PRAMANA
— journal of
physics

Vol. 58, Nos 5 & 6
May & June 2002
pp. 867–870

Preparation and characterization of MgB_2 superconductor

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Abstract. The MgB_2 superconductor, synthesized using solid-state and liquid-phase sintering methods, have been characterized for various properties. The upper critical field, irreversibility line and critical current density have been determined using magnetization data. The current–voltage characteristics recorded under an applied magnetic field revealed the existence of vortex glass transition. The surface analysis using X-ray photoelectron spectroscopy shows that MgB_2 is sensitive to atmospheric degradation.

Keywords. MgB_2 superconductor; vortex glass transition; X-ray photoelectron spectroscopy.

PACS Nos 74.70.Ad; 74.60.Jg; 33.60.Fy

1. Introduction

The recent discovery of MgB_2 superconductor ($T_c \sim 39$ K) has attracted great scientific as well as technological interest [1]. For device applications, various issues such as synthesis of dense samples under atmospheric argon pressure, dissipation mechanism, material stability etc., need to be fully investigated. In this paper, we present some of our results related to these issues.

2. Experimental

The synthesis of MgB_2 has been carried out using solid-state as well as liquid-phase sintering methods. The details of synthesis are described elsewhere [2]. Solid-state sintering process yielded polycrystalline samples having grain size of ~ 10 μm, while from the charge of liquid-phase-sintered material, dense MgB_2 grains (having a size up to $3 \times 2 \times 1$ mm^3) were retrieved. Powder X-ray diffraction measurements confirmed the formation of MgB_2 phase in both types of samples.

The temperature dependent resistivity measurements were carried out using standard four-probe method by mounting the sample in a closed cycle refrigerator. The magnetization data were recorded using quantum design make SQUID magnetometer (model

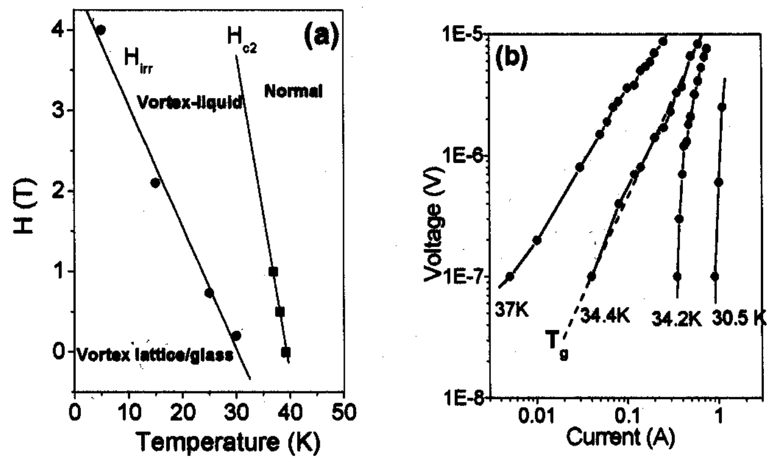

Figure 1. (**a**) H–T phase diagram showing irreversibility and upper critical field lines. (**b**) Current–voltage curves obtained at 0.3 T. Dashed line indicates power line behavior at vortex-glass transition temperature (T_g).

MPMS). X-ray photoelectron spectroscopic (XPS) measurements were carried out in RIBER system using Al K_α (1486.6 eV) radiation. The binding energy scale was calibrated to Au $4f_{7/2}$ line of 83.95 eV. The peak positions in the spectra were determined using the least-square fitting of data to Gaussian line shape.

3. Results and discussion

The four-probe resistivity measurements showed occurrence of superconducting transition (T_c) at 36 K and 39 K, respectively for the solid-state and liquid-phase sintered samples. The T_c was found to decrease with increasing applied magnetic field. From the field dependence of T_c, the slope dH_{c2}/dT was found to be -0.44 T/K. The value of $H_{c2}(0)$ determined using the WHH relation [3], $H_{c2}(0) = -0.69T_c dH_{c2}/dT$ is found to be ~ 12 T, which is comparable to that reported in literature [4]. The critical current density (J_c) of the MgB_2 grains determined from magnetization measurements was found to be $\sim 1 \times 10^5$ A/cm^2 (5 K, 1 T), which is among the best J_c values reported for MgB_2 superconductor. However, J_c is found to decrease sharply with magnetic field and tends to zero at fields much lower than H_{c2} indicating the existence of an irreversibility line. The H–T phase diagram showing the irreversibility and upper critical field lines are shown in figure 1a. At temperatures and fields below irreversibility line vortices are strongly pinned in the form of lattice or glass; while above this line vortices are weakly pinned or are in the liquid state. In order to investigate the vortex motion, the current–voltage (I–V) characteristics were measured under various magnetic fields. Typical I–V curves for temperatures from 37 K to 30.5 K under an applied magnetic field of 0.3 T are shown in figure 1b. These curves are similar to those reported for cuprate superconductors [5], and are indicative of vortex-glass transition behavior.

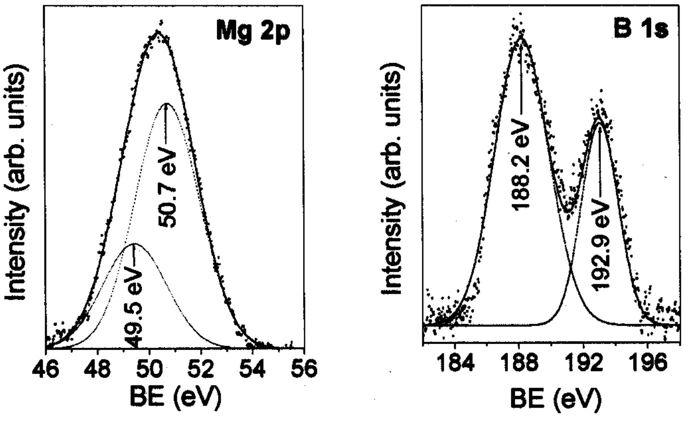

Figure 2. Core level Mg 2p and B 1s spectra recorded for the MgB_2 samples exposed to ambient atmosphere for several days.

To investigate the stability of MgB_2 under atmospheric exposures, the sample surface, exposed to atmospheric conditions for few days, was characterized by XPS. The core level Mg 2p and B 1s spectra are shown in figure 2. Mg 2p line could be deconvoluted into two peaks corresponding to binding energies of 49.5 and 50.7 eV, respectively indicating that Mg is in two different states. The lower Mg 2p binding energy is assigned to MgB_2; while the binding energy peak at 50.7 eV indicated presence of Mg in hydroxide and/or carbonate form [7]. The B 1s signal consists of two peaks occurring at binding energy values of 188.2 and 192.9 eV, respectively. The lower binding energy value is assigned to MgB_2 [6], and is consistent with BE values (187.2–188.5 eV) reported for transition metal diborides [7]. The existence of peak at 192.9 eV shows formation of B_2O_3 at the surface. The formation of oxides, hydroxides and carbonates were confirmed from O 1s and C 1s spectra. These results suggest that MgB_2 is a hygroscopic material and dissociates into $Mg(OH)_2$, $MgCO_3$ and B_2O_3 on exposure to moist air.

4. Conclusion

The MgB_2 superconductor has been found to exhibit an irreversibility line at much lower field and temperature values compared to the upper critical field line. The current–voltage characteristics show a vortex-glass like transition for MgB_2 superconductor. The surface of MgB_2 is found to be unstable under atmospheric exposures.

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