Magnetic measurement of transport critical current density of granular superconductors*

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We describe two magnetic techniques that may be used to determine the transport critical current density J_{ct} of granular superconductors by measuring the intergranular magnetization of a sample. In the first method, magnetization critical current density J_{cm} is used to estimate J_{ct} by isolating the intergranular magnetization and applying the critical state model. In the second method, magnetic detection is used to measure J_{ct} directly: intergranular magnetization hysteresis loops are obtained while increasing a transport current through a sample. The critical current density J_{ct} is that value of transport current density which causes the intergranular magnetization to collapse at a given magnetic field and temperature. Both methods give values of J_{ct} in fair agreement with values obtained from conventional transport measurements of J_{ct}. Magnetization was measured with both extraction and Hall probe magnetometers.

Keywords: critical currents; granularity; high temperature superconductors

High temperature oxide superconductors are often granular, although the deleterious effects of granularity on current conduction may be minimized by grain alignment in thin films and partially melted, textured specimens. Measurements of magnetization M, as a function of magnetic field H and temperature T, are often used to characterize superconductors because the measurements are sensitive and relatively simple, and M can be related to critical current density J_c by the phenomenological critical state model. The magnetization signal arises from shielding currents and penetrated flux in the superconductor.

Values of magnetization critical current density $J_{cm}(H,T)$ deduced from magnetic hysteresis loops on *uniform* samples are smaller than values of transport critical current density $J_{ct}(H,T)$ obtained from voltage as a function of current. One reason is that the criteria for J_{cm} are more sensitive than those for J_{ct}. As pointed out by Jin *et al.*[1], 'the voltage criterion . . . used for J_c determination .in transport measurement is orders of magnitude less stringent than in the magnetic measurement, and hence tends to overestimate J_c . . .'. As stated by Maley *et al.*[2], 'these measurements probe very different time scales and voltage sensitivities . . .'. The striking variations in measured J_c values as a consequence of different electric field criteria are illustrated graphically in Reference 3.

One can run into trouble when measuring J_{cm} in granular materials. Magnetic fields decouple granular superconductors, so at high fields J_{cm} refers to the grains and bears no relation to J_{ct} of the bulk sample. Only at low fields, where the grains are coupled, can J_{cm} approximate J_{ct}. However, the familiar critical state formulas for J_{cm} are not accurate below the full penetration field[4,5], especially in thin discs or films with the field applied perpendicular to the surface[6–9]. Furthermore, once the sample has been exposed to high fields, there are shielding currents in the grains even at low fields. The critical state model is applicable to homogeneous superconductors, and granular conductors are not homogeneous.

Consider a granular superconductor whose grains are decoupled at high fields. The critical state relationship is $J_{cm} \propto M/a$, where M is the volume magnetization and a is the appropriate dimension. At high fields, use of the bulk sample dimension for a will underestimate J_{cm} of the grains. Moreover, J_{cm} of the *sample* is not well defined, but if it were computed, it would *overestimate* J_{ct} of the sample: in this example of decoupled grains J_{ct} is zero by definition.

Magnetization M is magnetic moment per unit volume. Consider two Nb–Ti samples of known J_c. One is a cylinder of radius r and height t. The other is a cylinder of radius $r/2$ and height $4t$. Their volumes are the same but their critical state magnetizations in axial field are different: $M_1 = J_c r/3$ and $M_2 = J_c r/6$. Unlike magnetic materials, the magnetic moments of superconductors in the critical state scale with their transverse dimension, not their volumes. This follows from the property that the shielding current cannot exceed the critical current, and the current path is limited to the cross-sectional area of the sample. The restriction does not apply to the diamagnetic shielding state in which each cylinder would have a magnetization equal to $-H$.

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Despite limitations on the utility of the critical state model for granular superconductors, we present two magnetization techniques that may be used to obtain values of J_{ct}. They measure the intergranular magnetization and are used at low fields where the superconductor grains are in the shielding state.

Magnetization of samples carrying transport current

Intergranular magnetization

Magnetization critical current density J_{cm} can be used to determine transport J_{ct} when the intergranular magnetization can be separated from the magnetization of the grains. At temperatures approaching the critical temperature T_c, the intergranular magnetization decays strongly with field, decoupling the grains before flux has penetrated them and leaving the grains perfectly diamagnetic. We say that the intergranular magnetization is separated from that of the grains as long as the maximum field is below the lower critical field H_{c1} of the grains.

Figure 1 is a typical set of hysteresis loops of magnetization as a function of increasing maximum field for sintered $YBa_2Cu_3O_{7-\delta}$ at 76 K. Such intergranular hysteresis loops of high temperature superconductors have been described and discussed by others[5,10–15]. The major loop has a maximum field of 12 kA m^{-1} (150 Oe). Also shown is the central portion of a loop to 15 kA m^{-1} (190 Oe) (dots). The sample dimensions are 10.7 × 1.2 × 1.0 mm and the field was applied parallel to the long edge as shown in the sketch. The overall negative slope of the complete loops is due to the diamagnetism of the grains, non-hysteretic at these low fields.

The initial curve joins the extrema of the minor hysteresis loops, and the field where the initial curve meets the major loop is usually regarded as the full penetration field H_p. However, this identification of full penetration is tenuous; note how the descending branch of the 8 kA m^{-1} (100 Oe) loop, for example, does not meet the major loop until negative fields and how the loop does not develop a full width ΔM. That is, the value of ΔM is a function of the maximum applied field. Furthermore we have no assurance that the 12 kA m^{-1} (150 Oe) loop represents the fully penetrated state. We do know that increasing the field to 15 kA m^{-1} (190 Oe) results in some flux penetration of the grains because the final ascending branch of the loop falls below the universal initial curve.

Extraction magnetometer

The data in *Figure 1* were acquired with an extraction magnetometer. Long ago, ballistic magnetometers were used for magnetization measurements. Cylindrical samples were rapidly removed from a coil about their centre plane, and the induced voltage was read on a ballistic galvanometer. Modern extraction magnetometers[16,17] are based on this technique. Our magnetometer has the capability of simultaneous application of a transport current, or current density J, to the sample. The extraction magnetometer described in Reference 17 was adapted with a special sample rod fitted with current leads. Low resistance Ag contact pads for the current were sputtered on to the sample and annealed[18]. The sample was immersed in liquid nitrogen. In these measurements H was parallel to J, but in principle any orientation could be used, subject to sample space limitations in the magnetometer. A superconducting solenoid was operated in the normal state for improved low field resolution. Data were recorded every 400 A m^{-1} (5 Oe) and the points were connected by line segments to form the loops.

Several hysteresis loops with a maximum field of 8 kA m^{-1} (100 Oe) were measured with different values of d.c. transport current through the sample. *Figure 2* shows loops for 0, 2 and 4 A labelled in terms of current density J. A small magnetic moment proportional to the transport current contributed a constant offset to the magnetization. We increased the offset numerically to separate the loops in *Figure 2*. In addition to the effect of pinching together the ascending and

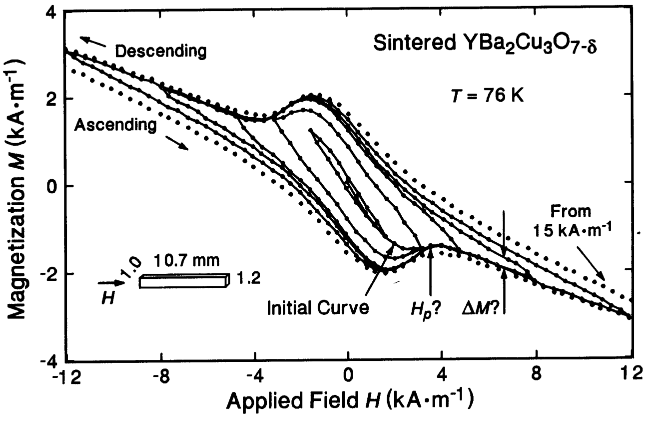

Figure 1 Magnetization M of sintered $YBa_2Cu_3O_{7-\delta}$ at 76 K as a function of applied field. The diamagnetic background of the grains is superimposed on the intergranular magnetization. Questions arise as to the meaning of the full penetration field H_p and the width of the hysteresis loops ΔM. Data were acquired with an extraction magnetometer

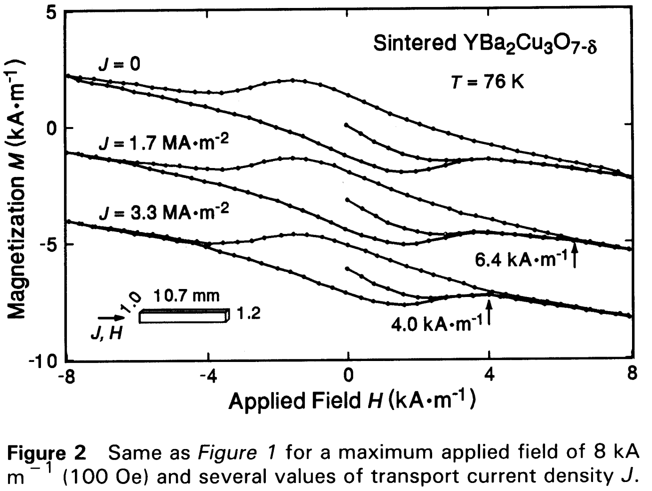

Figure 2 Same as *Figure 1* for a maximum applied field of 8 kA m^{-1} (100 Oe) and several values of transport current density J. The curves have been shifted vertically. The fields at which the loops pinch together are identified

descending branches of the loops, the current also reduced somewhat the magnetic signal of the sample at low fields, decreasing the magnetic hysteresis loss[19].

In a more complete treatment, hysteresis losses in the presence of transport current can be incorporated in a critical state model[20], but in this paper we are concerned with the collapse of the hysteresis loop, not the equations for the magnetization curves. Traditional a.c. loss and magnetization measurements of superconductors carrying transport current are done on coils of long lengths of wire, and the current is maintained below the critical current[21,22]. In the same general category as our experiments, alternating field susceptibility measurements on a sample carrying d.c. transport current were reported by Fisher *et al.*[23], Badía *et al.*[24], and Grishin *et al.*[25], who noted differences for currents above and below the critical current, and observed that the transport current suppressed intergranular diamagnetism.

Hall probe magnetometer

Figure 3 is a plot of the intergranular magnetization curve of a different, more weakly coupled sample of sintered $YBa_2Cu_3O_{7-\delta}$ in liquid nitrogen. Its critical temperature, determined by resistivity, was 92 K. The maximum applied field H was 2.8 kA m^{-1} (35 Oe), below the lower critical field H_{c1} of the grains. The sample dimensions were 12.5 × 3.3 × 2.4 mm, with H oriented as shown in the sketch. The field was produced by an electromagnet ramped linearly at 1 kA m^{-1} s^{-1} and was measured with a Hall probe gaussmeter. The measurements were independent of ramp rate down to 10 A m^{-1} s^{-1}. Such frequency independence in low fields is characteristic of intergranular flux[26,27]. The sample magnetization M was sensed with a cryogenic Hall probe oriented in the 'M configuration'[28,29]. However, the plane of this Hall probe was not positioned exactly parallel to H, but was tilted slightly to actually intercept H. The tilt angle was adjusted to exactly compensate the negative signal from the diamagnetism of the grains in the sample. (The tilt angle was kept constant for all values of transport current.)

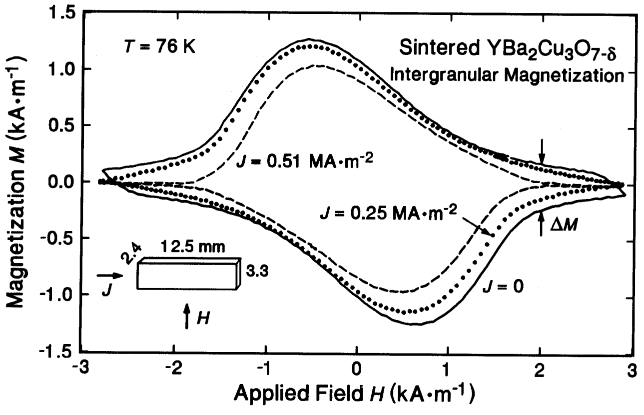

Figure 3 Intergranular magnetization M of sintered $YBa_2Cu_3O_{7-\delta}$ at 76 K as a function of applied field H for several values of transport current density J. The figure illustrates how transport critical current density can be determined by the width of the intergranular hysteresis loop ΔM and by the value of J required to pinch together the diamagnetic and paramagnetic branches of the hysteresis loop. These compensated loops were measured with a Hall probe magnetometer

The compensated hysteresis loops in *Figure 3* thus lack the familiar diamagnetic slope seen in *Figure 1*. The compensation is not at all required for the analysis but it serves to highlight the intergranular hysteresis loop; it could be done numerically. The M gaussmeter was calibrated using the superconductor sample measured on a vibrating sample magnetometer as a transfer standard. Magnetization was calculated as magnetic moment per total sample volume. The analogue outputs of both gaussmeters were input to low pass filters and then digitally recorded simultaneously. The curves in *Figure 3* are composed of about 200 data points connected by line segments.

During magnetization data acquisition the current was increased until the diamagnetic and paramagnetic branches of the hysteresis loop pinched together at about 2.8 and 2.0 kA m^{-1} (35 and 25 Oe). Hysteresis loops for two values of transport current (2 and 4 A) are shown in *Figure 3*, labelled in terms of J.

Critical current density

We can determine J_{ct} at a given field and temperature for each of two specimens in three ways: (1) application of the critical state model, (2) knowledge of the value of J required to pinch together the hysteresis loop and (3) voltage measurements.

Hall probe magnetometer data

(1) As a reference field we choose 2.0 kA m^{-1} (25 Oe). The vertical width of the hysteresis loop ΔM (for zero transport current) in the Bean critical state model is related to J_{cm}: $\Delta M = J_{cm}\,[a(1 - a/3b)]$, where, for our geometry[4,30], $2a = 2.4$ mm and $2b = 12.5$ mm. We get $J_{cm} = 0.37$ MA m^{-2}. Better accuracy may be obtained by accounting for the grain fraction in the sample[5,13,14,31]. A more sophisticated use of critical state theory to deduce J_{cm} for such compensated hysteresis loops would be to fit them with an exponential model for the field dependence of J_c[5,14,32,33].

(2) The current density required to close the hysteresis loop at the reference field was $J_{ct} = 0.51$ MA m^{-2} (*Figure 3*). In this method, *magnetic* detection is used to determine *transport* critical current density. This is one of the main points of the paper. What about a correction for the self field of the transport current? The average self field for this sample with 4 A of transport current was 270 A m^{-1} (3.4 Oe), but this is not a relevant parameter. Our method measures J_{ct} just as in a transport measurement. In both measurements the orientations and magnitudes of H and J are specified. There is no single, inherent value of J_{ct}; we must decide where the hysteresis loop collapses, in analogy with an electric field criterion.

(3) Finally, we measured J_{ct} using an automated four-probe apparatus[3] to measure voltage V as a function of current I. The magnetic field orientation with respect to the transport current was the same as in the magnetic measurements, and the sample was immersed in liquid nitrogen. We obtained values of $J_{ct} = 0.48$ MA m^{-2} for an electric field criterion of 1 μV cm^{-1} and 0.60 MA m^{-2} for 10 μV cm^{-1}. The values obtained by the three methods are summarized in *Table 1* along with values at 2.8 kA m^{-1} (35 Oe).

Table 1 Comparison of values of critical current density obtained by three methods for the sample described in *Figure 3* (Hall probe magnetometer)

Method	J_c (MA m^{-2})	
	H = 2.0 kA m^{-1}	2.8 kA m^{-1}
ΔM (critical state)	0.37	–
$\Delta M \rightarrow 0$ (pinch)	0.51	0.25
$V-I$ (1 μV cm^{-1})	0.48	0.30
$V-I$ (10 μV cm^{-1})	0.60	0.39

Extraction magnetometer data

(1) We refer again to *Figures 1* and *2*. As discussed above, there is uncertainty in the value of ΔM. We attribute this to inadequate isolation of the intergranular magnetization for this sample at 76 K. That is, if we raise the field high enough to penetrate the intergranular component fully, flux penetrates the grains. The critical state measurement of ΔM fails in this case. (Some benefit might be obtained by reducing the sample size perpendicular to the field direction. This would decrease the full penetration field H_p and shift the intergranular loop to lower field.)

(2) The loop pinched together at 4.0 kA m^{-1} (50 Oe) for $J = 3.3$ MA m^{-2}, at 5.1 kA m^{-1} (64 Oe) for $J = 2.5$ MA m^{-2} (not shown) and at 6.4 kA m^{-1} (80 Oe) for $J = 1.7$ MA m^{-2}, thus indicating the values of J_{ct} at those fields. The loop for a current of 1 A did not pinch together. Why is the $J = 3.3$ MA m^{-2} loop not as well pinched together as the 1.7 MA m^{-2} loop? We believe this is due to the irreversibility in J_c measured in ascending and descending fields, which we observe in our $V-I$ transport measurements. The irreversibility in J_c was 2% at 6.4 kA m^{-1} but 4% at 4.0 kA m^{-1}. A judicious choice of maximum field for a given level of transport current would ensure a well defined pinch. For example, a maximum field of about 6 kA m^{-1} for $J = 3.3$ MA m^{-2} would have been adequate.

(3) Comparison values of J_{ct} measured conventionally with H parallel to J using a 1 μV cm^{-1} criterion are 4.5 MA m^{-2} at 4.0 kA m^{-1}, 3.4 MA m^{-2} at 5.1 kA m^{-1} and 2.5 MA m^{-2} at 6.4 kA m^{-1}, somewhat higher than the values deduced from magnetization. The results are summarized in *Table 2* along with values obtained using other electric field criteria. The $\Delta M \rightarrow 0$ method appears to correspond to an effective electric field criterion of about 0.01 μV cm^{-1}.

Final remarks

The Hall probe and extraction magnetometers used in this study are especially suited for magnetization measurements of superconductors carrying transport current. The Hall probe magnetometer has a much faster field cycle. Measurements on thin films or tape samples would require H to be oriented perpendicular to the plane to achieve adequate sensitivity.

During the measurements, current through the samples was maintained even when the samples were in the normal state at the higher fields. This mandates a cryogen bath for adequate heat sinking. It is wise to use a current supply that switches from current to voltage regulation as the sample resistance increases. Samples stabilized with, for example, Ag substrate would require less protection.

The intergranular magnetization techniques described above are applicable to a range of temperatures and fields which may be limited by the need to isolate intergranular magnetization. However, they may be used readily for homogeneous conductors where intergranular effects are not an issue. In a granular material, when the intergranular magnetization is not separable at low fields, the hysteresis loop will not pinch together but will instead collapse upon the granular loop. For high current measurements on homogeneous conductors, sample quench protection would be required.

Magnetization measurements of samples carrying transport current, unlike traditional measurements for critical state estimates of J_{cm}, require samples with current contacts, which may be inconvenient. The benefit of magnetization measurements over voltage measurements might lie in their greater sensitivity for samples with adequate cross-section. Voltage measurements are one-dimensional, whereas magnetization measurements are two-dimensional and are likely to provide more complete characterization. Our goal in this paper was to discuss limitations of critical state estimates of J_c in granular superconductors and to characterize experimentally the effect of transport current on intergranular magnetization.

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Table 2 Comparison of values of critical current density obtained by two methods for the sample described in *Figure 2* (extraction magnetometer)

Method	J_c (MA m^{-2})		
	H = 4.0 kA m^{-1}	5.1 kA m^{-1}	6.4 kA m^{-1}
$\Delta M \rightarrow 0$ (pinch)	3.3	2.5	1.7
$V-I$ (0.1 μV cm^{-1})	4.2	3.1	2.2
$V-I$ (1 μV cm^{-1})	4.5	3.4	2.5
$V-I$ (10 μV cm^{-1})	5.0	3.8	2.9

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References

1 **Jin, S., van Dover, R.B., Tiefel, T.H., Graebner, J.E. and Spencer, N.D.** Critical current and magnetization in *c*-axis textured Bi–Pb–Sr–Ca–Cu–O superconductors *Appl Phys Lett* (1991) **58** 868–870

2 **Maley, M.P., Kung, P.J., Coulter, J.Y., Carter, W.L., Riley, G.N. and McHenry, M.E.** Behavior of critical currents in Bi–Pb–Sr–Ca–Cu–O/Ag tapes from transport and magnetization measurements: Dependence upon temperature, magnetic field, and field orientation *Phys Rev B* (1992) **45** 7566–7569

3 **Goodrich, L.F. and Bray, S.L.** High T_c superconductors and critical current measurement *Cryogenics* (1990) **30** 667–677, see *Figure 6*

4 **Chen, D.-X. and Goldfarb, R.B.** Kim model for magnetization of type-II superconductors *J Appl Phys* (1989) **66** 2489–2500

5 **Calzona, V., Cimberle, M.R., Ferdeghini, C., Putti, M. and Siri, A.S.** Critical current density in sintered $YBa_2Cu_3O_{7-y}$: A comparison between different kinds of measurements and problems in their interpretation *Meas Sci Technol* (1990) **1** 1356–1362

6 **Frankel, D.J.** Critical-state model for the determination of critical currents in disk-shaped superconductors *J Appl Phys* (1979) **50** 5402–5407

7 **Däumling, M. and Larbalestier, D.C.** Critical state in disk-shaped superconductors *Phys Rev B* (1989) **40** 9350–9353

8 **Conner, L.W. and Malozemoff, A.P.** Calculations of the dimensional dependence of the critical state in disk-shaped superconductors *Phys Rev B* (1991) **43** 402–407

9 **Angadi, M.A., Caplin, A.D., Laverty, J.R. and Shen, Z.X.** Nondestructive determination of the current-carrying length scale in superconducting crystals and thin films *Physica C* (1991) **177** 479–486

10 **Senoussi, S., Oussena, M., Ribault, M. and Collin, G.** Critical fields and the critical current density of $La_{1.85}Sr_{0.15}CuO_4$ *Phys Rev B* (1987) **36** 4003–4006

11 **Itoh, K., Wada, H., Kuroda, T., Kaieda, Y., Odawara, O. and Oie, T.** Superconducting critical current densities and flux trapping in sintered YBaCuO *Cryogenics* (1988) **28** 575–579

12 **Zhang, H., Yan, S.S., Ma, H., Peng, J.L., Sun, Y.X., Li, G.Z., Wen, Q.Z. and Zhang, W.B.** The magnetization properties of high-T_c superconductor $YBa_2Cu_3O_7$ at low magnetic fields *Sol State Commun* (1988) **65** 1125–1129

13 **Dersch, H. and Blatter, G.** New critical-state model for critical currents in ceramic high-T_c superconductors *Phys Rev B* (1988) **38** 11391–11404

14 **Calzona, Y., Cimberle, M.R., Ferdeghini, C., Putti, M. and Siri, A.S.** AC susceptibility and magnetization of high-T_c superconductors: Critical state model for the intergranular region *Physica C* (1989) **157** 425–430

15 **Radhakrishnamurty, C., Grover, A.K., Mishra, P.K., Chaddah, P. and Subbarao, G.V.** AC magnetic measurements and J_c measurements in high T_c superconductors *Physica C* (1989) **162–164** 333–334

16 **Cullity, B.D.** *Introduction to Magnetic Materials* Addison-Wesley, Reading, MA, USA (1972) 65–66

17 **Krause, J.K.** Extraction magnetometry in an ac susceptometer *IEEE Trans Magn* (1992) **28** 3066–3071

18 **Ekin, J.W., Panson, A.J. and Blankenship, B.A.** Method for making low-resistivity contacts to high T_c superconductors *Appl Phys Lett* (1988) **52** 331–333

19 **Wilson, M.N.** *Superconducting Magnets* Clarendon Press, Oxford, UK (1983) 171–174

20 **Minervini, J.V.** Two-dimensional analysis of ac loss in superconductors carrying transport current *Adv Cryog Eng (Materials)* (1982) **28** 587–599

21 **Ogasawara, T., Takahashi, Y., Kanbara, K., Kubota, Y., Yasohama, K. and Yasukochi, K.** Alternating field losses in superconducting wire carrying dc transport currents: Part 1, single core conductors *Cryogenics* (1979) **19** 736–740

22 **de Reuver, J.L., Beunk, H.D., Roovers, A.J.M. and van der Klundert, L.J.M.** AC field losses in a dc current-carrying conductor containing one layer of NbTi filaments *Int Conf Magnet Tech (MT-9)* (Eds Marinucci, C. and Weymuth, P.) Swiss Inst Nucl Res, Zurich (1985) 501–504

23 **Fisher, L.M., Il'in, N.V., Podlevskikh, N.A. and Zakharchenko, S.I.** Peak-effect in HTSC ceramics at a low magnetic field *Sol State Commun* (1990) **73** 687–690

24 **Badía, A., Angurel, L.A., Lera, F., Rillo, C., Navarro, R., Bartolomé, J., Blasco, J. and Aponte, J.** Inductive and transport simultaneous measurements of the superconducting properties of YBaCuO ceramics: A comparative study *Supercond Sci Technol* (1992) **5** S415–S418

25 **Grishin, A.M., Korenivski, V.N., Rao, K.V. and Ulyanov, A.N.** Suppression of intra- and intergranular properties by transport current in Bi-based ceramic superconductors *IEEE Trans Magn* (1992) **28** 2220–2222

26 **Nikolo, M. and Goldfarb, R.B.** Flux creep and activation energies at the grain boundaries of Y–Ba–Cu–O superconductors *Phys Rev B* (1989) **39** 6615–6618

27 **Caplin, A.D., Bungre, S.S., Cassidy, S.M., Laverty, J.R. and Shen, Z.X.** Dissipation in granular high-temperature superconductors *Physica A* (1990) **168** 268–276

28 **Cross, R.W. and Goldfarb, R.B.** Hall probe magnetometer for SSC magnet cables: Effect of transport current on magnetization and flux creep *IEEE Trans Magn* (1991) **27** 1796–1798

29 **Cross, R.W.** Transport current effects on flux creep and magnetization in Nb–Ti multifilamentary cable strands *Adv Cryog Eng (Materials)* (1992) **38** 731–736

30 **Campbell, A.M. and Evetts, J.E.** Flux vortices and transport currents in type II superconductors *Adv Phys* (1972) **21** 199–428, see p 270

31 **Chen, D.-X., Nogués, J. and Rao, K.V.** A.c. susceptibility and intergranular critical current density of high T_c superconductors *Cryogenics* (1989) **29** 800–808

32 **Chen, D.-X., Sánchez, A. and Muñoz, J.S.** Exponential critical state model for magnetization of hard superconductors *J Appl Phys* (1990) **67** 3430–3437

33 **Chen, D.-X. and Sánchez, A.** Magnetic properties of high-T_c superconducting grains *Phys Rev B* (1992) **45** 10793–10796