Zeitschrift: Helvetica Physica Acta

Band: 61 (1988)

Heft: 7

Artikel: Low-field magnetic susceptibility measurements of high-T_C granular superconductors

Autor: Pollini, A. / Mota, A.C. / Visani, P.

DOI: https://doi.org/10.5169/seals-115979

Helvetica Physica Acta, Vol. 61 (1988) 1007–1012
0018–0238/88/071007–06$1.50 + 0.20/0

Low-field magnetic susceptibility measurements of high-T_c granular superconductors

By A. Pollini, A. C. Mota and P. Visani

Laboratorium für Festkörperphysik
ETH Hönggerberg, 8093 Zürich, Switzerland

(11. III. 1988)

Abstract. Low-field isothermal dc susceptibility measurements can distinguish between the contribution of shielding currents across different grains and the intrinsic diamagnetism of granular superconductors. In powdered Sr–La–Cu–O at $T \ll T_c$, the first flux penetration into the voids encircled by different grains joined by Josephson junctions occurs at $H_A = 1$ mOe. On raising the field, more intergrain persistent loops become critical gradually from $H_A = 1$ mOe up to $H_B = 0.5$ Oe. On cycling the field up to 30 Oe, the magnetization is reversible above H_B and irreversible below this field. Constant low amplitude ac susceptibility is insensitive to this effect.

In a recent paper [1] we briefly discussed the behaviour of the low field susceptibility in Sr–La–Cu–O and Ba–La–Cu–O. In the high-T_c superconducting oxides, determinations of the Meissner effect as well as of the lower critical field H_{c1} are complicated due to the granular nature of the available specimens [2, 3]. Here we discuss in more detail our measurements of the isothermal ac and dc susceptibilities at $T \ll T_c$ as a function of magnetic field in Sr–La-Cu–O in powdered and sintered form. As pointed out by Hein [4] both types of measurement respond to *magnetic shielding* which is different from *expulsion of magnetic flux* – the Meissner effect – out of the specimen. Nevertheless, we find that by measurements of $\chi_{ac}(H)$ and $\chi_{dc}(H)$, we can clearly separate the contribution of shielding currents across different grains joined by Josephson junctions from the diamagnetic signal of the grains by themselves. Shielding currents encircling voids in the specimen give a highly hysteretic response while the diamagnetic signal corresponding to the grains by themselves is completely reversible at low enough fields.

In our experimental arrangement $\chi_{ac}(H)$ or $M(H)$ at a constant temperature are both detected with SQUIDs. No superconducting shielding or persistent coils are used. The earth's field is reduced by means of a cryoperm shield at 4.2 K to about 2 mOe. The external field coil, primary coil, and secondary coil are all wound tightly around the experimental space.

A calibration of χ_{ac} against $\chi_{dc} = \Delta M/\Delta H$ for $H \to 0$ was obtained with classical superconductors like Ta and also with superconducting Cu and Ag

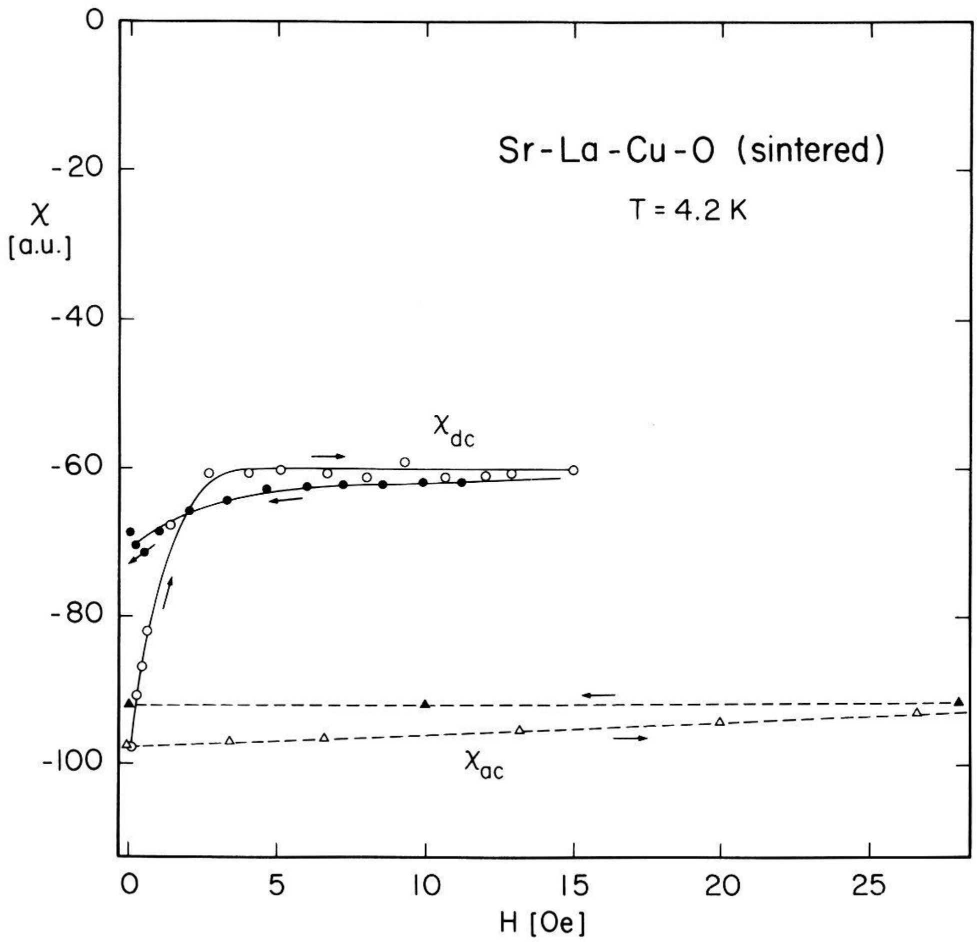

Figure 1
dc susceptibility (circles) and ac susceptibility (triangles) in arbitrary units as a function of magnetic field for a *sintered* Sr–La–Cu–O specimen. The open symbols correspond to raising the field H and the closed symbols to lowering H.

induced by the proximity effect [5]. The frequency and peak to peak amplitude ranges of the exciting field in the χ_{ac} measurements are $16\,\mathrm{Hz} < \nu < 160\,\mathrm{Hz}$ and $0.07\,\mathrm{mOe} < H_{pp} < 33\,\mathrm{mOe}$. The temperature range is $5\,\mathrm{mK} < T < 9.5\,\mathrm{K}$.

In Fig. 1 we show the real component of the ac isothermal susceptibility χ_{ac} ($H_{pp} = 1.5\,\mathrm{mOe}$) as well as χ_{dc} taken from the slope of a virgin magnetization curve, $\chi_{dc} = \Delta M/\Delta H$, as a function of the external dc field H for a sintered Sr–La–Cu–O specimen in the form of a parallelepiped ($1.7 \times 1.7 \times 8\,\mathrm{mm}^3$). These data were taken at $T = 4.2\,\mathrm{K}$. The units in this graph have been chosen to correspond to percents of the diamagnetic signal obtained for a bulk type I superconductor of the same size and geometry. We notice in Fig. 1 that at $H \simeq 0$, χ_{dc} and χ_{ac} are equal. As the external field is raised (open symbols) the absolute value of χ_{dc} and χ_{ac} are reduced with $|\chi_{dc}| < |\chi_{ac}|$ at all fields. Furthermore, for $H < 3$ Oe, χ_{dc} is very irreversible when the field is cycled at constant temperature, while for $H > 3$ Oe, it is almost reversible for the fields shown in Fig. 1.

The same type of data is shown in Fig. 2 for powdered Sr-La-Cu-O. We notice that in this case the irreversible enhancement of $|\chi_{dc}|$ occurs for fields below $H_B = 0.5$ Oe, and that it amounts to about 15% of the reversible diamagnetic signal as compared to 65% for the sintered specimen.

In both cases, the low field enhancements of $|\chi_{dc}|$ are due to the granular nature of the specimens. At low fields, persistent currents across different grains joined by Josephson junctions contribute to the measured χ_{dc} and χ_{ac}. As the field is raised, currents are induced such that for some very small field H_A (not given in Figs. 1 and 2) some of the intergrain loops become critical, i.e. they carry the critical current of the weakest Josephson junction in the loop. At the field H_A, flux is allowed to enter into the void enclosed by the loop which has first become critical. The critical currents of the intergrain loops are much lower than the intrinsic critical currents of the grains and depend on the quality of the sintering process. In Fig. 1 the leveling off of χ_{dc} at $H_B \simeq 3$ Oe and in Fig. 2 at $H_B \simeq 0.5$ Oe correspond to the fields beyond which all the possible intergrain loops carry critical currents.

In Figs. 1 and 2 we notice that the values of χ_{dc} at the reversible regions above H_B are 60% and 55% respectively of the full diamagnetic signal displayed by a Ta specimen of equal size and geometry. These numbers are to be expected when one takes into account the high value of the field penetration depth λ in these materials as compared to typical grain sizes. In Fig. 3 we show a scanning electron microscope photograph where grain sizes above 10 μm as well as much below 1 μm can be clearly seen in the powdered Sr-La-Cu-O specimen of Fig. 2.

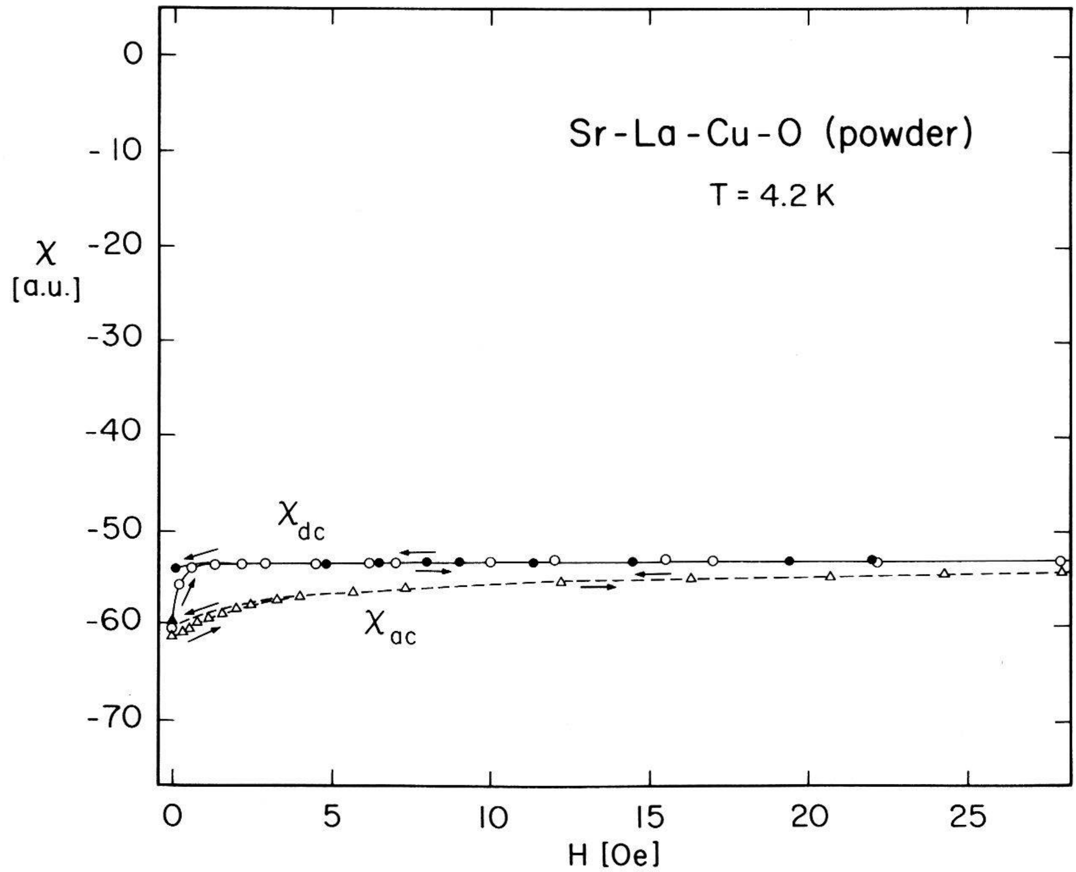

Figure 2
dc susceptibility (circles) and ac susceptibility (triangles) in arbitrary units as a function of magnetic field for a *powdered* Sr-La-Cu-O specimen. The open symbols correspond to raising the field H and the closed symbols to lowering H.

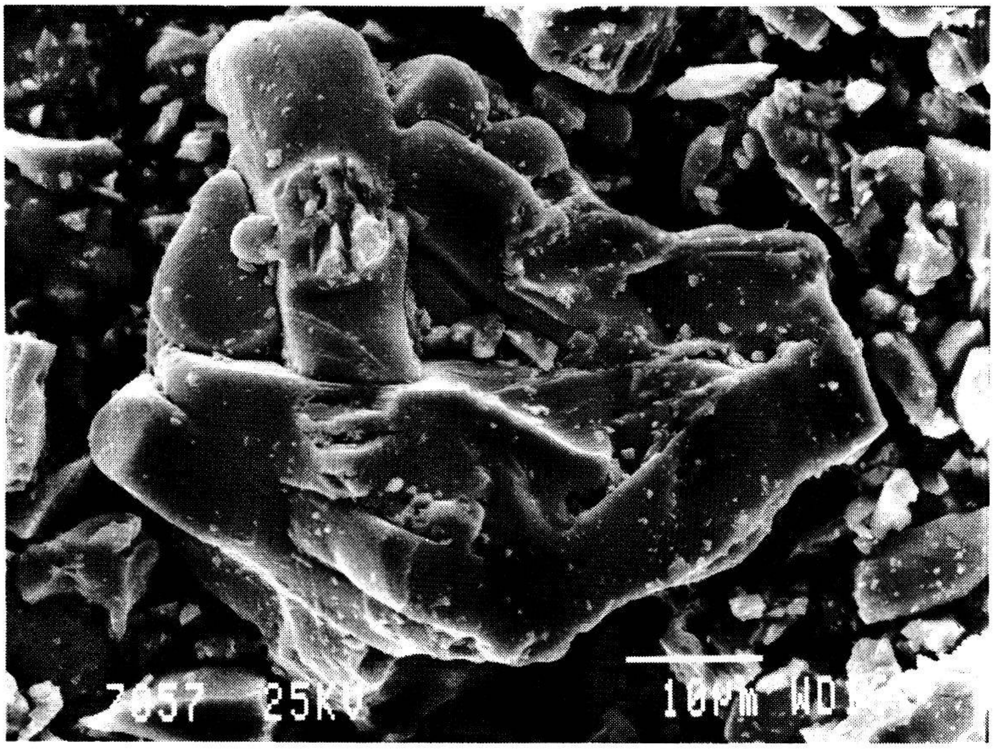

Figure 3
Photograph of powdered Sr-La-Cu-O taken with a scanning electron microscope.

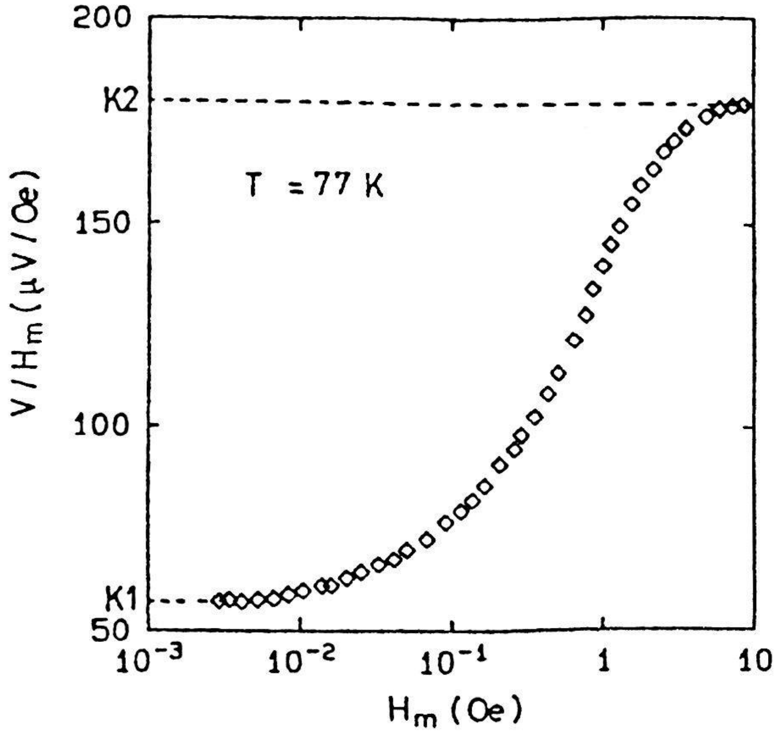

Figure 4
ac susceptibility of a sintered Y-Ba-Cu-O specimen at 3560 Hz as a function of ac field amplitude H_m. The vertical scale is proportional to χ_{ac}. This figure has been taken from Ref. 2.

As shown in Figs 1 and 2 the ac susceptibility, measured at a constant ac field peak to peak amplitude of 1.5 mOe, does not follow the continuous intergrain flux penetration between H_A and H_B as displayed by χ_{dc}. On the other hand, if the amplitude of the ac measuring field is increased to values such that the induced currents of the intergrain loops become critical, as is done in the experiments of Raboutou et al [2], a response similar to the $\chi_{dc}(H)$ measurements is obtained. This can be seen in Fig. 4 taken from their paper. Here the susceptibility χ_{ac} is given as a function of the amplitude of the exciting ac field (H_m in their notation) for a sintered Y-Ba-Cu-O specimen. The measuring frequency is $\nu = 3560$ Hz. Their data show that the first flux penetration occurs at $H_A = 5$ mOe and that the leveling-off of χ_{ac} occurs at $H_B = 8$ Oe. Furthermore, Raboutou et al [2] found that Y-Ba-Cu-O behaves similarly to a system made of weakly coupled classical superconducting grains, i.e. Nb grains, joined by 10^8 Josephson contacts [6].

Measurements of χ_{ac} as a function of the amplitude of the measuring field are shown in Fig. 5 for the powdered Sr-La-Cu-O specimen of Fig. 2. We can clearly determine that the first flux penetration occurs at $H_A = 1$ mOe ($H_{pp} = 2$ mOe) and that about 15% of the contribution to the susceptibility from intergrain shielding currents is destroyed in fields as low as 16.5 mOe ($H_{pp} = 33$ mOe).

The results of this work may be summarized as follows: isothermal dc susceptibility measurements in the high-T_c ceramics distinguish clearly three different regimes at low fields. There is a reversible region from $H = 0$ up to a field H_A of the order of few millioersted, where the first flux penetration into the voids of the granular material occurs. This is followed by an irreversible region between H_A and H_B. In this second region, as the field is increased beyond H_A,

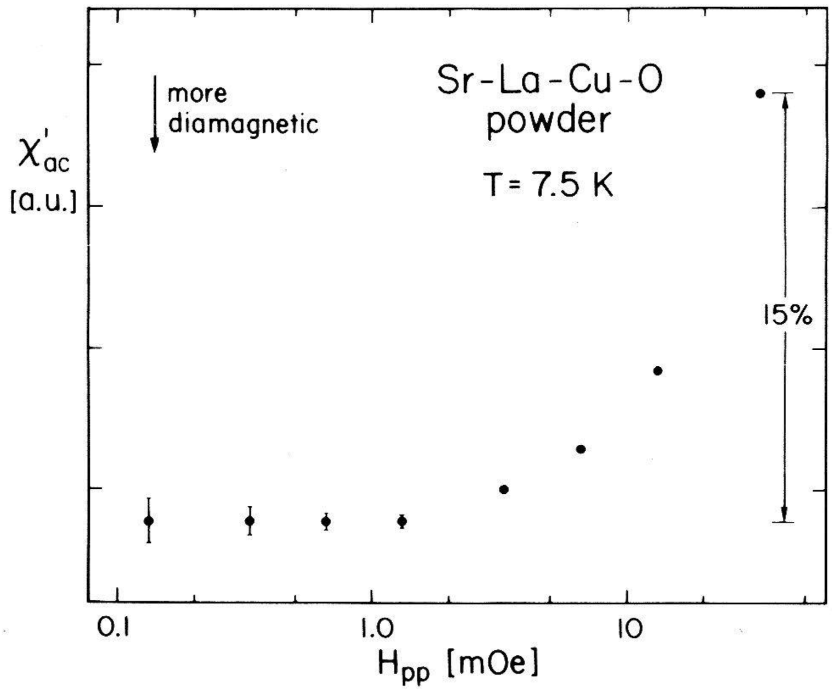

Figure 5
ac susceptibility in arbitrary units as a function of p-p amplitude of the ac field for the powdered Sr-La-Cu-O specimen of Fig. 2.

the intergrain shielding currents successively become critical. Typical values of H_B are few oersted, depending on the sintering process. For fields $H > H_B$, flux penetrates freely into the voids and the susceptibility $\chi_{dc}(H)$ is again reversible for H below the critical field H_{cl} of the specimen. The ac susceptibility $\chi_{ac}(H)$ is almost insensitive to the granular behaviour described here as long as the exciting field amplitude is smaller than H_A. A similar behaviour to χ_{dc} can be obtained in measurements of χ_{ac} taken at variable exciting field amplitudes.

Acknowledgement

We are grateful to J. L. Olsen and J. Rosenblatt for fruitful discussions. We are also indebted to P. Wägli for the SEM photograph. This work was partially supported by the Schweizerischer Nationalfonds zur Förderung der wissenschaftlichen Forschung.

REFERENCES

[1] A. C. MOTA, A. POLLINI, P. VISANI, K. A. MÜLLER and J. G. BEDNORZ, *High Temperature Superconductivity*, Eds. S. LUNDQVIST, E. TOSATTI, M. TOSI, YU LU (ICTP, Trieste 1987) World Scientific. pp. 255–262
[2] A. RABOUTOU, P. PEYRAL, J. ROSENBLATT, C. LEBEAU, O. PEÑA, A. PERRIN, C. PERRIN and M. SERGENT, Europhysics Lett., *4*(11) 1321 (1987).
[3] D. A. ESPARZA, C. A. D'OVIDIO, J. GUIMPEL, E. OSQUIGUIL, L. CIVALE and F. de la CRUZ-preprint.
[4] R. A. HEIN, Phys. Rev. *B33*, 7539 (1986).
[5] A. C. MOTA, P. VISANI and A. POLLINI, Jap. J. of Applied Phys. *26*, 1693 (1987).
[6] A. RABOUTOU, J. ROSENBLATT and P. PEYRAL, Phys. Rev. Lett., *45*, 1035 (1980).