Flux jumps anomalous behaviour in FAST-processed MgB_2 composites

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Superconductor MgB_2 has a significant potential for practical applications, but undesirable flux jumps in magnetization curves at low temperature and at moderate or intermediate magnetic fields prevent its widespread use. In the present work, we analyzed the flux-jumps tendency in MgB_2 bulk doped with SiC, B_4C and metallic Mg. Samples were prepared by field-assisted-sintering (FAST) as composites with up to three intercalated Fe-foils. Samples produced by FAST showed quite different phase structures clearly influencing pinning and thermo-magnetic instability features due to inhomogeneous materials structure or due to dopants. The largest flux jumps are observed in samples with high critical current density, as usually observed in conventional MgB_2 bulks. However, jumps patterns are different and do not perfectly follow the jumps – critical current dependence, especially for the samples with Mg-addition.

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1. Introduction

For superconductor (SC) applications, high transition temperature T_c and high superconducting critical current density J_c are desirable. MgB_2 with a transition temperature of 39 K and high critical current density, J_c, is a promising candidate. However, MgB_2 exhibits a thermo-magnetic instability (TMI) with flux jumps (FJ) in magnetization curves. , [1-4]. Magnetic diffusion rate becomes faster than the thermal diffusion rate at low temperature, such that magnetic flux abruptly moves to cause flux jumps (FJ) and induces adiabatic critical state [5]. Some adiabatic models for flux jumps in bulk superconductor [5-7], thin superconductors [8] and in some metal-clad MgB_2 wires [9] were applied to explain the experimental data. There are some information mentioned in literature concerning the flux and temperature distributions in the adiabatic critical state after a jump and, there are also some indications about the threshold temperature above which no flux jumps occur [8]. Previous studies show that jump size depends on the applied field. There is a stable limit of field difference in the superconductor [7-9]. All these parameters are of high practical importance for MgB_2 superconductor in future high-field applications.

Field-assisted sintering technique (FAST) is known to consolidate different difficult-to-sinter powders (e.g. [10]). When using this technique, the sample is under a pulsed electric current/field during compressive pressure application. Although the physics involved is not completely understood, this method provides an excellent way to achieve high density MgB_2 [11, 12], while preventing the increase of grain size. Reduced grain size is a critical feature of superconductors with dominant grain boundary pinning given that the grain boundary depresses the critical current density. FAST also has the advantage of considerably shortening the process duration. FAST has several specific features such as creation of hot spots, enhanced (electro) diffusion and perhaps the controversial development of states close to plasma. These features can induce properties that can not be achieved by common processing techniques.

In this work, we have studied the flux jumps in superconducting SiC and B_4C-doped MgB_2 samples containing extra Mg or/and up to three layers of pure Fe sintered by FAST. Generally, metal-layers are introduced into MgB_2 intermetallic compound to improve thermal behaviour of the composites to suppress or remove the thermo-magnetic instabilities [9]. We note that addition of dopants such as SiC and/or B_4C, usually known to produce strong pinning centers in conventionally processed MgB_2 ceramic, may also influence thermo-magnetic instabilities. Presently it is not clear whether the instabilities are due only to high pinning features, and, hence to high J_c-values as currently thought [13] or dopants play a different role. To answer this dilemma, samples with different impurities/dopants and produced by unconventional technologies are highly recommended because they can trigger significantly different chemistries, thus imparting special pinning properties. This aspect justifies the selection of FAST technique for samples preparation. The approach is also a new and promising way to produce Fe-MgB_2 composites in the future, including superconducting tapes.

2. Experimental

Polycrystalline samples of MgB_2 with additions of SiC (Merck, 45 nm) or B_4C (HC Starck Grade HS, 0.8 μm) were prepared from commercially available powders of MgB_2 (Alpha Aesar, 2.3 μm). In some samples, metallic Mg powder was introduced. The molecular ratio between MgB_2 and additives was 95/5 or 90/5/5. The reagents were mixed for 1800 s in an agate mortar under argon atmosphere in a glove box. In the samples we intercalated. One or more Fe foils (discs) (Goodfellow Metals Inc. thickness 0.1 mm) were intercalated to form a sandwich structure of alternate layers of Fe and MgB_2.

The mixture of powders with Fe foil(s) was placed into a graphite die that allows a good electrical contact. The die with powder-foils sandwich was mounted in a 'Dr Sinter' spark-plasma-sintering system (Sumitomo Coal Mining Co) – SPS 1050, vacuumed down to 15 Pa. The surface of the Fe-foil(s) was perpendicular to the applied uniaxial pressure. The samples contained one (1), two (2) or three (3) Fe-discs of the same diameter as the sample. The samples were heated up to the desired temperature at a constant rate while applying a constant pressure (Table 1). The heating was realised by a pulsed current of an average value increasing in time to almost 1000 A and having an on/off time ratio of the pulses of about 12/2. After dwelling for 120-240 s, specifically until the rate of shrinkage became zero, the sample was cooled down, first, to 873 K when the pressure was released and then the cooling went on until room temperature. The procedure was repeated several times with different parameters until a constant contraction of the sample was reached. Samples were characterized for density, micro structural and electromagnetic properties. Bulk density on the MgB_2 bulk specimens and parts of the composite after removal of the Fe-foils was measured by Archimedes method. The crystal phases of the powder samples were determined by x-ray diffraction (XRD) using Cu Kα radiation (Scintag) and a scanning step of 0.02°.

Table 1. Processing parameters and some characteristics of the samples.

Sample	Composition	T_{max} (K)	Heating rate/ pressure/ No. of Fe-foils (K/s)/($\cdot 10^6$ Pa)/ foils)	T_{ic}* (K)	Density (of the MgB_2 part from the composite) ($10^3 \cdot$kg/m^3)	T_c^{offset} (offset where M(T) = const.) (K)
1	Fe-$(MgB_2)_{0.95}(B_4C)_{0.05}$	1283	2.8/70/ 1	1073	2.57	25.3
2	Fe-$(MgB_2)_{0.95}(SiC)_{0.05}$	1283	2.8/70/ 2	1203	2.59	25.2
3	Fe-$(MgB_2)_{0.9}(SiC)_{0.05}(Mg)_{0.05}$	1233	3.1/63/ 3	1183	2.50	33.3

*T_{ic} is the temperature at which the compact starts increasing its density.

Microstructural measurements were performed with a JEOL JSM-6400F microscope equipped with EDAX system. The magnetic properties, specifically the field H and temperature T dependence of the magnetization M, were evaluated with an MPMS dc SQUID magnetometer (Quantum Design) in the range 0-5 T and 1.8-46 K.

3. Results and discussion

The best samples have a density (only MgB_2 from the composite specimens) of 98% theoretical value (e.g. samples 1 and 2, Table 1). This is a positive and promising result and proves that FAST technique is suitable for MgB_2 composite tapes fabrication. Furthermore, all the samples are of relatively large size, with a diameter of 19 mm. We note that synthesis by conventional methods of large size MgB_2-bulk, or in our case of layered sandwich MgB_2-based composite, is not a trivial task. The temperature values of the shrinkage initiation, T_{ic}, are between 1073 K and 1233 K, and indicate quite a different sintering behaviour. The different T_{ic} values are probably due to changes of the rheological properties at high temperature of the MgB_2 powder with additions (see Fig. 1).

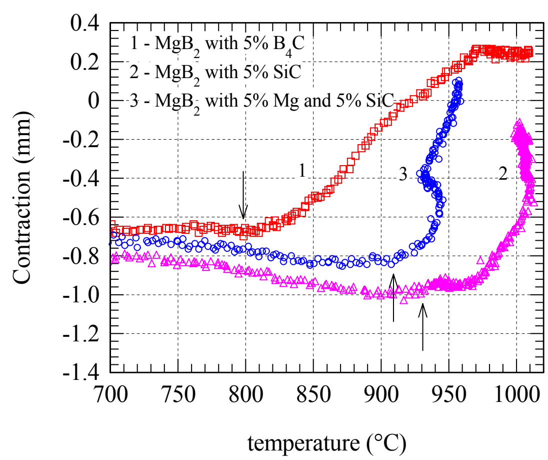

Fig. 1 Contraction vs. FAST-temperature for 1-3 MgB_2 samples from Table. Arrows indicate the temperature of the shrinkage initialization T_{ic}.

Different thermo-rheological behaviour of the MgB_2-based mixtures is expected to influence not only the necessary time and the maximum achievable density, but also phase purity, crystal quality and superconducting properties of the final composite product

Fig. 2 displays the phase's composition as revealed by the XRD investigations. All samples show impurity peaks of MgB_4 and MgO. The amounts seem to be different among the samples with a higher amount of both impurities being apparently detected for sample 1.

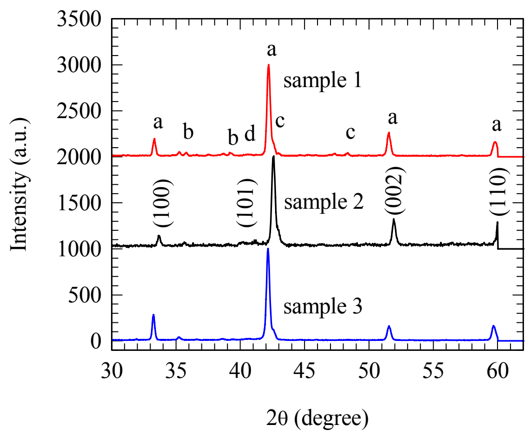

Fig. 2. X-ray diffraction patterns of FAST-processed Fe-MgB_2 composites. The letters correspond to the following phases: a-MgB_2, b-MgB_4, c-MgO, and d-Mg_2Si.

From M-T curves measured at 0.002 T in zero-field-cooling arrangement, ZFC, we extracted critical temperatures. The onset critical temperature, T_c^{onset} is the same for all samples, i.e., 38.8 K. On the other hand T_c^{offset} values (Table 1) show significant differences. Samples 1 and 2 show lower T_c^{offset} temperature. These samples are without Mg additions and are FAST-treated at a higher maximum temperature than the sample 3, so that the evaporation of Mg was probably stronger in their case. This result suggests that some grains in samples 1 and 2 are Mg-poor leading to lower T_c^{offset} values. In addition, sample 2 shows residual Mg_2Si and the diffraction lines of MgB_2 are shifted to higher 2θ values. This aspect can be explained by SiC decomposition and B substitution with C in MgB_2 [14]. For the conventionally processed samples B substitution with C, usually reduces T_c onset value. This is not the case for our FAST samples and the reasons are probably related to specific features of the FAST processes and/or to the uniformity of the substitution: relatively clean MgB_2 grains with high T_c onset are still available in the material. Bigger quantity of the MgB_4 phase was detected in sample 2 and somehow in sample 1, i.e. for samples without Mg-addition. In sample 3, the presence of Mg-addition suppressed the formation of the MgB_4 phase and of the Mg-deficient MgB_2 grains with lower T_c^{offset}, as already mentioned above. The amount of MgO impurity phase seems constant for all samples, or it is slightly higher for sample 2.

Magnetisation measurements at 5 K are presented in Fig. 3. All curves show the characteristic flux jumps. These phenomena may be explained as follows. The vortex avalanche process depends on the relation between thermal and magnetic diffusivities. It is thought that the stability of flux jumps in MgB_2 may be analyzed within the framework of the adiabatic approximation [5]. As pointed out by Chabanenko et al. [5], the magnetic diffusivity is much larger than the thermal diffusivity in MgB_2 samples, because the heat capacity becomes small below about 10 K at low magnetic field [15, 16].

It is rather surprising that for the sample doped with SiC and two Fe foils (sample 2) the flux jump is no more visible while increasing the magnetic field. However, this sample shows also the lowest irreversibility. Actually, the highest irreversibility occurs in the samples with 3 layers of Fe, sample 3 [17].

The absence of jumps in the first quadrant of M-H loops, in the sample with two Fe layers, sample 2, may be related with a crossover from adiabatic regime to dynamic regime due to the thermal and electrical conductivity of the Fe sheets. It is still unclear why this is not true for the sample with 3 layers (sample 3) and what the role of Mg is, but most likely can be assigned to the increased full penetration field beyond the condition of flux stability [18]. It is useful to use the following ratio [5] to characterize the flux stability:

$$\alpha = \frac{\text{critical current density after the flux jump}}{\text{critical current density before the flux jump}} \quad (1)$$

For α = 0 the critical current density goes to zero at $\mu_0 H$ = 1T and the flux jump is complete, as it is the case of sample 3 in the first and second quadrates of M(H) curves. For sample 1, α = 0.145 at μ_0H = 1.65 T. Similar experimental values were obtained by Romero-Salazar et al. [7] and were sustained by their numerical calculations. For sample 2, α = 0.15 at μ_0H = 0.25 T in the second quadrate of M(H) graph and the flux jump is incomplete as for sample 1.

As pointed out by Murai et al. [13], flux jumps occur more frequently in samples with higher J_c as the magnetic hysteresis indicate in Fig. 3 for sample 3.

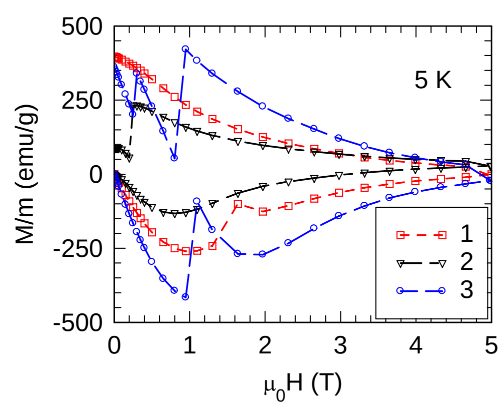

Fig. 3 The magnetization curves on Fe-MgB_2 composites at 5 K.

4. Conclusion

We investigated the anomalous behaviour of the flux jumps in 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 by thermal transport. It is remarkable that, while samples produced by FAST show quite different phases composition clearly influencing pinning feature, the largest flux jumps are observed for samples with high critical current density. However, Mg addition, SiC and B_4C doping and Fe-foils interlayer(s) produced asymmetric flux-jumps patterns on the magnetic-field increase and decrease curves of the M-H loops that are not following the relationship (1) between the flux-jumps and J_c.

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