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J. Horvat
University of Wollongong, jhorvat@uow.edu.au

Saeid Soltanian
University of Wollongong, saeid@uow.edu.au

Xiaolin Wang
University of Wollongong, xiaolin@uow.edu.au

S. X. Dou
University of Wollongong, shi@uow.edu.au

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Effect of sample size on magnetic J_c for MgB_2 superconductor

J. Horvat,[a] S. Soltanian, X. L. Wang and S. X. Dou,
Institute for Superconducting and Electronic Materials, University of Wollongong, NSW 2522, Australia

Abstract
A strong effect of sample size on magnetic $J_c(H)$ was observed for bulk MgB_2 when J_c is obtained directly from the critical state model. Thus obtained zero-field J_c (J_{c0}) decreases strongly with the sample size, attaining a constant value for the samples larger than a few millimetres. On the other hand, the irreversibility field (H_{irr}) defined at J_c = 100 A/cm^2 increases with the sample size. The decrease of J_{c0} is described in terms of voids in the bulk MgB_2 samples and magnetic screening around the cells of agglomerated crystals between these voids because of concentration of the current in the narrow bridges connecting the cells. For samples larger than a few millimetres, the value of magnetic J_c is in agreement with the transport J_c and it is restricted by the voids. The critical state model is not suitable for obtaining J_c for smaller bulk MgB_2. The increase of H_{irr} with the sample size is an artefact of defining H_{irr} by the value of J_c at which magnetic decoupling occurs within the cells.

Superconducting wires based on MgB_2 superconductor are currently in the process of fast development, resulting in improved values of J_c, $J_c(H)$ and H_{irr} [1,2,3]. We will show that incorrect conclusions can be deduced when comparing these values for the samples of different size. The value of J_c obtained from the critical state model for the samples larger than a few millimetres corresponds to the overall screening current density of the whole sample. This is equivalent to the J_c obtained in transport measurements. A direct application of the critical state model to calculate J_c of smaller MgB_2 samples leads to erroneous results, as the measured magnetic moment is contributed to by magnetic screening at various length-scales.

The field dependence of J_c was obtained from measurements of magnetic hysteresis loops, using a critical state model for appropriate geometry and the dimensions of the whole sample. The value of H_{irr} was obtained as the field at which thus obtained J_c equals to 100 A/cm^2. We will show that this model should not be used for small MgB_2 samples. Nevertheless, we use it to demonstrate its unsuitability for small MgB_2 samples and for large fields. The measurements were performed by a Quantum Design PPMS magnetometer, with the sweep rate of the field of 50 Oe/s. Two groups of samples were measured. An MgB_2 pellet was prepared by reacting magnesium and boron powders at 850°C under isostatic pressure of 150 MPa for 1 hour. The density of thus prepared pellet was 1.9 g/cm^3. The T_c of 38.9K was obtained from measurements of ac susceptibility, with the transition width lower than 1K. The pellet was cut into a rectangular rod and measured. Subsequent measurements were performed after reducing all three dimensions of the same sample by 20%, with their proportions remaining the same. In this way, any geometrical effects on our results were eliminated. The sample dimensions are shown in Table 1. The field was applied along the longest dimension of the sample, x. The second group of samples were round iron sheathed MgB_2 wires, prepared by powder in tube method, described elsewhere [4]. The iron sheath was removed before the measurements. Two groups of the wires were measured (Table 1). In the group D, the length of the wire was kept constant and its diameter was decreased for each of the measurements. In the group Z, the diameter was kept constant and its length was decreased. For each of the measurements, the field was applied along the cylindrical z-axis of the wire. Therefore, the dimension parallel to the field was varied in the group Z. On the other hand, the dimension perpendicular to the field was varied in the group D.

Figure 1 shows the field dependence of J_c at 5 and 20K, for a series of samples with subsequently decreasing volume. The J_c for low fields at 5K could not be calculated because of the flux jumps[5]. There was a strong influence of the sample size on $J_c(H)$ as well as on the zero-field J_c (J_{c0}). For example, the J_c dropped by more than two orders of magnitude at 7 T and 5K when the sample dimensions decreased from 25 to 0.26mm^3. This was accompanied by an increase of J_{c0} as the sample size decreased.

The increase of H_{irr} with the volume (V) of the sample is shown in Figure 2 for T = 20K. H_{irr} increased very fast with V for $V < 3.5$mm^3, followed by a much more gradual increase for $V > 3.5$mm^3. The transition between these two regimes occurred at the same volume for all temperatures, however H_{irr} vs. V for different temperatures were not scaleable to a unique curve. Inset to Figure 2 shows the field dependence of J_c for two different MgB_2 samples at 5K, obtained from magnetic and transport measurements. The voltage contacts in transport measurements were at a distance of 1 cm, whereas the sample size for magnetic measurements was 3.5 x 3 x 0.5 mm^3. There is a good agreement between the two types of measurements.

Figure 3 shows the dependence of J_{c0} on the sample volume. Inset to Figure 3 shows the field dependence of J_c, detailing the increase of J_{c0} as the sample volume was decreased. Normalising J_{c0} to its value at V=12.8 mm^3, the experimental points obtained at 10, 20 and 30 K overlapped. As the sample volume decreased from 25 to 6.9 mm^3, the value of J_{c0} increased by only 10%. However, the increase of about 60 % of the initial J_{c0} was obtained as the volume decreased from 6.9 to 0.25 mm^3. It was difficult to decrease the volume below this value, because of the sample fragility. However, the dependence of J_{c0} on V was progressively stronger as V decreased, and multiple increase of thus calculated J_{c0} can be expected for smaller volumes. This implies that the reported high values of J_{c0} for samples with V < 1mm^3 should not be directly compared with J_{c0} obtained for larger samples.

Figure 4 shows the dependence of J_{c0} on the length of the wire (samples Z), therefore on the dimension parallel to magnetic field. J_{c0} again decreases with the length of the sample. Inset to Figure 4 shows the dependence of J_{c0} on the diameter of the round wire (samples D), with the field along its z-axis. J_{c0} also decreases with the sample diameter. This excludes the change of the electrical field (E) as the origin for the sample size dependence of J_{c0}. Namely, for a cylindrical sample with magnetic field along its z-axis: $E = D/2*dB/dt$ on its surface, where D is the diameter of the sample. However, our measurements show that J_{c0} also changes with the length of the cylinder (Fig.4), where E remains constant.

The explanation for the sample size dependence of J_{c0} should instead be sought in the terms of sample homogeneity. The density of bulk MgB_2 is quite smaller than the density of MgB_2 crystal (about 70%). The bulk MgB_2 consists of a matrix of well-connected superconducting grains. Voids of the size of a few tens of micrometers are scattered through the matrix[6], dividing it into cells of agglomerated grains between the voids connected by narrow bridges. The screening currents flowing around the sample are concentrated in these bridges, resulting in an increased current density in the bridges. The current transport through the bridges limits the overall screening of the whole sample. The cells and bridges consist of the same material and have the same J_{c0}. However, the cross-sectional area for the current flow in the cells is larger than for the current in the narrow bridges. Because of this, extra screening is possible, with the current loops closing around the cells. This resembles the inter- and intra- grain current in high-temperature superconductors, with a significant difference that the bridges between the cells in high quality MgB_2 samples are not Josephson junctions. The sample size dependence of magnetically obtained J_c can be explained by a model devised for high-temperature superconductors [7]. Approximating the shape of the sample and cells by round cylinders, the reversible magnetic moment obtained from the hysteresis loop is [7]:

$$\Delta m = \left(afJ_{ca} + DJ_{cb}\right)2V/3, \qquad (1)$$

where J_{ca} and J_{cb} are the current densities of the small loops around the cells and of the overall sample screening, respectively. D is the diameter of the sample, whereas a and f are the typical diameter of the cells and their filling factor, respectively. J_{ca} and J_{cb} are not critical current densities of the MgB_2 crystals. J_{ca} is restricted by the vortex pinning in the crystals and by the bottlenecks to the current flow between MgB_2 crystals inside the cells, whereas J_{cb} is additionally restricted by the bottlenecks created by the voids between the cells. This gives $J_{ca} > J_{cb}$. The contribution of J_{ca} to Δm is negligible for large samples ($D >> a$ in Eq. (1)), resulting in a sample size independent J_c equal to J_{cb}. With lowering the sample size, the contribution of J_{ca} starts increasing. Further, the length scale used for calculating J_c in the critical state model (i.e. D) starts approaching the size of the cells. Because of this, and because $J_{ca} > J_{cb}$, thus calculated value of J_c increases with the decrease of the sample size. As the sample approaches the size of the cells, J_{cb} decreases because the bridges between the cells are broken up. Finally, for $D = a$, the calculated J_c equals J_{ca}. The simple critical state model cannot be used to calculate J_c when both J_{ca} and J_{cb} contribute significantly to Δm. The fit of normalised J_{c0} vs. V, using Eq. (1) for Δm in the critical state model with $J_{cb}/J_{ca} = 1/3$ and $af = 25$ μm, is shown by solid line in Fig. 3. Microscopic examination of the samples shows that the average size of the cells is about 35 μm and the density of the sample gives $f = 0.7$, which results in $af = 25$ μm. The value of $J_{cb}/J_{ca} = 1/3$ was obtained by the fitting. This is a sensible value, considering that the difference between J_{ca} and J_{cb} arises because of the bottlenecks to the current flow between the cells.

Figures 2 and 3 show that J_{c0} and H_{irr} have the opposite dependence on the sample volume. This is seemingly contradicting, because larger H_{irr} signifies stronger vortex pinning and consequently, a larger J_{c0}. However, Figure 1 shows that the values of H_{irr} were obtained from the part of the $J_c(H)$ curve exhibiting a steep decrease of J_c. This step in $J_c(H)$ was explained by breaking down of the sample into separated screening islands[5]. These islands were agglomerates of crystals and their size in the first bulk MgB_2 was about 200μm[5]. TEM examination of the more advanced samples reveals smaller agglomerates, of about 200nm, consisting of sub-grains of the size of 10 nm[8]. Our preliminary measurements indicate the size of these screening islands for the samples reported here of about 100 nm. This indicates that the sample size dependence of H_{irr} in Fig.2 is an artefact of the magnetic breakdown into ~100 nm large agglomerates of grains inside the cells. Larger samples require a larger field in order to obtain full decoupling of the islands closer to the centre of the sample. This is manifested in an increase of the field at which the step in $J_c(H)$ occurs, and thus of the apparent H_{irr}, with the sample size.

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Table 1: The dimensions of the samples measured. Samples a-h are rectangular pellets whose all three dimensions (*x*, *y*, *z*) were reduces by 20% before each subsequent measurement. The field was oriented along *x*. Samples Z are round wires whose length was reduced before each subsequent measurement. The field was along the length of the samples, i.e. along the cylindrical *z*-axis. Samples D were round wires with constant length, but the diameter was reduced before each subsequent measurement. The field was also along the cylindrical *z*-axis.

sample	x (mm)	y(mm)	z (mm)	V (mm^3)
a	7.15	3.27	1.07	25.01
b	5.72	2.62	0.86	12.87
c	4.65	2.12	0.7	6.90
d	3.64	1.68	0.57	3.49
e	2.92	1.34	0.46	1.78
f	2.29	1.08	0.36	0.89
g	1.87	0.85	0.29	0.46
h	1.42	0.68	0.24	0.23

sample	Diameter (mm)	Length (mm)
D1	1.54	3.91
D2	1.23	3.91
D3	0.93	3.91
Z1	1.54	6.23
Z2	1.54	3.91
Z3	1.54	1.95
Z4	1.54	1.50

Figure 1: Field dependence of J_c for samples of different size (Table 1).

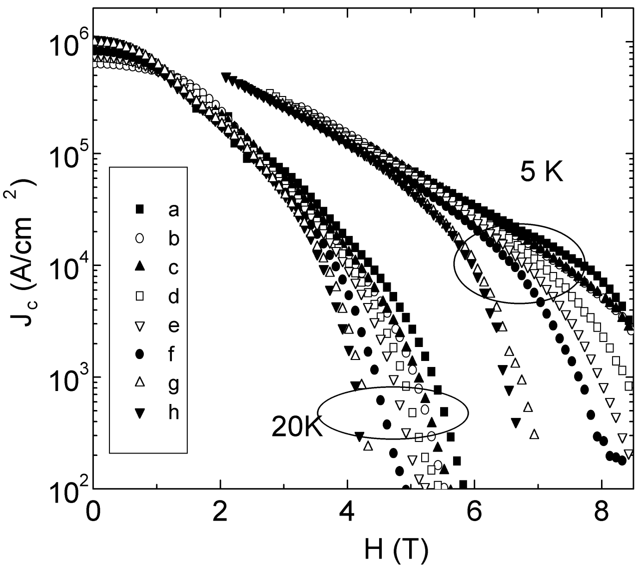


Figure 2: Dependence of the irreversibility field on the sample volume for the rectangular samples a-h at T=20K. Inset: Field dependence of J_c for MgB_2 samples at 5K, obtained from magnetic (solid line) and transport (open symbols) measurements.

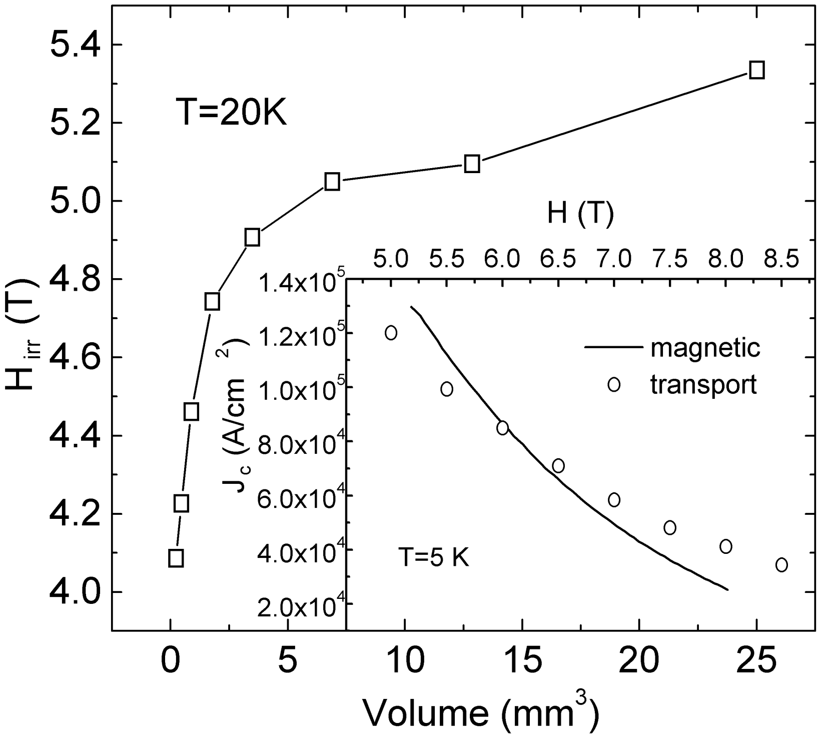


Figure 3: Dependence of the normalised J_{c0} on the sample volume for samples a-h (Table 1). J_{c0} for T = 10, 20 and 30 K was normalised to its value for V = 12.87 mm^3. Solid line is fit with Eq.(1). Inset: Field dependence of J_c showing the increase of J_{c0} as the volume decreases.

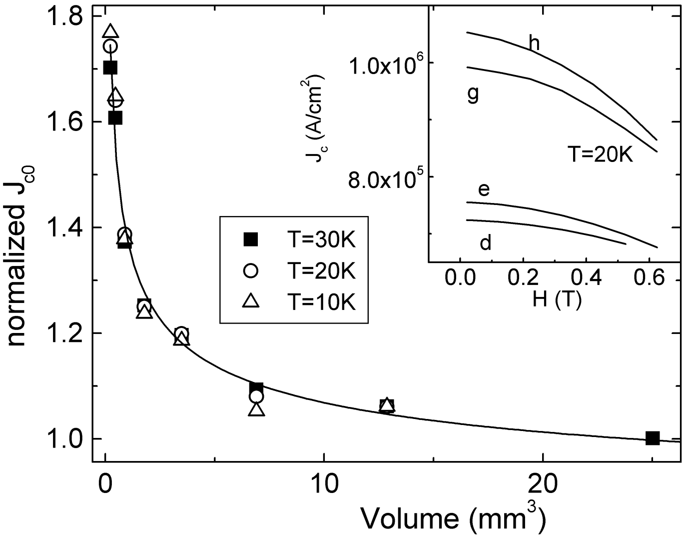
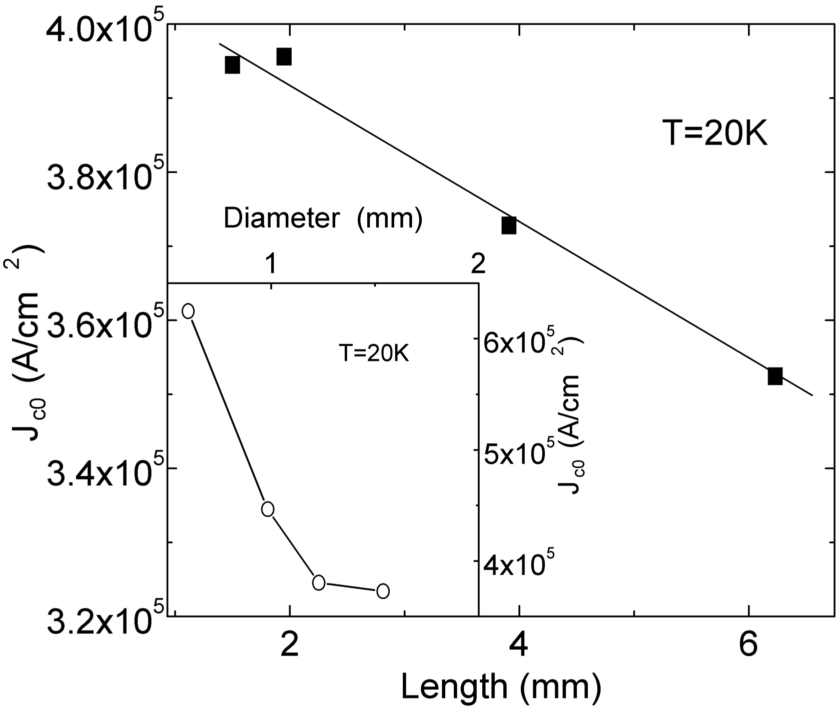


Figure 4: Dependence of J_{c0} on the sample length of the MgB_2 wire, where the diameter of the wire did not change (samples Z in Table 1). Inset: Dependence of J_{c0} on the diameter of the MgB_2 wire, with fixed wire length (samples D in Table 1).



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