Available online at www.sciencedirect.com
SCIENCE DIRECT®
Physica C 415 (2004) 51–56
PHYSICA C
www.elsevier.com/locate/physc

Structural and low-field magnetic characterization of superconducting MgB_2 wires

A. Kılıç [a], S. Okur [b], N. Güçlü [c,*], U. Kölemen [c], O. Uzun [c], L. Özyüzer [b], A. Gencer [a]

[a] Faculty of Sciences, Department of Physics, Ankara University, 06100-Tandogan/Ankara, Turkey
[b] Izmir Institute of Technology, Department of Physics, 35437-Urla/Izmir, Turkey
[c] Faculty of Sciences and Art, Department of Physics, Gaziosmanpaşa University, 60100-Taşlıçftlik/Tokat, Turkey

Received 19 April 2004; received in revised form 7 July 2004; accepted 25 July 2004
Available online 3 September 2004

Abstract

Superconducting MgB_2 composite wires were prepared by packing blend of MgB_2 inside of Cu tubes using powder in tube (PIT) method. The produced samples of the wires were then characterised by using SEM, XRD and AC susceptibility measurements. The measured fundamental susceptibility is compared with Bean model. We have obtained an empirical functions for the penetration field $H_p = H_\alpha(1-t)^\beta$, where t is the reduced temperature. In addition, ac losses were calculated at the same fixed temperatures to compare theoretical solutions. There is a qualitative agreement between the experimental results and theory.

PACS: 74.25.Ha; 74.70.Ad; 74.25.Sv
Keywords: MgB_2 wire; AC susceptibilities; Bean Model

1. Introduction

The discovery of superconductivity in MgB_2 [1] has stimulated interest in determining its ac response in the superconducting state as well as in its novel properties in the normal state [2–6]. Recently, many researchers have spent much effort on the characterization and optimization of superconducting properties [7–11]. MgB_2, consists of a comprised interleaved two-dimensional boron and magnesium layer. The key element responsible for the superconductivity is considered to be boron. The compound has been attractive because the external field-dependences of the T_c and J_c appears to be encouraging. The recent reports are related with the fabrication of MgB_2 wires by PIT

* Corresponding author. Tel.: +90 356 2521582x3099; fax: +90 356 2521585.
E-mail address: guclu06@hotmail.com (N. Güçlü).

0921-4534/$ - see front matter
doi:10.1016/j.physc.2004.07.017

techniques [2] using either Ag or Cu sheath and MgB_2 strands [5] by directly filling Nb-lined, monel tubes with commercially available MgB_2 powders followed by drawing, rolling into tapes, and sintering [12]. The powder-in-tube (PIT) method appears to be the most practical and advantageous technique, since MgB_2 itself is mechanically hard and brittle to form a wire without a suitable, non-reactive sheath material. The cladding material provides magnetic screening to the applied magnetic fields on the superconducting core. In this method, the packing density of MgB_2 powder, which depends on the sheath materials, is very important to obtain a high critical current density J_c. In PIT technique, some metals such as stainless steel SS [13], Cu [2,14], Ag [2], Ag/SS [2], Fe [15], have been used as cladding material. However, among those, copper has been one of the most suitable sheath material for the fabrication of MgB_2 composite wires due to its low cost and high ductility.

For a superconductor, magnetic characterization is essentially to determine its magnetic properties. AC harmonic susceptibility ($\chi_n = \chi_n' + i\chi_n''$, with n is an integer number) has become a common tool as a method to understand the magnetic properties and to estimate the critical current density by use of suitable models. It is well known that χ_1'' is related to ac losses and χ_1' is associated with flux expulsion of the sample. The original interpretation of harmonic susceptibilities was first made by use of Bean's critical-state model [16]. In the model, J_c, independent on the local magnetic field, therefore a symmetric magnetization is assumed. Thus, odd-order harmonic susceptibilities are only generated. The out-of phase component χ_1'' is related to ac losses as $\chi'' = A_H/\mu_0\pi H_{ac}^2$, where A_H is the area embraced by the hysterics curve. Later, the Kim–Anderson model was developed [17]. In the model, it is assumed that J_c is dependent on the local magnetic field. This dependence leads to a non-symmetric magnetization and cause generation of even-order harmonic susceptibilities. In addition, a superimposed dc field gives rise to a non-symmetric magnetization.

In this paper, MgB_2 superconductor wires are characterized using SEM, XRD and AC susceptibility measurements. The temperature dependence of the magnetic field H_p has been determined to make comparison between the measured and calculated fundamental susceptibility. Best fit to experiments was found with the function $H_p = H_\alpha(1-t)^\beta$, where t is the reduced temperature. AC losses were also calculated at some fixed temperatures to compare with experimental results.

2. Experimental

Blend of MgB_2 powder (−325 mesh) was filled into a Cu tube with a wall thickness of 1 mm and outer diameter of 5 mm in air. Only hand pressing was applied to density the MgB_2 powder in Cu tube. The filled tube was cold drawn in a number of steps with about 5% of section reduction to a round Cu-clad MgB_2 wire with a core diameter of 0.7–0.8 mm and with an outer diameter of 1.5 mm, while the superconducting fill factor corresponds to about 25–30% for the whole conductor volume. The samples were then annealed in a tube furnace in a high purity Ar gas flow under ambient pressure at 800 °C for 3 min and quenched to air for short annealing process. Then the samples have been investigated in terms of SEM, XRD and AC susceptibility measurements. The susceptibility measurements were carried out by a commercial Lake Share 7130-model ac susceptometer employing a mutual inductance coil system with a closed cycle refrigerator.

3. Model calculations

The measurements show field-dependent behaviour, especially at temperatures close to T_c. The χ_1'' exhibited single maximum shifted to lower temperature on increasing applied field. These results show that Bean model is quite applicable to the qualitative description of the material. In this model, the critical current density, J_c, is assumed to be independent of local magnetic fields. The model also depends on the geometry of sample. Goldfarb et al. [18] derived the equations of ascending and descending portions of the magnetisation using the Bean model for cylindrical-geometry. In our calculations, we use their set of equations (29)–(38) in Ref. [18] to compute harmonic susceptibilities. Harmonic susceptibility ($\chi_n = \chi'_n + i\chi''_n$) is defined as

$$\chi'_n = \frac{1}{\pi H_{ac}} \int M(t)\sin(nwt)\,d(wt) \tag{1}$$

$$\chi''_n = \frac{1}{\pi H_{ac}} \int M(t)\cos(nwt)\,d(wt) \tag{2}$$

in-phase and out-of-phase components of harmonic susceptibilities, respectively.

4. Results and discussion

Fig. 1 shows SEM picture of transverse cross-section of a 1.5 mm in diameter of an annealed Cu-sheathed MgB_2. The diameter of the superconducting core was measured about 0.8 mm from SEM picture. The formation of $MgCu_2$ interface layer on the Cu sheath wall is not so visible from the SEM picture taken using back-scattered electron imaging.

The effect of cold working and annealing temperature and annealing time on the microstructural development of Cu-sheathed wire on the MgB_2 phase was analysed by X-ray diffraction measurements as shown in Fig. 2. The comparison of Fig. 2b and c shows Mg diffusion from pure MgB_2 superconducting core after annealing of Cu-clad MgB_2 wire at 800 °C for 3 min. As a result, X-ray diffraction pattern in Fig. 2c looks blur due to excess amorphous B in the core due to the loss of Mg during the formation of intermetallic $MgCu_2$ in the Cu sheath wall.

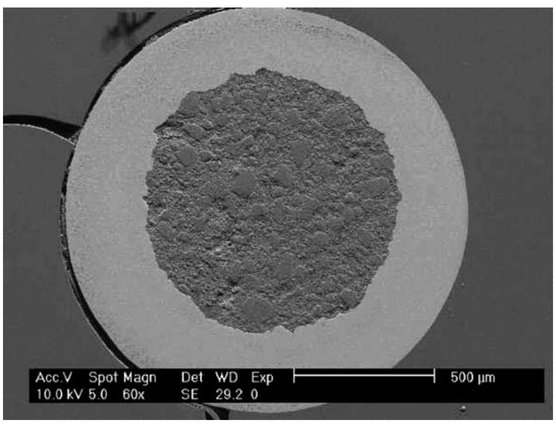

Fig. 1. SEM micrograph of cross-section of an annealed Cu-sheathed MgB_2 composite wire with 1.5 mm in diameter.

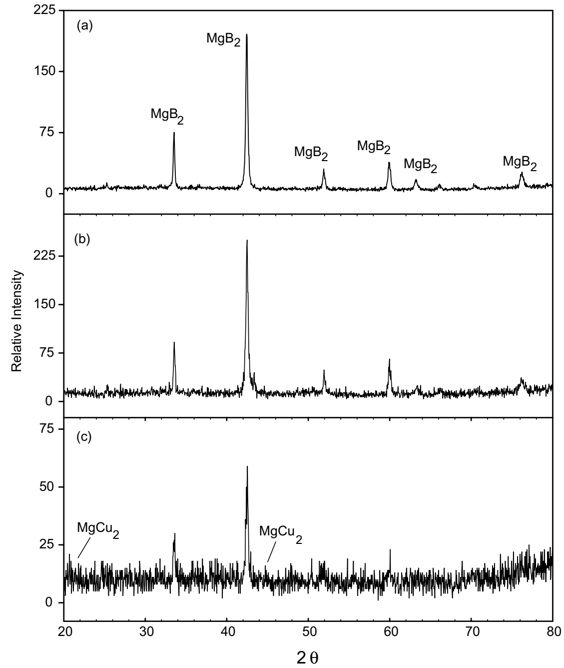

Fig. 2. X-ray diffraction patterns of the MgB_2 powders, (a) before packing, (b) after removing the Cu sheath mechanically after the cold drawing process to a wire with outer diameter of 1.5 mm, (c) after annealing at 800 °C for 3 min.

Fig. 3 exhibits the effects of ac field amplitude depence on the measured susceptibility of the MgB_2 composite wires. All the measurements reported are normalized at 25 K. When H_{ac} is increased from 20 (A/m) to 1600 (A/m), the peak height in χ''_1/χ_0 increased as the peak moved to lower temperatures. Fundamental susceptibility shows a typical single-step transition. This single-step transition, reflecting the flux penetration into the bulk of MgB_2, indicates the presence of a strong coupling between the grains of MgB_2. The peak temperature, T_p is 37.56, 37.40, 37.30, 37.17, 37.00, 36.78 and 36.80 K for ac fields of 20, 80, 160, 320, 640, 1280 and 1600 (A/m) respectively.

Fig. 4 displays H_p versus peak temperature, T_p. At the peak values of χ''_1/χ_0, ac field amplitudes are

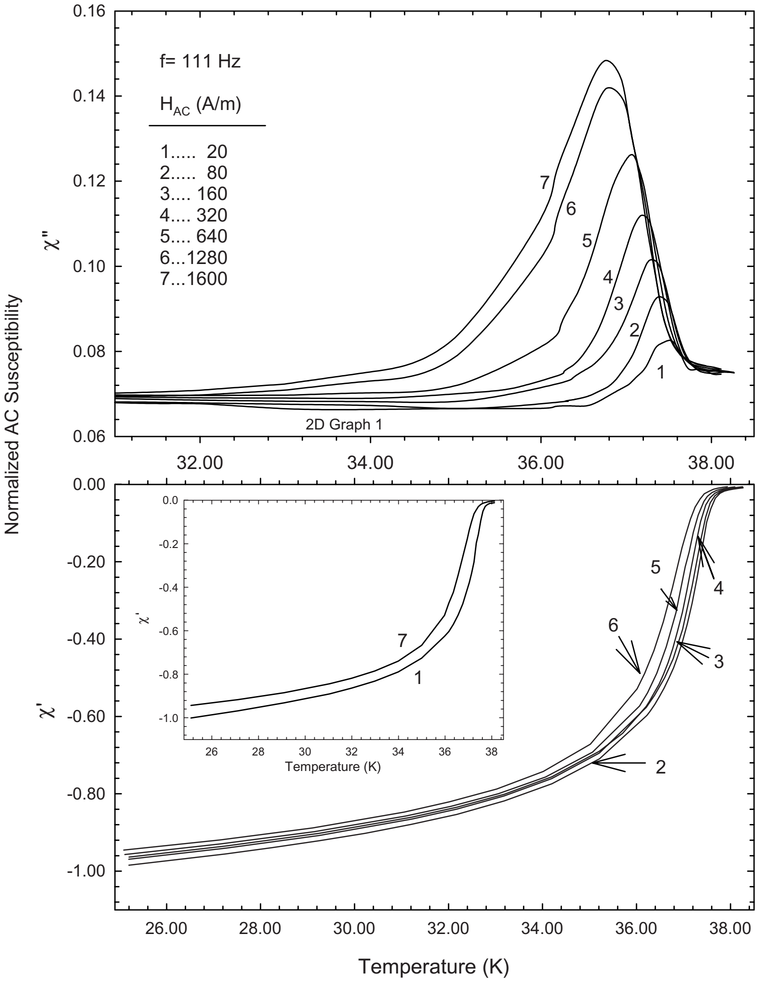

Fig. 3. Experimental measurements of fundamental harmonic susceptibilities are given for H_{ac} = 20, 80, 160, 320, 640, 1280, 1600 A/m (rms) and $H_{dc} = 0$, $f = 111$ Hz.

equal to the full penetration field the H_p. A function of the form $H_p = H_\alpha(1 - t)^\beta$ with parameters $H_\alpha = 26772651$ A/m and $\beta = 2.7$ fits best to the data observed. The lower temperature part of the curve was drawn by extrapolation. The value of 2.7 is compared with 1.9 for the BSCCO [19] and 2 for YBCO [20]. The results appear to support that J_c in the MgB_2 becomes less sensitive to magnetic field when compared to high temperature superconductors for example the BSCCO and the YBCO.

Fig. 5 shows the calculated fundamental susceptibility. The theoretical calculation was carried by using Bean model with the fitting equation $H_p = H_\alpha(1-t)^\beta$ for cylindrical-geometry. The results obtained from model calculations exhibit that, the peak temperature, T_p are 37.64, 37.52, 37.46, 37.36, 37.20, 37.05, 37.00 K for ac fields of 20, 80, 160, 320, 640, 1280, and 1600 (A/m) respectively. As H_{ac} increases, the peak moves to lower temperature, but, the height of the peak of imaginary part is not affected. As can be seen in Fig. 3 and Fig. 4, the experimental values were mimicked with the model calculations. They are also consistent with the works in Refs. [21,22].

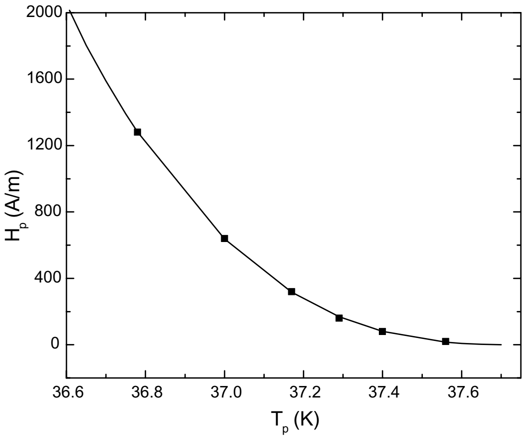

Fig. 4. The full penetration field, H_p versus peak temperature, T_p is shown.

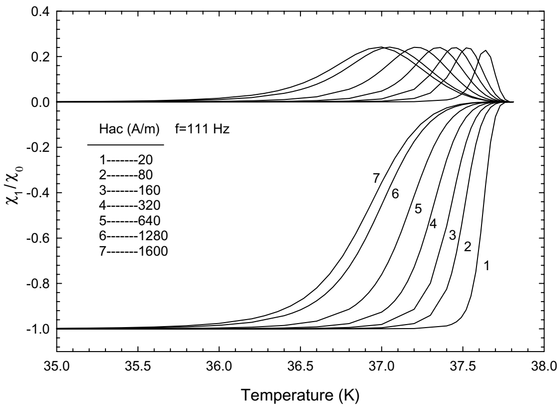

Fig. 5. Numerical solutions of fundamental harmonic susceptibilities to Eqs. (1) and (2) versus temperature are shown for H_{ac} = 20, 80, 160, 320, 640, 1280, 1600 A/m (rms) and H_{dc} = 0.

We have also plotted the calculated and experimentally measured ac losses versus ac field amplitude in Fig. 6 for three temperature (T = 37.4, 37.2 and 37.0 K). Open symbols and filled symbols show the experimental and theoretical calculations, respectively. It can be found from Fig. 4 that at low fields, the calculated and experimental values of the ac losses agree reasonably well, where ac losses essentially consist of the hysteretic intergranular coupling.

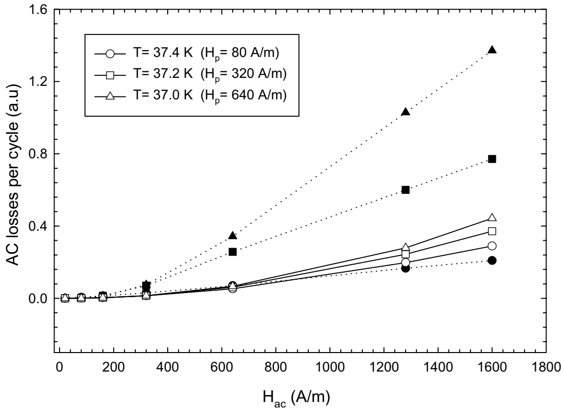

Fig. 6. The plot of experimental and theoretical ac losses versus H_{ac} for three different temperatures is given.

5. Conclusion

MgB_2 composite wires have been investigated by means of SEM, XRD and AC susceptibility measurements. The measured fundamental susceptibility has been calculated from Bean model with the fitting parameters. AC losses were also calculated at the same fixed temperatures. As a result, the model calculations are in qualitative agreement with the corresponding experimental measurements. In addition, suitable temperature dependence for the penetration field $H_p(T)$ is given.

Acknowledgment

We acknowledge that this research is supported by Turkish State Planning Organisation (DPT) under contract number DPT 2002-K-120130-5 and TUBİTAK (Scientific and Technical Research Council of Turkey) project number TBAG-2215.

References

[1] J. Nagamatsu, N. Nakagawa, T. Muranaka, Y. Zenitani, J. Akimitsu, Nature 410 (2001) 63.
[2] B.A. Glowacki et al., Supercond. Sci. Technol. 14 (2001) 193.
[3] C. Buzea, T. Yamashita, Supercond. Sci. Technol. 14 (2001) R115.
[4] C.B. Eom et al., Nature 410 (2001) 558.

[5] P.C. Canfied et al., Phys. Rev. Lett. 86 (2001) 2423.
[6] S.X. Dou et al., Physica C 361 (2001) 79.
[7] I. Kusevic et al., Solid State Commun. 122 (2002) 347.
[8] G.J. Xu et al., Physica C 399 (2003) 8.
[9] S.H. Zhou et al., Physica C 387 (2003) 321.
[10] O. Ozogul et al., Physica C 402 (2004) 209.
[11] T.A. Prikhna et al., Physica C 402 (2004) 223.
[12] M.D. Sumption et al., <cond-mat/0102441 (2001)>.
[13] H. Kumakura et al., Appl. Phys. Lett. 79 (2001) 2435.
[14] S. Soltanian et al., Supercond. Sci. Technol. 16 (2003) L4.
[15] H.L. Suo et al., Appl. Phys. Lett. 79 (2001) 3116.
[16] C.P. Bean, Rev. Mod. Phys. 36 (1964) 31.
[17] T. Ishida, R.B. Goldfarb, Phys. Rev. B 41 (1990) 8937.
[18] R.B. Goldfarb et al., in: R.A. Hein et al. (Eds.), Magnetic Susceptability of Superconductors and Other Spin Systems, Plenum Press, 1991, p. 64.
[19] J.L. Gonzales et al., Physica C 255 (1995) 76.
[20] K.H. Muller, Physica C 159 (1989) 717.
[21] M J Qin et al., Phys. Rev. B 64 (2001) 060505(R)-1.
[22] A. Gencer, Supercond. Sci. Technol. 15 (2001) 247.