mg and transition-metal diborides, respectively.

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