Magnetic properties of MgB$_2$-Fe sandwiches produced by Field-Assisted-Sintering Technique

1G. Aldica, 1V. Sandu, 1C. Plapcianu, 1P. Badica, 2J. R. Groza

1National Institute of Materials Physics, 105 bis Atomistilor, Magurele, Ilfov, 077125, Romania
2Department of Chemical Engineering and Materials Science, University of California, One Shields Avenue, Davis, CA 95616-5294, U.S.A.

Abstract

Superconducting SiC and B$_4$C-doped MgB$_2$ samples containing up to three layers of pure Fe have been prepared by Field-Assisted-Sintering Technique (FAST). The adhesion between the Fe and doped-MgB$_2$ layers is excellent due to the enhanced local interdiffusion and increased solubility of Fe into MgB$_2$ grains that are typical for the non-equilibrium processes.

Magnetic measurements showed the largest irreversibility, hence, the largest critical current density in the B$_4$C-doped sandwich with one Fe foil. Additionally, the drawback of flux jumps at low temperatures is almost absent for one Fe-layer sandwiches. However, the irreversibility is smaller than in pure MgB$_2$ and SiC-doped MgB$_2$ samples prepared by the same technique, but without the insertion of Fe foil.

When the number of Fe foils is increased to three and an excess of 5 %wt. Mg is added, the SiC-doped sandwiches show almost similar irreversibility values and field dependence (at 5 K and at 20 K) as the pristine MgB$_2$ samples without Fe. But, the flux jump is recovered at the same level found for the pristine MgB$_2$ without Fe. An explanation in terms of doping dependence of the ratio between the thermal and magnetic diffusion constants is proposed.

1. Introduction

MgB$_2$ superconductor has been imposed as a promising material for low and intermediate temperature applications due to its attractive properties like large coherence length, hence, grain border transparency [1], acceptable upper critical field susceptible to enhancement by appropriate doping, high critical current density, and a satisfactory critical temperature $T_c$. The transparency of the grain boundaries, hence, the absence of the week links, makes this material a good candidate for fabrication of wires and tapes if proper sheaths are available. The sheaths are necessary because MgB$_2$ has both poor mechanical properties and Mg is volatile leading to microstructures with low density and bad grain connectivity. Several groups reported the fabrication MgB$_2$ of wires and tapes based on this material. [2-10]. Good results were achieved through the powder-in-tube (PIT) approach, either using pre-reacted MgB$_2$ (ex situ method) or by reacting the main components directly within the tube (in situ method). One of the important aspects in the PIT procedure is to avoid severe interaction between the material loading the tube and the tube walls during the thermal treatment because both Mg and B form intermetallic compounds or solid solutions with almost all metallic sheaths of interest [11]. On the other hand a good adhesion between MgB$_2$ and the sheath is highly desired for the improvement of the mechanical properties of the composite wires or tapes. In addition to chemical compatibility, a good sheath must have acceptable workability. Among different materials, iron seems to be a good choice. However, above 825°C, especially for the in situ method, Fe starts to react with B producing a FeB$_3$ interface [11, 12] which worsens both the workability and the critical current density. Higher processing temperatures and longer times are favourable to this reaction. The decrease of these two parameters [13, 14] would avoid the indicated problem and will also have a positive economic impact. An interesting un-conventional method is FAST, which combines application of a low uniaxial pressure with pulsed and/or dc electrical fields. The method
produces specific non-equilibrium effects resulting in significant decrease of the sintering time and temperature as well as in high densities for difficult to consolidate materials such as MgB$_2$. The method can be imagined for continuous FAST processing of PIT tapes or wires using a rolling machine on which pulsed and/or dc electrical field is applied to the rolls [15].

To investigate the possibilities of such idea, as a preliminary step, we have fabricated MgB$_2$-Fe sandwich bulk composites using commercially available FAST equipment and characterized them by magnetic measurements.

2. Experimental

Mixtures of commercially available MgB$_2$ and SiC or B$_4$C with molecular ratio 95/5 were intercalated with up to three Fe foils (ex situ method) and further placed into a graphite die. When three foils are inserted an extra amount of Mg powder was added. The whole arrangement was mounted for FAST processing into a “Dr. Sinter” system (Sumitomo Coal Mining Co), vacuumed down to 15 Pa and heated up to the desired temperature at a constant rate. Uniaxial pressure was applied perpendicular the iron foils surface. A pulsed current with the amplitude of 1000A and the on/off ratio of 12/2 was passed through the dye. The dwelling time and the temperature at which the pressure was released were settled so that a constant contraction of the sample should be obtained.

Microstructure of the samples was observed by scanning electron microscopy (SEM, JEOL JSM-6400F/EDAX). Magnetic characterization was performed with a MPMS (Quantum Design) magnetometer.

3. Results and discussions

Generally, all samples displays the same morphology of the doped MgB$_2$, prepared by the same method, as reported elsewhere [16] except a thin area around the interface between the two partner of the sandwich (see figure 1a). Therefore, as EDS data have shown, deep in each layer is present either MgB$_2$ (A in figure 1b) or iron (B) grains. Going closer to the interface, on the MgB$_2$ side of the interface, a textured layer of around 15 $\mu$m thickness is visible (C). It is made of larger Fe-diffused MgB$_2$ grains where the average ratio Mg/Fe $\approx$ 8.8. A possible liquid phase during the SPS processing would explain the growth of these large grains. On the Fe side, the grains are much smaller due most likely to the boron diffusion within Fe layer, a morphology that was also reported for the samples prepared by PIT method. However, the contact between the two layers is much better than reported in tapes made by conventional methods.

![Figure 1](image_url)

a) b)

Figure 1. SEM microographies of the MgB$_2$-Fe sandwich (sample with 1 Fe layer). a) $\times$200; b) $\times$1000 (A-MgB$_2$, B-Fe, C and D interfaces)
Figures 2 show the magnetization $M$ vs. field $H$ measurements at 5 (figure 2a) and 20 K (figure 2b), respectively. It is striking that for the sample doped with SiC and two Fe foils the flux jump is no more visible in increasing field. However, this sample shows also the lowest irreversibility (figure 3). Actually, the highest irreversibility in the doped samples is observed for SiC-doping with 3 layers of Fe. The reversible magnetization, $M_{\text{rev}}$ shows a curious dependence on the number of Fe layers. Though it is expected that Fe introduce a magnetic contribution to the total magnetization, the SiC-doped samples have almost zero reversible magnetization for three Fe foils and the maximum contribution for 2 Fe foils (figure 4). It is not clear the reason for this dependence unless we take into account that in the sample with 3 layers a small amount of Mg powder was introduced. It is possible that during the SPS process a certain amount of Mg should evaporate hence depleting some grains, hence, diminishing their superconducting properties, and hence exposing the Fe foils to the field. In the case of the sample with 3 Fe foils this depletion seems to be compensated by the Mg powder.

![Figure 2](image1.png)

**Figure 2.** Magnetization loops for the pure and sandwiched doped-MgB$_2$ a) $T = 5$K; b) $T = 20$ K.

![Figure 3](image2.png)

**Figure 3.** Field dependence of the irreversible magnetization of the doped-MgB$_2$-Fe layers.

![Figure 4](image3.png)

**Figure 4.** Field dependence of the reversible magnetization of the doped-MgB$_2$-Fe layers.
Concerning the absence of jumps in the sample with two Fe layers we consider it is connected to the crossover from adiabatic regime to dynamic regime due to the thermal and electrical conductivity of the Fe sheets. Why this is not true for the sample with 3 layers and which is the role of Mg is still unclear, but most likely can be attributed to the increased full penetration field beyond the condition of flux stability [17].

In conclusion, we investigated composite samples of MgB$_2$ superconductor intercalated with Fe foils obtained by FAST. The layered structure reduces the flux jump by improving the temperature diffusion time, but in the sample with Mg addition, this effect is hindered by the high magnetic penetration field. However, this latter sample has critical properties very close to the bare MgB$_2$ samples.

Acknowledgments
CP is grateful for Research Scholar support from UC Davis, USA. The authors acknowledge Prof. K Togano (NIMS, Japan), and Profs K Watanabe and S Awaji (Tohoku University, Japan) for support with SQUID measurements. This work was performed under CEEX 27/2005.

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