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Effect of microstructure on properties of MgB_2 synthesized by SHS method

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Abstract

MgB_2 samples were obtained by self-propagating high-temperature synthesis (SHS). Microstructure, phase and chemical analysis of the samples were studied by XRD and cross-sectional TEM–SAD. MgB_2 samples contained very small uniformly distributed MgO particles. Temperature dependence of the upper critical field and of the critical current densities were determined from ac magnetic susceptibility measurements. Normal metallic state was characterised by measurements of valence band structure by ultraviolet photoelectron spectroscopy (ARUPS). We concluded that MgB_2 is a hard II type superconductor and that SHS method is suitable to fabricate material with strong pinning centers of MgO.

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Keywords: MgB_2; SHS method; Microstructure; Critical current

1. Introduction

Magnesium diboride (MgB_2) is considered as a new superconductor [1] having great potential for application and a lot of research activity has raised recently on this compound (for a review, see e.g. Ref. [2]). Magnesium diboride is a layered material with boron atoms forming graphite like layers and with magnesium atoms forming the hexagonal lattice. MgB_2 is a type II superconductor with highly anisotropic coherence length, penetration depth, critical fields and other electronic superconducting and normal state characteristics [3].

Here, we report on microstructure, temperature dependence of resistivity, upper critical field and critical current density as well as electron photoemission band spectra of MgB_2, prepared by the SHS technique, in view to fabricate a material useful for application.

2. Experimental results and their analysis

MgB_2 bulk sample was prepared by the SHS technique, described in detail elsewhere [4].

To analyse the nanostructure of the MgB_2, conventional transmission electron microscopy (TEM), energy dispersive spectroscopy (EDS) and selected area diffraction (SAD), using a Philips CM 20, equipped with a Link eXL EDS system, were used. Electron-transparent thin foils of 300 nm were prepared by dimpling on a Gatan Dimpler and ion-milled on a Gatan DuoMill 600.

XRD, using Ni-filtered CuKα radiation made on MgB_2 sample, revealed the presence of well-crystallised MgB_2 phase. Some small impurities of MgO and Mg were also identified [4].

Fig. 1 shows bright field TEM image of the MgO particles in the MgB_2 matrix. The studied sample is dense and contains very small MgO particles ranging from 10 to 70 nm in comparison to about 200 nm estimated from previous SEM observation [4]. The volume fraction of MgO particles in the MgB_2 matrix measured on basis of direct measurement of the particle

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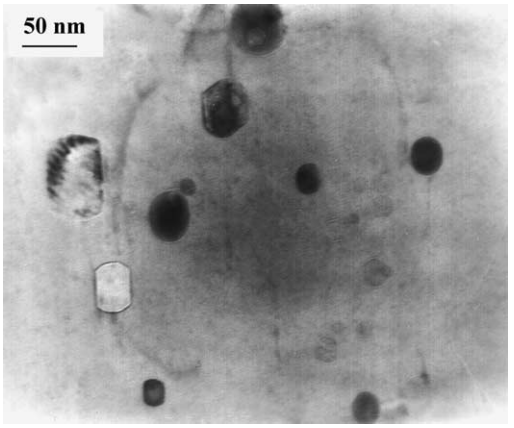

Fig. 1. Bright field TEM image of MgO particles in MgB_2.

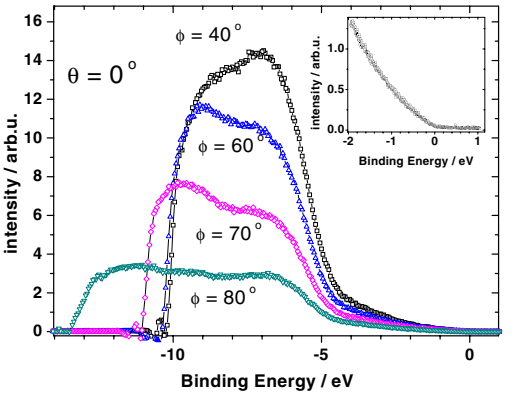

Fig. 3. ARUPS spectra for normal to surface ($\theta = 0°$) incidence ultraviolet radiation with He I energy 21.2 eV for the given angles of outcoming electrons Φ at room temperature.

projections performed on TEM micrographs, using TGZ-3 (Zeiss) apparatus was approximately 0.6% with the standard deviation 0.5%.

Basic physical characteristics were measured previously [4]. Resistive transition is $T_c = 37.4$ K (inset in Fig. 2). The normal state resistivity obeys the relation $\rho(T) = \rho_0 + \rho_1 T^n$ with $n \cong 2$ as for Fermi liquid. The width of transition is very sharp, $\Delta T = 0.6$ K, proving very good structural and chemical homogeneity of the material. The temperature dependence of upper critical field, $B_{c2}(T)$, shown in left panel of Fig. 2, was determined from ac susceptibility measurements for various ac and dc magnetic fields, taking into account the half of full Meissner state value of χ' susceptibility. The temperature dependence of critical current density, J_c was derived from the position of the peak of the absorption susceptibility, $(\chi'' T)$, at various ac and/or dc magnetic field employing the Bean critical state model and its extensions [5]. The critical current density was estimated from the relation $J_c \cong 2B^*/d$, where $B^* = B_{ac} + B_{dc}$ is a

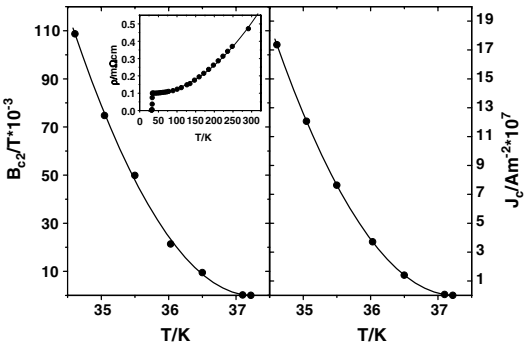

Fig. 2. Temperature dependencies of upper critical field, B_{c2} (left panel), and critical current density, J_c (right panel). The inset shows temperature dependence of resistivity. The solid lines are relevant fitted curves (see text).

sum of the ac and dc magnetic field and $d = 1$ mm is the sample dimension perpendicular to the magnetic field. Both, $B_{c2}(T)$ and $J_c(T)$, obey the $(1 - T/T_c)^n$ power relation with $B_{c2}(0) = 20.9 \pm 0.1$ T and $n = 2.0 \pm 0.1$, $J_c(0) = 1.7 \times 10^{10}$ A m^{-2} and $n = 2.2 \pm 0.2$, respectively. This empirical scaling relation was successfully applied for many superconductors, including high-T_c superconductors [6,7]. The very high critical field, about 20 T at helium temperature, makes this material a promising candidate for applications. From the obtained value $B_{c2}(0)$ an estimate of the BCS superconducting coherence length is $\xi(0) \cong 40$ nm. The length is comparable to the average distance between the MgO particles so that such secondary phase is strong pinning centre matrix. All these superconducting parameters indicate that MgB_2 is a hard type II superconductor.

Normal metallic state of the material was characterised also by measurements of valence band structure by angle-resolved ultraviolet photoemission spectroscopy (ARUPS) with OMICRON AR 65 spectrometer (Fig. 3).

We observed the angle dependence of valence band structure due to grain alignment of the highly anisotropic material. We did not detect any trace of the Fermi edge at E_F (see inset of Fig. 3). We concluded that the material is rather poor metal with small density of states at the Fermi energy. However, this problem needs further investigation.

Acknowledgements

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