Flux pinning and microstructure of a bulk MgB₂ doped with diverse additives

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Abstract

In bulk high-temperature superconductors, Ag is usually used as an additive thus improving mechanical performance. In MgB₂ doped with Ag, the Ag reacts with Mg, forming Mg–Ag phases acting as a vortex pinning medium. In this work, we analyze the electromagnetic and pinning properties of bulk MgB₂ doped with 1 wt% MgB₄, 4 wt% Ag, and 1 wt% Dy₂O₃, prepared at the Shibaura Institute of Technology (SIT), Tokyo. In three compounds of MgₓB₂ + 4 wt% Ag with x = 1, 1.075, and 1.1, the effect of Mg excess was studied. The magnetic moment was measured by a vibrating sample magnetometer (VSM). Pinning was studied in terms of a pinning diagram, i.e. the field dependence of the normalized pinning force density, \( F_n = \frac{F}{F_{\text{max}}} \). In all studied samples, the peak of the \( F_n(b) \) dependence (\( b = \frac{B}{B_{\text{irr}}} \), \( B_{\text{irr}} \) being the irreversibility field) was observed at around \( b = 0.2 \), indicating a prevailing flux pinning at grain boundaries. A slight shift of the peak with decreasing temperature indicated a defect size distribution in the pinning landscape. Transmission electron microscopy (TEM) showed a granular structure of all samples composed of MgB₂ grains of about 230 nm (average size), with ensembles of small grains (22 nm in average) of Ag₃Mg, AgMg, Ag, Dy₂O₃, and MgB₄. While the large MgB₂ grains control the main pinning mechanism, the small precipitates seem to determine details of the current flow through the grain boundaries.

Keywords: MgB₂, MgB₄, Ag-doping, flux pinning, microstructure analysis, critical currents, pinning force density

(Some figures may appear in colour only in the online journal)

1. Introduction

High-\( T_c \) superconductors represent one of the most promising materials as regards a close horizon for many unique applications. For example, bulk superconducting magnets were proven to trap magnetic fields up to 17 T [1, 2], an order of magnitude above the best present ferromagnets. One of the problems hindering a massive utilization of high-\( T_c \) superconductors (cuprates) in practice is their price. The middle-\( T_c \) materials, like MgB₂, might be a good compromise. Their chemical composition and technology of preparation are relatively simple; moreover, no rare earth, noble, or embanked elements are needed. In contrast to granular cuprates, MgB₂ does not suffer from weak-link problems at grain boundaries. However, the technology of the relatively cheap MgB₂ requires optimization of the process and an appropriate choice of dopant(s). Wide varieties of dopants and processes to improve MgB₂ synthesis and enhance electromagnetic properties have been tested. For example, Ag [3, 4] and Cu [4–6], were utilized to improve MgB₂ sintering kinetics and mechanical properties. Ag is frequently used in rather brittle high-\( T_c \) superconductors to mechanically reinforce them, while not significantly deteriorating the \( T_c \) and helping in flux pinning [7]. While in high-\( T_c \) superconductors Ag mainly plays the mechanical role [8, 9], in MgB₂ it reacts with Mg, forming Mg–Ag precipitates that might serve as an additional pinning medium [10].
To optimize the sintering performance, it was found that use of carbon-encapsulated boron in MgB$_2$ synthesis significantly improved superconducting properties of the final product [11], due to a regular distribution of carbon over the material. The previously studied doping with SiC [12, 13] or C$_6$H$_4$N$_2$ [14] probably had a similar effect. Further alternatives for doping MgB$_2$ were, e.g. ZrB$_2$ [15], Zr [16], MgB$_4$ [17], various rare earths, and others.

In the present work, we focus on the role of Ag in vortex pinning enhancement, studying Ag–Mg alloying on the dependence of the Mg excess, and compare it with the effect of small additions of MgB$_4$ and Dy$_2$O$_3$.

2. Experimental details

MgB$_2$ was prepared using an in-situ solid-state reaction. High-purity commercial powders (Furu-uchi Chemical Corporation, Japan) of Mg metal (99.9% purity, 200 meshes) and amorphous boron (99% purity, 300 meshes) were mixed in a nominal atomic ratio Mg:B = 1:2. The mixed powder was pressed into pellets of 20 mm in diameter and 7 mm thickness using a uniaxial press. The consolidated pellets were then wrapped in tantalum foil and sintered in a tube furnace at 1050 °C for 2 h in Ar atmosphere. To remove MgO, the pellets were thoroughly ground into powder again and immersed in a bath of HNO$_3$ (1 M) solution for 60 min. The powder was then cleaned with ethanol to wash out the acid, and finally with distilled water to remove the remaining acid and ethanol. In the same way, MgB$_4$ powder was prepared [16]. Next, 1 wt% of MgB$_4$ was added to MgB$_2$ powder and thoroughly mixed in a glove box in Ar atmosphere and then pressed into pellets 20 mm in diameter and 7 mm in thickness using a uniaxial press. The consolidated pellets were then wrapped in tantalum foil and subjected to the heat treatment in Ar atmosphere in a tube furnace. The samples were heated to the sintering temperature of 775 °C and kept there for 3 h in flowing Ar gas. Finally, the temperature was lowered to room temperature at a cooling rate of 100 °C h$^{-1}$. The sample with Dy$_2$O$_3$ was prepared in the same way, only MgB$_4$ was replaced with the same amount (1 wt%) of Dy$_2$O$_3$. Alternatively, MgB$_2$ was prepared as described before, only the amorphous boron powder was encapsulated by 1.5 wt% carbon [11]. Based on this MgB$_2$ precursor, three samples were prepared, all added with 4 wt% of high-purity metallic Ag powder. It is known that Ag interacts with Mg forming AgMg and Ag$_3$Mg. To understand the compound balance with respect to the potential Mg loss in the MgB$_2$ system, excessive Mg was added in two of these three samples, in amounts of 7.5 at% and 10 at%. For comparison, one of the samples was left with the nominal amount of Mg. As before, to avoid oxide formation, powder mixtures were rigorously ground in a glove box in Ar atmosphere and then pressed into pellets 20 mm in diameter, 7 mm thick using a uniaxial hydraulic press. These pellets were immediately wrapped in tantalum foil and heat treated similarly to the other MgB$_2$ samples.

A vibrating sample magnetometer (VSM) installed in the Physical Property Measuring System (PPMS) equipped with a 9 Tesla magnet was used for inductive magnetic measurements. Samples for the measurements were of dimensions about 1.5 × 1.5 × 0.5 mm$^3$. The tests were done at 7, 10, 15, 20, 25, 30, and 35 K. The electromagnetic properties were analyzed in terms of the critical current density, $J_c$, induced in the sample by a change in the applied magnetic field. $J_c$ was deduced from a magnetic hysteresis loop (MHL) via the extended Bean formula for rectangular samples [18],

$$J_c = \frac{2\Delta m}{a^2bc(1-a/3b)}$$

where $\Delta m$ is the difference in the magnetic moments measured on the descending and ascending field branches of the MHL, respectively, $a$ and $b$, $a \leq b$, are the sample dimensions transversal to the magnetic field direction, $c$ is the sample thickness, and $J_c$ is the critical current density. For $\Delta m$ given in emu units (1 emu = $10^{-3}$ Am$^2$) and $a$, $b$, and $c$ in mm, $J_c$ comes out in $10^5$ A cm$^{-2}$. The $J_c$ values are presented in section 3.

Transmission electron microscopy (TEM) observation was performed on an FEI Tecnai G2 F20 X TWIN microscope at 200 kV. Samples were prepared by a focused ion beam (FIB) lift-out technique, using an FEI Quanta 3D FEG scanning electron microscope (SEM).

3. Experimental results

3.1. $J_c$ and $B_{irr}$

For simplicity of magnetic field units, we present all magnetic fields in terms of magnetic induction, $B = \mu_0 H$, and omit the difference in magnitude between the internal and external magnetic field, even at low fields. The pinning properties of the samples are here interpreted in terms of critical current density, $J_c$, irreversibility field, $B_{irr}$, and pinning force density, $F = J_c B$. Classical phenomenological models [19, 20] associate the prevailing vortex pinning interaction with the position of the maximum on the $F(I/B)$ plot, where $F_n$ is $F/F_{max}$, and $B = B/B_{irr}$ [21].

Critical current density as a function of the magnetic field approaches zero exponentially (figure 1). Therefore, the irreversibility field, $B_{irr}$, needs to be determined using a precision criterion. In our case, we chose this criterion as $J_c \approx 10$ A cm$^{-2}$ (the bottom line of figure 1). Beyond this value, the $J_c(B)$ curves got strongly noisy. At 10 K and 7 K, all the samples except for MgB$_2$ + 4 wt% Ag exhibited flux jumps at low magnetic fields, observed commonly in MgB$_2$ samples [9, 22–24]. This phenomenon is a magneto-thermal effect, associated with a reduction in specific heat at low temperatures [24] and local overheating by a fast flux motion. The presence of Ag, a good thermal conductor, had no effect, most probably due to its small concentration and dispersion in the material. Due to these flux instabilities, the self-field $J_c$ could be compared only above 10 K. The $J_c$ and $B_{irr}$ of the studied samples at 15 K are summarized in table 1.

We see that in the samples doped with Ag, both $J_c$ and $B_{irr}$ grew with increasing Mg excess. It indicates that more Mg–Ag...
Figure 1. Critical current densities, $J_c$, of the investigated samples as a function of the applied magnetic field. $B_{irr}$ was determined at $J_c = 10 \text{ A cm}^{-2}$ (bottom line of the figures). The missing data (20 K, 30 K) were replaced by extrapolation.

Figure 2. Comparison of the normalized pinning force density, $F_n/F_{max}$, of the investigated samples as a function of magnetic field induction normalized to $B_{irr}$, at 35 K.

Table 1. $J_c (B = 0)$ and $B_{irr}$ at $J_c = 4 \text{ A cm}^{-2}$ at 15 K.

| Sample                  | $J_c (B = 0) \times 10^3 \text{ A cm}^{-2}$ | $B_{irr}$ T |
|-------------------------|--------------------------------------------|-------------|
| MgB$_2$ + 1 wt% MgB$_4$ | 341                                        | 6.1         |
| MgB$_2$ + 4 wt% Ag      | 288                                        | 7.0         |
| Mg$_{1.05}$B$_2$ + 4 wt% Ag | 318                                 | 8.5         |
| Mg$_{1.0L}$B$_2$ + 4 wt% Ag | 502                                  | 9.5         |
| MgB$_2$ + 1 wt% Dy$_2$O$_3$ | 434                                  | 8.8         |

phases were created, contributing to flux pinning. This is one of the points that will be dealt with in the discussion, together with microstructure analysis. The other samples, doped with MgB$_4$ and Dy$_2$O$_3$, did not contain Ag but also exhibited a remarkable electromagnetic performance. This implies that the effect of various precipitates depends on their type (here all are normal), average size, and amount.

3.2. Pinning force density

Classical pinning diagrams, developed for conventional superconductors with very low magnetic relaxation [25, 26], are still used for data interpretation in high- and middle-$T_c$ superconductors, only with $B$ normalized to $B_{irr}$ (a quantity controlled by relaxation) instead of the thermodynamic quantity of $B_{c2}$. This somewhat deforms the original scheme, however, for a comparative study the procedure is useful. Figure 2 compares $F_n (b)$ plots of the five investigated samples at the same temperature, $T = 35$ K. Although $J_c$ and $B_{irr}$ values differ (figure 1.), the normalized $F_n (b)$ dependences resemble each other, all exhibiting peaks slightly below $b = 0.2$. Only the curve width slightly varies, implying some differences in the pinning landscape details. Figure 2 manifests that the pinning mechanism is in principle the same in all studied samples. Figure 3 shows how the pinning diagram varies with temperature. With decreasing temperature, the $F_n (b)$ curves of all the samples shift to lower magnetic fields and become narrower. It implies that the pinning defect ensemble is not uniform, but consists of a defect type and size distribution with the pinning optimum shifting with temperature. The position of the $F_n (b)$ curves close to $b = 0.2$ in all samples, irrespective of the dopant type, indicates that the principal pinning mechanism is pinning by grain boundaries [19–21]. Why $J_c$ and $B_{irr}$ differ with, in
principle, the same pinning mechanism, should be elucidated by the microstructure study (section 3.4).

3.3. Relaxation

The prevailing activator of relaxation in superconductors is thermal excitation. In high-$T_c$ superconductors, the operating temperature is usually high and thus thermal excitation, too. It, however, is not the only reason for ‘giant’ relaxation [26] in high-$T_c$ superconductors. Coherence length and the related vortex core diameter are much smaller than in conventional superconductors, which implies a much smaller pinning defect size. On average, much less energy is needed to release a pinned vortex from its trap than in conventional superconductors. The amount of relaxation is usually quantified by the conventional normalized relaxation rate, the logarithmic dependence of the magnetic moment on time, $S = -\frac{d\ln m}{d\ln t}$. Alternatively, we can utilize the fact that the MHL is a dynamic object, the size of which depends on the magnetic field sweep rate, and quantify relaxation in terms of the dynamic relaxation rate [27] as

$$Q = \frac{d\ln J_c}{d\ln \left(\frac{dB}{dt}\right)} \approx \frac{\Delta \ln J_c}{\Delta \ln \left(\frac{dB}{dt}\right)}.$$  (2)

For two different magnetic field sweep rates, we got two different MHL heights and thus two different $J_c$ values (figure 4). As the magnetic field sweep rate is constant during each individual MHL measurement, the denominator in equation (2) is constant and $Q$ is proportional to the difference in the logarithms of $J_c$ measured with different field sweep rates $\frac{dB}{dt}$, here $0.72 \text{ T min}^{-1}$ and $0.36 \text{ T min}^{-1}$. The result, typical for the samples investigated here and for MgB$_2$ at all [28], is shown in figure 5. It demonstrates that the $Q(B)$ dependence is nearly exponential over a major part of the investigated field range, with the $\ln Q/B$ slope rising with increasing temperature. At low magnetic fields, $Q$ falls far below the level commonly observed in cuprates (1%–4%), indicating that the thermally activated magnetic relaxation is rather weak in MgB$_2$, similar to conventional metallic superconductors.

3.4. Microstructure

The preceding sections proved that even a tiny doping of MgB$_2$ significantly changes its magnetic properties. Reasons for this effect should be seen on the material microstructure. TEM analysis made on lamellas prepared by the FIB technique
clearly showed the details of the microstructure. Figure 6 shows the TEM/EDS image of MgB$_2$ + 1 wt% MgB$_4$. A few hundred nm-sized red (grey in the black and white (BW) version) rather large grains of MgB$_2$ are seen, with much smaller blue (white in the BW version) spots of MgB$_4$ phase located at the boundaries of MgB$_2$ grains. The details are given in table 2. The special care paid to elimination of MgO in this sample was evidently successful. According to table 2, the amount of the oxide is negligible. In figure 6 it is not seen at all. The same was observed in the sample doped with Dy$_2$O$_3$. MgB$_2$ of the samples with Ag was prepared in another way, without special measures to eliminate MgO; therefore, it appears in larger amounts (table 2).

Analyses of the grain size distribution of MgB$_2$ and MgB$_4$ grains from figure 6 are presented in figures 7 and 8, respectively. The average grain size of MgB$_4$ grains (22 nm) is about one order of magnitude smaller than that of the MgB$_2$ grains (228 nm). The MgB$_2$ grain size only slightly varied in the studied samples. Figure 9 shows the microstructure of three MgB$_2$ samples with Ag addition and different amounts of Mg. The character was always the same; small particles of secondary phases always segregated at the MgB$_2$ grain boundaries. The main secondary phase, formed during processing, was AgMg and its concentration grew with Mg excess, while its average grain size fluctuated around a nearly constant value. In figure 9 AgMg is colored green. Another secondary phase, Ag$_3$ Mg, and MgO appeared in nearly the same but much a smaller amount in all three samples with Ag. The average grain size of both these compounds was about 20 nm. In figure 9, Ag$_3$ Mg is presented in light blue and MgO in dark blue (various grey shades in the BW version). The percentage appearances of different compounds formed during the sample processing, together with the average grain size of each, are collected in table 2. Two spots at each sample were always analyzed. Table 2 shows that the relative portion of MgB$_2$ slightly decreased with increasing excess of Mg (from about 85% to 73%), while the amount of spare Ag stayed nearly constant. Both the spare Ag and the secondary phases AgMg, Ag$_3$ Mg, and MgO segregated at MgB$_2$ grain boundaries. Their grain sizes were typically by order of magnitude smaller than those of the MgB$_2$ grains. While the size of the precipitates was nearly the same and did not change much with Mg excess, the concentration of AgMg slightly increased. It is particularly apparent when comparing the properties of each spot separately. It is now a question of to which extent the Ag–Mg reaction was responsible for the grain size changes, or if the effect could not be due to a slight departure of the sintering conditions from optimum.
Table 2. The percentage and the average grain size of different phases in four MgB$_2$ samples, each analyzed in two spots.

|      | Ag [%] nm$^{-1}$ | AgMg [%] nm$^{-1}$ | Ag$_3$ Mg [%] nm$^{-1}$ | MgO [%] nm$^{-1}$ | MgB$_2$ [%] nm$^{-1}$ | MgB$_4$ [%] nm$^{-1}$ |
|------|-----------------|-------------------|------------------------|------------------|-----------------------|----------------------|
| Mg$_1$B$_2$ + 4%Ag | 7.8/17          | 6.1/17            | 3.2/18                 | 1.9/20           | 80.9/71               | 0                    |
| Mg$_{1.07}$B$_2$ + 4%Ag | 4.5/22          | 1.4/26            | 1.3/18                 | 2.2/39           | 90.6/122              | 0                    |
| Mg$_{1.1}$B$_2$ + 4%Ag | 9.2/17          | 10.5/21           | 2.8/25                 | 0.8/14           | 76.8/133              | 0                    |
| MgB$_2$ + MgB$_4$ | 5.1/21          | 5.5/21            | 1.6/38                 | 1/46             | 86.8/152              | 0                    |
|      | 5.7/17          | 19.3/12           | 1.5/25                 | 0.9/12           | 72.6/215              | 0                    |
|      | 9.9/18          | 5.4/24            | 4.9/22                 | 6.3/12           | 73.5/126              | 0                    |
|      | 0               | 0                 | 0                      | 0.3              | 90.6/228              | 9.1/22               |

Figure 8. Grain size distribution of the MgB$_4$ phase from figure 6.

4. Discussion

MgB$_2$ is naturally a granular material. A big advantage of pure MgB$_2$ is that its grain boundaries are not barriers for critical current flow [25], in contrast to cuprates [29, 30]. Thus, the difference between the two types of currents induced in a granular superconductor smears and, with respect to critical current flow, the material behaves like a single crystal [31] or a single-grain bulk [32]. The main difference between MgB$_2$ and the single-crystal and single-grain bulk cuprates lies in the distribution of nanoscopic defects. While in MgB$_2$, they are segregated at grain boundaries, in single-grain cuprates they are nearly regularly distributed throughout the entire bulk [33]. The smeared difference between intra- and inter-grain currents in the MgB$_2$ samples is supported by the fact that their low-temperature MHLs do not exhibit anomalies observed in BiSrCaCuO tapes or in artificially granular superconductors at low temperatures [31, 32], like the central peak shifted to positive applied fields or the enhanced magnetic moment of the descending field branch of the MHL. From a microscopic point of view, MgB$_2$ is composed of superconducting grains. Boundaries of these grains, occupied by nanoscopic (point-like) normal grains of dopants and secondary phases, behave like a network of large pinning defects, controlling pinning of the flux lattice in the sample. TEM brought evidence of the formation of various secondary phases at a nearly unchanging system of MgB$_2$ grain boundaries. The small secondary phase grains are normal. The average size of all these precipitates, around 20 nm, is close to 2$\xi$, where $\xi$ is the vortex core diameter, the size thus being ideal for core pinning. Here, the core pinning seems to enhance the main mechanism of grain boundary pinning. The pinning core is reflected in $J_c$ and $B_{irr}$ but not in the $F_n(b)$ peak position, governed by MgB$_2$ grain boundary pinning. So, the flux pinning by secondary phases at grain boundaries is not in controversy with the $F_n(b = 0.2)$ peak position. In this way, the increasing amount of the secondary phase precipitates at the MgB$_2$ grain boundaries coincides with the $J_c$ and $B_{irr}$ enhancement by promoting an increase in (core) vortex pinning at these boundaries.

The series of Ag-doped samples indicates that the Ag–Mg phases, especially AgMg, effectively enhance the current flow in the inter-granular space by increasing flux pinning in this area. The increasing excess of Mg is very helpful ($J_c$ increased with the increasing AgMg amount 15 times). Doping with MgB$_4$ and Nd$_2$O$_3$ had a similar effect to the best sample with Ag. These systems, however, need a more indepth study to find their optimum composition and sintering route.

5. Summary

Electromagnetic and structural properties of a series of five MgB$_2$ samples with diverse additions were investigated. The series consisted of three Mg$_x$B$_2$ samples with 4 wt% Ag and a variable amount of Mg: $x = 1, 1.075$, and $1.1$, one MgB$_2$ sample doped with 1 wt% MgB$_4$ and another one with 1 wt% Dy$_2$O$_3$. Samples with Ag exhibited significant growth in $J_c$ and $B_{irr}$ with increasing excess of Mg. TEM/EDX identified the creation of nanoscopic grains of secondary phases in these samples, preferentially AgMg, but also small amounts of Ag$_3$ Mg and MgO. These precipitates, with the grain size around 20 nm, were always segregated at MgB$_2$ grain boundaries, with MgB$_2$ grains being about one order of magnitude larger than the precipitates. The same applied for the samples doped with MgB$_4$ and Nd$_2$O$_3$. At 15 K, the self-field $J_c$ reached $3.44 \times 10^5$ A cm$^{-2}$ in MgB$_2$ + 1 wt% MgB$_4$, in accordance with previously published results [16]. In the Ag-doped Mg$_x$B$_2$ samples with $x = 1, 1.075$, and $1.1$, the self-field $J_c$ values were at 15 K 2.88 $\times 10^5$ A cm$^{-2}$, 3.18 $\times 10^5$ A cm$^{-2}$, and 5.02 $\times 10^5$ A cm$^{-2}$, respectively, and 4.34 $\times 10^5$ A cm$^{-2}$ in the sample with 1 wt% Dy$_2$O$_3$. Magnetic relaxation measured at 10, 20, and 30 K showed a weak relaxation in MgB$_2$, in accordance with
the previous study [27]. As regards the additions, the best electromagnetic properties had the Ag-doped sample with 10 at% Mg excess (Mg$_1$B$_2$ + Ag). The sample doped with Dy$_2$O$_3$ was the second best, followed by the sample with MgB$_2$. All these results are within the relatively narrow range of 1–6 $\times$ 10$^5$ A cm$^{-2}$, where high-quality self-field $J_c$ data are published in the literature [14, 16, 17, 24, 25, 34]. The combined electromagnetic and microstructure analyses indicate an interesting combination of grain-boundary pinning with core pinning induced by normal secondary phase precipitates segregated at the MgB$_2$ grain boundaries.

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