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Large upper critical field and irreversibility field in MgB$_2$ wires with SiC additions

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Resistive transition measurements are reported for MgB$_2$ strands with SiC dopants. The starting Mg powders were 325 mesh 99.9% pure, and the B powders were amorphous, 99.9% pure, and at a typical size of 1–2 μm. The SiC was added as 10 mol % of SiC to 90 mol % of binary MgB$_2$ [(MgB2)0.9(SiC)0.1]. Three different SiC powders were used; the average particle sizes were 200 nm, 30 nm, and 15 nm. The strands were heat treated for times ranging from 5 to 30 min at temperatures from 675 °C to 900 °C. Strands with 200 nm size SiC additions had $\mu_0 H_{\rm irr}$ and B_{c2} which maximized at 25.4 T and 29.7 T after heating at 800 °C for 30 min. The highest values were seen for a strand with 15 nm SiC heated at 725 °C for 30 min which had a $\mu_0 H_{\rm irr}$ of 29 T and a B_{c2} higher than 33 T. © *2005 American Institute of Physics*. [DOI: 10.1063/1.1872210]

It has been demonstrated that in some cases the irreversibility field, $\mu_0 H_{\rm irr}$, and upper critical field, B_{c2}, of MgB$_2$ can be enhanced from the values seen from the binary compound. This has been most evident in thin-film results,[1–3] with B_{c2} reaching 49 T and even higher at 4.2 K. This is understood to be due to increased scattering in the conductor, an effect seen in a number of materials but more pronounced for MgB$_2$ because of its two-gap nature.[4] A full understanding of just what material modifications enable the B_{c2} enhancement in champion B_{c2} films is lacking, however, one possibility seems to be lattice distortion induced in part by C substitution in the B sublattice.[2] Several efforts to generate similar B_{c2} enhancements in MgB$_2$ wires and bulks has been stimulated by these results. A number of researchers have investigated C doping in bulks[5,6] to increase $\mu_0 H_{\rm irr}$ and B_{c2}, which are, of course, strongly correlated (in this context, see Ref. 2), and more generally high-field properties in wires with SiC.[7–11] Bulk samples which had been exposed to excess Mg vapor showed high B_{c2} values as well, with a $B_{c2}(0)$ estimate of 29 T.[2] Numerous other additives have also been investigated,[12,13] in some cases with the express purpose of changing the B or Mg sublattice, or otherwise changing the electronic state of the system.

Given the variation in preparation conditions and the sensitivity of MgB$_2$ critical fields to κ, there is some variation in the reported values of $\mu_0 H_{\rm irr}$ and B_{c2}. Irreversibility fields for *ex situ* MgB$_2$ tapes have been seen at ≈12 T for field perpendicular to the tape face[14] [*ex situ* tapes are anisotropic such that $H_\parallel \approx 1.4\, H_\perp$ (Ref. 15) due to partial alignment of the Mg and B planes parallel to the broad face of the tapes during rolling]. Suo and Flukiger found $\mu_0 H_{\rm irr}$ values of 8 T and 10.4 T in perpendicular and parallel orientations of the applied field, and, under the same orientations, B_{c2}s of 11.9 T and 15.1 T.[16,17] Recent results from Goldacker *et al.*[18] would extrapolate to higher values at 4.2 K. Matsumoto and Kumakura[19] used SiO$_2$ and SiC in the *in situ* process, enhancing $\mu_0 H_{\rm irr}$ from ≈17 T to ≈23 T at 4 K. ZrSi$_2$, ZrB$_2$, and WSi$_2$ additions were also attempted,[20] with some increases seen. Hydride-based MgB$_2$ powder with SiC dopants was also investigated, and seen to give B_{c2} values of ≈23 T.[21] Dou *et al.*[7–10] showed improved high-field critical current results for SiC, and similar wires measured in this laboratory showed improvements in the apparent irreversibility field as might be extrapolated from transport results.[11] In this work, strands of similar construction were investigated systematically, and high-field resistive transitions were used to demonstrate relatively large values of $\mu_0 H_{\rm irr}$ and B_{c2} with various kinds of SiC dopants under various heating conditions.

Round, monofilamentary, powder in tube (PIT) MgB$_2$ strands were fabricated by continuous (described previously in Ref. 11) and standard powder in tube methods. The outer sheath was Fe enclosed in Cu 30 wt % Ni. The starting Mg powders were 325 mesh 99.9% pure, and the B powders were amorphous, 99.9% pure, and at a typical size of 1–2 μm. For strands A and B, the powders were mixed in a rotating V-shaped tube and then planetary milled.[11] Coarse SiC (200 nm) powders were added during this process in the proportion 10 mol % of SiC to 90 mol % of binary MgB$_2$ [(MgB2)0.9(SiC)0.1]. Strands C and D, fabricated at the University of Wollongong, were also made from an *in situ* route, in this case with 10 wt % of "fine" SiC powder (15 and 30 nm). All strands were heated in flowing Ar at temperatures ranging from 640 °C to 725 °C for 30 min. Ramp-up and ramp-down times were short. Transport J_c results for similar wires have been previously reported,[11] val-

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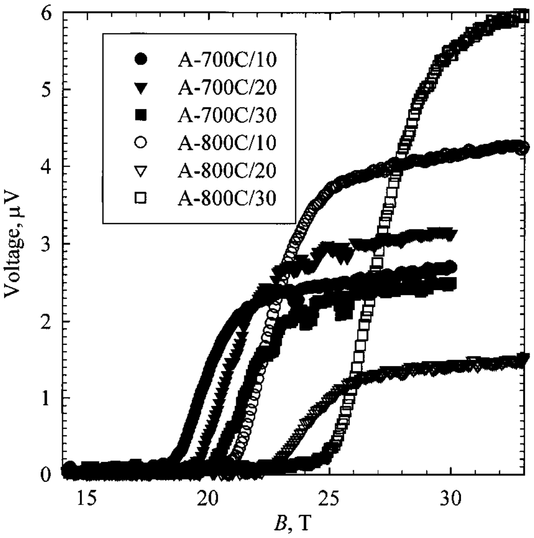

FIG. 1. Resistive transition (vs B) at 4.2 K for Strand A (200 nm SiC-doped MgB_2).

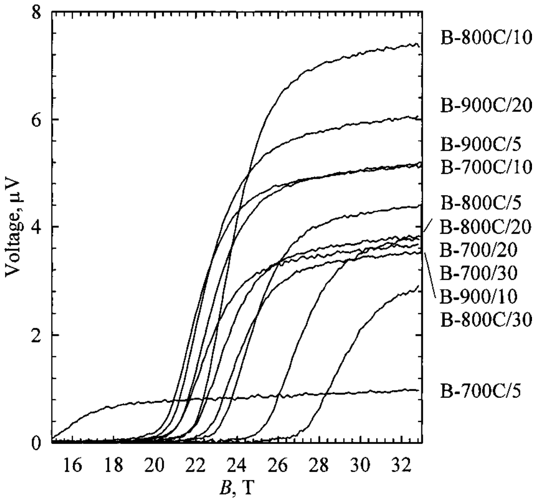

FIG. 2. Resistive transition (vs B) at 4.2 K for Strand B (200 nm SiC-doped MgB_2). Lines are used here rather than individual data points for clarity.

ues for samples from Strand A of the present series gave $8 \times 10^4\ \mathrm{A/cm^2}$ at 5 T and 4.2 K.

Four-point transport measurements were made on 1 cm long samples at the National High Magnetic Field Laboratory in Tallahassee, Florida. Standard Pb–Sn solder was used for forming the contacts on the outer sheath, and the distance between the voltage taps was 5 mm. The applied current was 10 mA, and current reversal was used. All measurements were made at 4.2 K in applied fields ranging from 0 to 33 T. The samples were placed perpendicular to the applied field, values of $\mu_0 H_{\mathrm{irr}}$ and B_{c2} being obtained taking the 10% and 90% points of the resistive transition. Resistive transitions in self-field were used to obtain T_c curves. In this case the samples were from neighboring sections of strand, and were 6 cm long, with voltage taps 3 cm apart.

Figure 1 shows the resistive transitions for Strand A after heating for various times at 700 °C and 800 °C. It can be clearly seen that both $\mu_0 H_{\mathrm{irr}}$ and B_{c2} increase with increasing heating time and temperature in the ranges investigated. The results for Strand A are given in Table I. These results are significantly higher than those of earlier reports for binary MgB_2 (for example, 8–10.4 T for $\mu_0 H_{\mathrm{irr}}$ and 12–15 for B_{c2}).[16,17] In particular, the $\mu_0 H_{\mathrm{irr}}$ and B_{c2} values for 800 °C and 30 min of 25.4 T and 29.7 T represent quite high $\mu_0 H_{\mathrm{irr}}$ and B_{c2} for SiC doped MgB_2 strands. The $\mu_0 H_{\mathrm{irr}}$ values are also higher than might have been expected from the extrapolation of high-field critical current results using a Kramer method.[11]

The response of Strand B is shown in Fig. 2, where trends similar to those of Strand A are seen. Curves of $\mu_0 H_{\mathrm{irr}}$ and B_{c2} for Strand B heated at various temperatures are plotted versus heating time in Fig. 3. In this case, a 900 °C curve is also present, which is lower than the 800 °C curve. Heating at 800 °C for 30 min gives the highest values, 29.4 T and 31.3 T for $\mu_0 H_{\mathrm{irr}}$ and B_{c2}, respectively. The T_c onset values (from resistive transitions under self-field) for the B-series samples heated for 5, 10, 20, and 30 min at 800 °C were 34.2 K, 34.4 K, 37.8 K, and 34.4 K respectively, with transition widths (as measured from 10% to 90% of the transition) of 1.2 to 1.4 K. The T_c value of about 33.2 K (for 10 and 30 min heatings) corresponds to an expected enhancement of B_{c2}.[2] Overall, while T_c is depressed from optimal values for MgB_2, no strong systematic correlation is seen between T_c and B_{c2} in these samples. This is likely to be due to inhomogeneity in the wire. It may be that various current paths exist, some of which have different compositions as well as different orientations of the MgB_2 grains with respect to the external field. This interpretation would be consistent with the lower $\mu_0 H_{\mathrm{irr}}$ that has been extrapolated from higher current

TABLE I. $\mu_0 H_{\mathrm{irr}}$ (4.2 K) and B_{c2} (4.2 K) for Strand A (200 nm SiC).

Sample name	Tracer ID	Heat treatment (°C/min)	$\mu_0 H_{\mathrm{irr}}$, T	B_{c2}, T
A-700C/10	HTR398	700/10	19.7	21.6
A-700C/20	HTR398	700/20	19.8	22.8
A-700C/30	HTR398	700/30	20.3	23.9
A-800C/10	HTR398	800/10	22.5	25.6
A-800C/20	HTR398	800/20	21.3	24.7
A-800C/30	HTR398	800/30	25.4	29.7

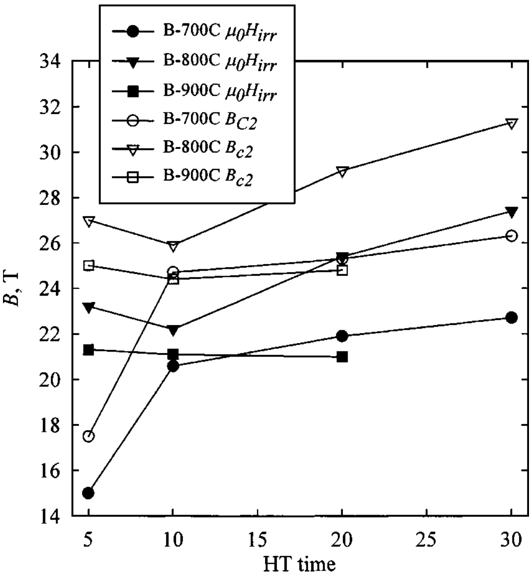

FIG. 3. Curves of $\mu_0 H_{\mathrm{irr}}$ (4.2 K) and B_{c2} (4.2 K) for Strand B heated at 700 °C, 800 °C, and 900 °C for various times.

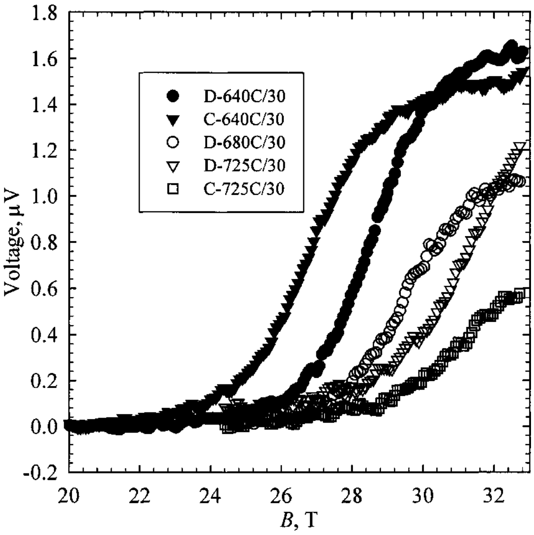

FIG. 4. Resistive transition (vs B) at 4.2 K for Strands C and D (15 nm and 30 nm SiC-doped MgB_2).

transport measurements on these wires (note that significant "tails" were present).[11]

Figure 4 shows the resistive transitions for Strands C and D after various heating temperatures and times. These two strands had the smallest SiC powder sizes (at 15 and 30 nm average size). No clear distinction between the results of these two strands is seen, although as a group they have higher values of $\mu_0 H_{\mathrm{irr}}$ and B_{c2} than do Strands A and B containing the coarse SiC. The highest values are seen for higher temperatures within this set, consistent with the results of Strands A and B. The highest values seen were for Strand C (15 nm SiC) which had a $\mu_0 H_{\mathrm{irr}}$ of 29 T and a $B_{c2} > 33$ T (see Table II).

In this work, we have presented $\mu_0 H_{\mathrm{irr}}$ and B_{c2} for MgB_2 strands with SiC additions made using PIT, *in situ* powder methods. Higher values of $\mu_0 H_{\mathrm{irr}}$ and B_{c2} were seen for strands heated at higher temperatures and, in some cases, longer times. Strands with finer SiC powders also had larger $\mu_0 H_{\mathrm{irr}}$ and B_{c2} values. In particular, strands with 200 nm size SiC additions had $\mu_0 H_{\mathrm{irr}}$ and B_{c2} which maximized at 25.4 T and 29.7 T for strands heated at 800 °C for 30 min. Strands with 15 nm and 30 nm additions had even higher values. The highest critical-field values were seen for a strand with 15 nm SiC additions which after 725 °C for 30 min had a $\mu_0 H_{\mathrm{irr}}$ of 29 T and a B_{c2} greater than 33 T.

TABLE II. $\mu_0 H_{\mathrm{irr}}$ (4.2 K) and B_{c2} (4.2 K) Strands C and D (15 nm and 30 nm SiC).

Sample name	Tracer ID	SiC size (nm)	Heat treatment (°C/min)	$\mu_0 H_{\mathrm{irr}}$, T	B_{c2}, T
D-640C/30	S1	30	640/30	26.4	30.4
C-640C/30	S2	15	640/30	24.2	28.2
D-680C/30	S3	30	680/30	27.0	31.2
D-725C/30	S5	30	725/30	≈28	>33
C-725C/30	S6	15	725/30	≈29	>33

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