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Irreversibility and flux creep properties of the superconducting oxide $\text{La}_{1.85}\text{Sr}_{0.15}\text{CuO}_4$

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Résumé.— Des mesures de couple magnétique sur un échantillon céramique de l'oxyde La-Sr-Cu-O donnent des informations sur ses propriétés supraconductrices. Sous une rotation régulière du champ magnétique les signaux de couple sont observés et indiquent des réponses de la structure des lignes de flux qui sont rigides ou viqueuses selon l'histoire magnétique de l'échantillon et la force du champ magnétique. Si au temps zéro le champ magnétique est réorienté dans une nouvelle direction, le signal en fonction du temps montre des effets de fluage à deux champs bien plus faibles que H_{c2} . Les données sont discutées en termes d'une image granulaire de la supraconductivité. L'ancrage intra-grain semble être faible.

Abstract.— Torque measurements on a ceramic sample of the oxide La-Sr-Cu-O give information on its superconducting properties. Under steady rotation of the magnetic field torque signals are observed which indicate rigid or viscous responses of the flux line structure depending on the magnetic history of the sample and the strength of the applied field. If the field is turned to a new direction at time zero, the time dependent torque signal displays strong flux creep for fields far below H_{c2} . The data are discussed in terms of a granular description of the superconductivity. Intrinsic intra-grain flux pinning appears to be weak.

1. Introduction.

Magnetic measurements on superconductors provide basic information on important physical properties. Samples of the superconducting oxides produced by standard ceramic techniques appear to show granular superconductivity even though the samples are essentially single phased crystallographically [1]. Being chemically single phased does not of course exclude strong grain boundary effects, particularly in highly anisotro-

pic systems. The fact that low critical current densities are generally observed in bulk ceramic samples [2] is certainly related to this granular structure. Magnetization cycle curves of typical samples show hysteresis at rather low fields (10 gauss) followed by a quasi reversible regime and then by a strong irreversibility for higher applied fields [3]. The field up to which $m(H)$ is broadly linear and reversible has been loosely identified with H_{c1} [4]. From a careful study of

$m(H)$ curves it has been concluded that this field represents flux penetration into superconducting grains [5]. In addition, because irreversibility on temperature cycling and non-exponential relaxation were observed [6] it has also been suggested that the low field state can be interpreted as a "superconducting glass" with superconducting grains interacting through weak links.

We have studied the torque signals of a ceramic $\text{La}_{1.85}\text{Sr}_{0.15}\text{CuO}_4$ sample to obtain further information on the irreversibility and on the relaxation, or creep. In torque experiments a signal is observed when the magnetization does not remain aligned with the applied field, when the latter is rotated. For a superconductor this means that the flux lines are pinned in such a way that they are not fully free to turn to follow the field. We find clearly defined systematic changes in behaviour with the applied field. Below a characteristic field (related to " H_{c1} " as defined above) torque signal are weak ; above this field there is a crossover to strong torque. Within each of these regimes we have "rigid" or "viscous" signals, depending on the strength of the turning field. The viscous behaviour is associated with creep occurring over a wide time spectrum.

2. Experimental.

The sample was prepared in the form of a cylindrical sintered pellet following standard methods. A capacity cell torque meter was used for the measurements. Fields up to 8.7 kG could be applied.

A number of alternative experimental procedures could be followed.

1) After zero field cooling (ZFC), the field is raised to a value H_t in the initial direction $\theta=0$, and is then rotated at a constant rate through 360° and back (about 15 minutes for a full rotation). The torque signal is monitored permanently.

2) After ZFC the field is cycled up to our maximum value (8.7 kG) and back to zero before raising the field to H_t and rotating as in 1).

3) As in 2) except that after raising the field to H_t it is turned through an angle θ and kept at this angle while the signal relaxation is monitored as a function of time.

4) The sample is field cooled (FC) in H and after stabilizing at temperature T the field is rotated.

We can outline schematically what we could

expect for standard type II superconductors. For fields lower than H_{c1} there is no flux penetration so when the field is turned the diamagnetic magnetization turns freely to remain aligned on the new direction of the field. There is zero torque. For fields between H_{c1} and H_{c2} the flux lines have penetrated the sample ; if the vortices are pinned then the magnetization is not free and there will be a torque signal.

For non zero torque we can distinguish between two extreme type of signal : rigid or viscous. In the first case, if the flux lines are held firmly fixed in the sample by the pinning forces, then the torque $\Gamma = \mathbf{m} \wedge \mathbf{H}$ will be equal to $mH \sin\theta$ and will be reversible, i.e. will depend only on θ and not on the field rotation sequence. In the second case, when the field is rotated the flux lines reorganize to adjust to the new field direction. If the rotation is continued long enough in the same sense the magnetization direction will lag behind the field direction by a constant angle giving a constant torque. The signal will inverse when the sense of the rotation is reversed. Intermediate cases which are partly rigid and partly viscous can also occur.

3. Results.

We will essentially describe data taken at 4.2 K. In figures 1-5 we show a number of recordings of torque signals observed during rotation in field H_t after ZFC (in fact a cooling field less than 1 gauss). Curve A of figure 1, in $H_t = 2.5$ G, shows no detectable torque within the sensitivity of the set up (better than 0.2 ergs) and only a weak parasitic background signal appears. Curve B for $H_t = 10$ G shows a weak but definite torque signal. For curve C, $H_t = 20$ G, there is a typical and fully developed viscous signal showing the magnetization lagging behind the field. If after ZFC we cycle the field to 20 G and then use a turning field of 2.5 G, we find a weak rigid signal, indicating a pinned remanent magnetization.

Returning to the series of rotation cycles after ZFC, curves with $H_t = 50$ and 100 G (Fig.2 curve A) are almost identical to curve C of figure 1, *so up to these field there is no change in behaviour*. However from about 120 G up the torque signal strength begins to increase dramatically, figure 2 curve B. Now we turn to the results obtained after field cycling to 8.7 kG and back to zero. If we use a turning field of 45 G there is a large quasi-rigid torque signal showing that there is a remanent magnetization which is

anchored to the field cycling direction $\theta=0$, figure 3. As we rotate in a series of higher fields (after following the same field cycling procedure each time) the signal goes over gradually to the viscous type, showing that the flux line pattern is reorganizing in response to the field rotation, figures 3-5.

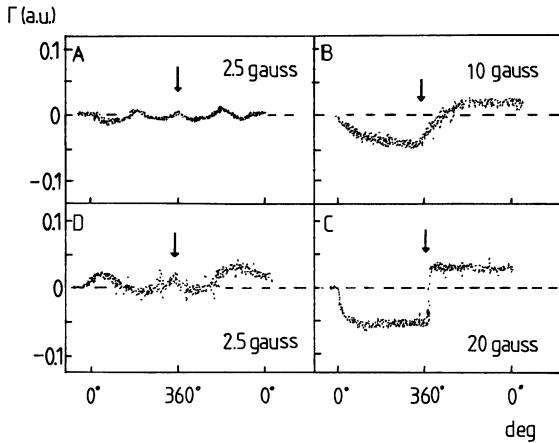


Fig.1.- Torque intensity against the angle between the magnetic rotating field H_t and the its initial direction when the field is rotated at a constant rate. The arrows at 360° indicate that the sense of rotation of the field was reversed. Curve A : after ZFC, $H_t = 2.5$ Gauss. Curves B and C : as curve A with H_t equal to 10 Gauss and 20 Gauss respectively. Curve D : after field cycling at $\theta=0$ to 20 Gauss and back to zero, rotating field $H_t = 2.5$ Gauss. The oscillation in A are a background parasitic signal. All the rotation figures presented in this paper are given using the same arbitrary units for the torque signal. (1 a.u. is about 500 erg/cc).

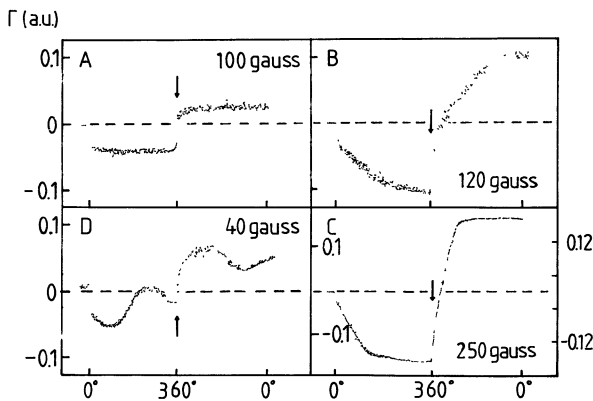


Fig.2.- As for figure 1. Curves A, B, C : after ZFC with different H_t . Curve D : after field cycling to 250 Gauss and back, $H_t = 40$ Gauss. Arrows again indicate inversion of field rotation.

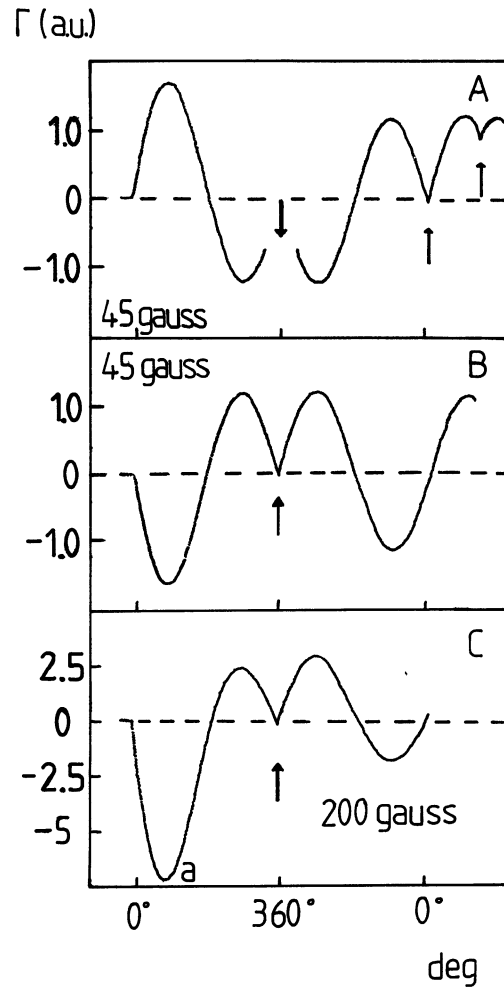


Fig.3.- Recordings made after field cycling to 8.7 kG and back to zero followed by rotation in turning fields H_t of 45 and 200 Gauss. In A and B the initial sense of rotation was clockwise and anticlockwise respectively. In A further inversions were performed at the second and third arrows.

If in this viscous limit situation we calculate the value of the moment perpendicular to the field ($\Gamma = m_\perp H$ by definition) we find $m_\perp \approx 2$ emu/g for fields above about 1 kG. This moment is about as strong as the longitudinal moment obtained by cycling the field without rotating it [5]. The slow rotation of the field then results in a situation where the overall moment is always at an angle of about 90° to the instantaneous direction of the field. The screening currents are reacting to the change in the field (which is perpendicular to the field direction) and thus produce a strong perpendicular moment.

Field cooling in 100 G followed by rotation in the same field, figure 6, shows that this procedure produces a rigid moment which is about one tenth of the moment produced by field cycling to 8.7 kG after ZFC, and which is completely dif-

ferent from direct application of 100 G at 4.2 K. This can be ascribed to flux lines being frozen in irreversibly during the cooling procedure.

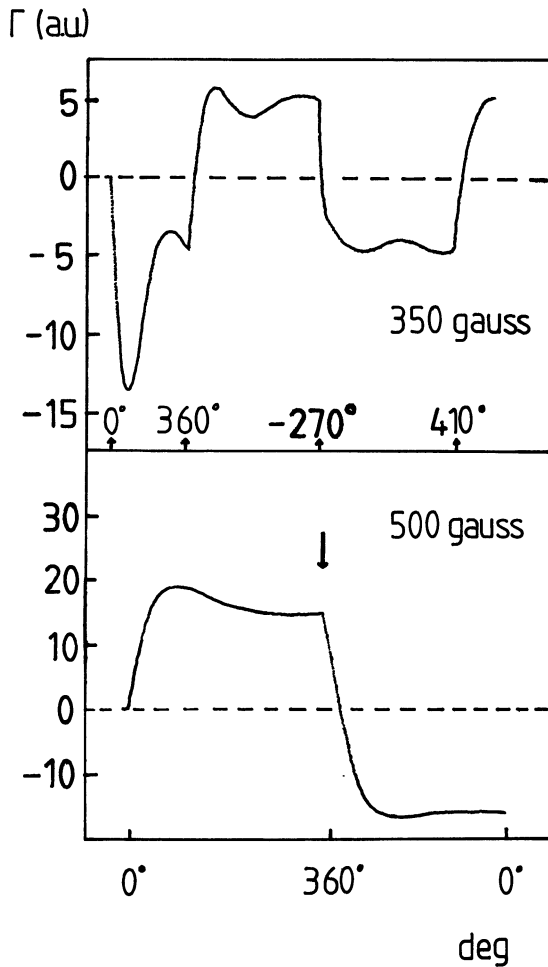


Fig.4.- As figure 3, with H_t values of 350 and 500 Gauss. For 350 Gauss three successive rotations were made as indicated.

We have made measurements of the torque signal relaxation for $H_t = 200$ G and higher after field cycling [7]. The relaxation is not exponential; the overall decay of the signal $\Delta\Gamma(t)/\Gamma(0)$ at time scale of 2500 seconds is maximum (for $\theta = 5^\circ$) at $H_t \simeq 1$ kG. For higher fields we are in the viscous limit and the relaxation appears to stabilize at a lower rate and takes up a power law form $\Delta\Gamma(t) \simeq t^{-\alpha}$ with α going from 0.048 to 0.031 for field values going from 1 kG to 8.7 kG.

For fixed values of field and angle, relaxation is faster at higher temperature. We have not yet explored the temperature dependence thoroughly.

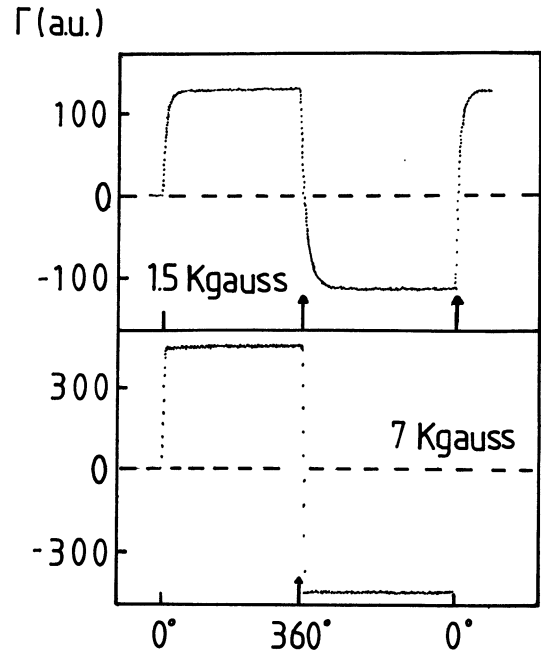


Fig.5.- As figure 3, with H_t values of 1.5 and 7 kG. Note that with increasing H_t the torque curves have evolved from rigid to viscous form.

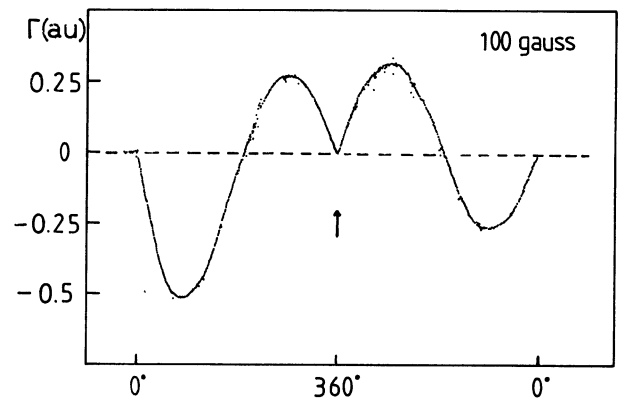


Fig.6.- As for previous figures, but after FC in 100 Gauss, $H_t = 100$ Gauss.

4. Discussion.

We can interpret our results using a phenomenological granular superconductivity description [5,8,9]. Suppose the sample consists of strongly superconducting grains coupled by weak links. We do not yet have a very clear idea of what grains and links are in the sample, but we can imagine that the "grains" can either be identified with the real physical grains or with a smaller scale defect structure inside the physical grains. The effective grain size may de-

pend to some extent on the applied field. We can distinguish various field regimes. At very low field ($H < 1\text{G}$) the flux is completely excluded from the sample according to magnetization data (Squid or magnetometer) [9] ; as the torque sensitivity is low in low applied fields, we see no torque signal until about 5 G. Then at rather higher field the flux lines can penetrate at least partly into the weak link structure ; this leads to a weak viscous torque signal rather than zero signal as the weak link system is not completely destroyed by the field. If our sample is cycled to 100 G and back to a much lower field value, there is a weak rigid signal, showing that the weak remanent moment is then pinned in direction.

Finally cycling to higher fields gives stronger torque signals ; we take this as indicating that the flux lines have started to penetrate the grains irreversibly and that thereafter the nature of the flux line structure is quite different. The onset field for flux penetration into the grains can be identified with an effective H_{c1} for the grains. The torque signal is particularly sensitive to this onset, so the H_{c1} estimated from this criterion is lower than that obtained from magnetization curves [5].

It is clear that the viscous response for turning fields greater than about 500 G indicates a limit to the pinning forces for the flux lines with the grains.

For the relaxation, in the fully viscous regime (i.e. at a combination of field and angle such that the signal has reached the plateau in figure 5) the relaxation tends to a power law form $\Gamma(t) \simeq t^{-\alpha}$, more or less independent of field and angle. At intermediate conditions, before the viscous regime has fully set in the relaxation is faster and changes form. In still lower fields where the signal on rotation is closer to a rigid response the relaxation becomes slower again.

In similar samples, non exponential relaxation of the remanent magnetization has been observed after switching off fields of up to 300 G [6]. The data was parametrized as $m = A - B \ln t$, and the analogy with spin glass was pointed out. In fact a wide class of disordered systems show non-exponential relaxation and suggestions have been made of mechanisms having very general validity, independent of the microscopic origin of the disorder [10]. In this sense the viscous behaviour and the relaxation we have observed is

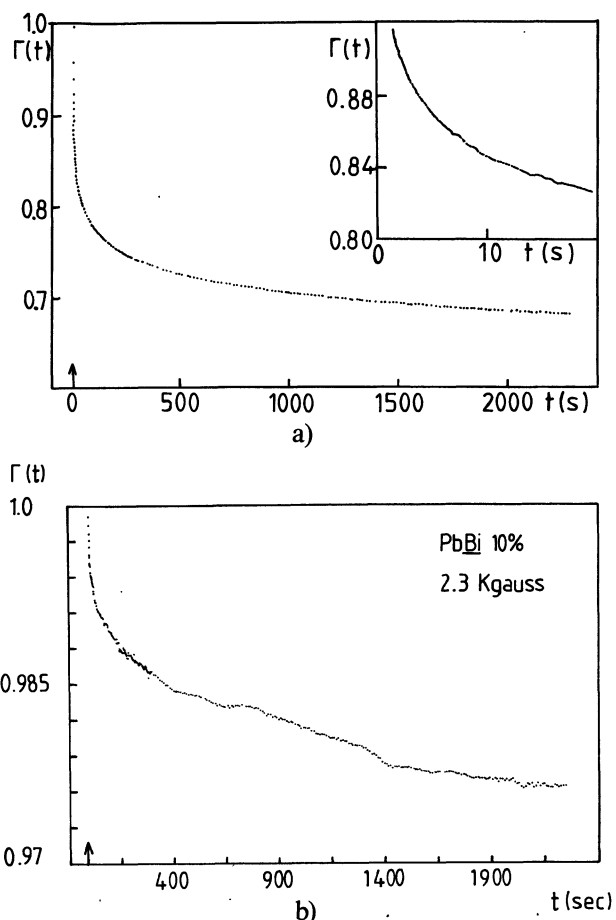


Fig.7.- a) Torque signal given by the La-Sr-Cu-O sample at 4.2 K, $H_t = 4$ kG, $\theta=5^\circ$ as a function of the time in seconds. Normalized to the signal immediately after turning the field. Inset : an example of jumps in signal observed in some runs. b) Torque signal for a sample of PbBi 10% at 4.2 K with $H_t = 2.3$ kG, $\theta=5^\circ$. Note the difference in vertical scale compared with figure 7a.

evidence that the flux line structure in these materials is "glassy", although we find curves which are not fitted by a logarithmic law. We can also remark that the power law decay form appears as the limiting "equilibrium" relaxation law observed below the glass transition in spin glasses. We are observing it in the limiting case where we have induced a fully viscous response in the sample but where we are far from equilibrium. The time scale for relaxation is considerably shorter than that for conventional type II superconductors, particularly when we note that we are far below T_c and H_{c2} . To illustrate this we have included torque data taken on an orthodox type II superconductor close to H_{c2} where the relaxation is observable but much less rapid than for the oxide. It is not yet clear if the rapid relaxation is due to an intrinsic weak pinning of the flux lines in these materials which would still be

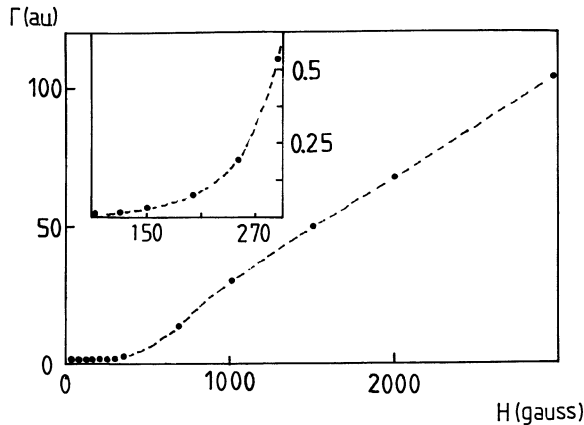


Fig.8.- Saturation torque intensity after ZFC procedure as function of H_t . Inset : closeup of the region $H_t = 100$ to 300 Gauss. The signal starts to rise steeply starting from somewhere near 150 Gauss.

present in single crystals for instance, or if it is related to the granular nature of the presently available samples.

Models have been presented for superconducting glasses based on a weak link network image [11] which corresponds to what we suggest our low field situation to be. However we still have evidence for typical "glassy" behaviour in high fields where the weak links are mainly broken and where the flux lines have penetrated into the grains, and hence where the model should no longer be applicable.

Data on Y-Ba-Cu-O samples which will be presented elsewhere show similar but rather slower relaxation behaviour.

5. Conclusion.

The results we have presented reinforce the granular superconductivity description for sintered samples of oxide superconductors, with a weak link network at low fields, and flux lines penetrating the grains irreversibly above a characteristic field. The torque measurements are particularly sensitive to behaviour we associate with onset of

flux line penetration into grains. For our sample this onset occurs at about 120 G at 4.2 K which can be considered as an effective H_{c1} for the grains. From magnetization measurements or from extrapolating the torque signal down linearly from high fields, we could estimate a value of about 350 G. This indicates that the "effective H_{c1} " has a range of values because of the disordered state of the sample.

In the "weak link" regime the flux line structure is only very weakly pinned against the field rotation. After flux penetration into the grains, the pinning is stronger but an almost completely viscous behaviour sets in for rotating fields of only ≈ 500 G, with the viscosity accompanied by a rather rapid non-exponential relaxation, tending to power law form $\Gamma \approx t^{-\alpha}$.

We can compare with magnetization relaxation results. Mota *et al.* [12] found that $dm/d\ln t$ in BaLaCuO increased rapidly with increasing field in the range 25-385 G. Our measurements (by a different technique so not directly comparable) show a relaxation rate $d\Gamma/\Gamma dt$ reaching a maximum at about 1 kG, followed by a lower plateau at higher fields. The maximum of relaxation seems to be associated with mean field penetration of flux lines into the grains.

Magnetic relaxation results so far reported are restricted to fields mainly in the weak link regime. The important fact that we observe rapid flux line creep after field penetration into the grains suggests that this is an intrinsic property for this material, indicating that the flux line pinning is weak. This would be severely detrimental to most practical applications. The superconducting glass models built up on a weak link description appear not to be directly relevant to this high field regime which nevertheless shows "glassy" characteristics.

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