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AC susceptibility measurements : a quality test for high T_c superconducting bulk materials.

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Abstract

Temperature dependent measurements of high T_c bulk material reflect the quality of the high T_c grains and the intergranular weak links. Besides, ac field dependent measurements give access to a quantitative quality label of the bulk material : its critical current density J_c. Herefore critical state models as the Bean and the Kim model are used. Our measurements on long orthorombic samples with different cross sections *a* x *b* show that the Kim model is the most realistic one. It leads to a *b/a* independent average value of J_c, which equals the J_c value of transport measurements and of magnetic SQUID measurements at low dc fields.

1. Introduction

An ac susceptometer is an interesting and efficient tool to study high T_c bulk material. Temperature dependent measurements do not only reveal the diamagnetic ac shielding of the material through steps in the real part of the magnetic susceptibility (χ'), but also the ac losses through maxima in the imaginary part of the susceptibility (χ''). Besides, the ac shielding and ac losses differ for the high quality grains and for the lower quality intergranular weak links in a bulk sample. So both fundamental elements of the granular high T_c structure can be studied separately.

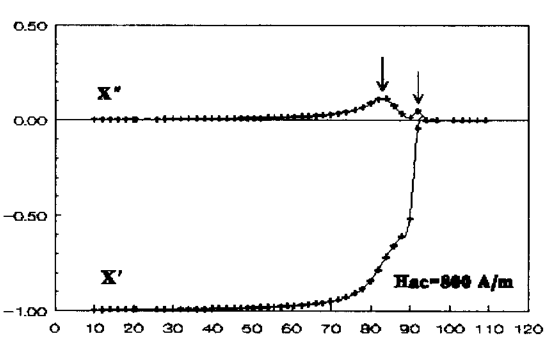

Figure 1 : Typical temperature dependence of the ac susceptibility of a $YBa_2Cu_3O_{7-x}$ bulk sample.

Figure 1 illustrates the general view of a $\chi(T)$ curve for a bulk high T_c material. The position and the steepness (width) of both χ' steps (χ'' maxima) reflects the quality of the grains (step and maximum at high temperature) and of the intergranular weak links (step and maximum at low temperature). The depth of the χ' step at high temperature is a qualitative measure of the volume fraction of the grains. However, the *quantitative* quality criterion by excellence of a high T_c bulk superconductor is its intergranular critical current density J_c. This can be derived from susceptibility measurements as a function of the ac field strength H_{ac}, with the aid of a critical state model.

2. J_c determination from χ

2.1 Critical state models

The use of critical state models to derive the intergranular J_c from ac susceptibility measurements is based on the following. The intergranular ac losses become maximum as soon as the ac field completely penetrates the sample, that is when the penetrated shielding current density times the penetrated volume becomes maximum [1]. In this critical condition critical state models assume the penetrated shielding currents to flow at a density equal to the critical current density $J_c(H_i)$, where H_i is the local internal field. The different critical state models can be divided according to the presumed $J_c(H_i)$ dependence. In the Bean model [2] J_c is independent of the local field. The Kim model [3] assumes a J_c inversely proportional to H_i, as is expected on the basis of the Josephson character of the intergranular weak links. Specifically Kim's $J_c(H_i)$ dependence looks like

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$$J_{c,Kim}(H_i) = k / (H_0+|H_i|) \quad (1)$$

Herein k and H_0 are temperature and sample dependent parameters, determined by the ac field at the χ'' (intergranular) maximum, the height of that maximum, and the dimensions and shape of the sample [4,5]. Both k and H_0 are sensitive to the exact values of χ''. Since these χ'' values are only approximately known, due to small uncertainties in the sample volume and the demagnetisation factors, some noise on the J_c values derived with the aid of the Kim model is to be expected.

In the Bean model the field independent J_c is given by

$$J_{c,Bean} = f\, H_{ac,max} / a \quad (2)$$

for long cylinder shaped samples with radius a, or for slab shaped samples with thickness $2a$. f is a shape dependent factor with a value between 0.75 and 1, and only slowly dependent of the exact values of χ'' [4]. Therefore the noise on the J_c values derived with the aid of the Bean model will be very small.

It is interesting to remark that equation (1) of the Kim model is completely equal to equation (2) of the Bean model as soon as $H_0 \gg H_i$ [4].

2.2 Experimental results

Our measurements, realized with a LakeShore Series 7000 susceptometer at zero dc field, have been performed on $YBa_2Cu_3O_{7-x}$ bulk samples. These samples were prepared from commercialy available YBCO powder, improved by the heat treatment developed in our lab [6], or by more traditional heat treatments. SEM-studies showed that all of our bulk samples are very homogeneous, containing only few and very small pores.

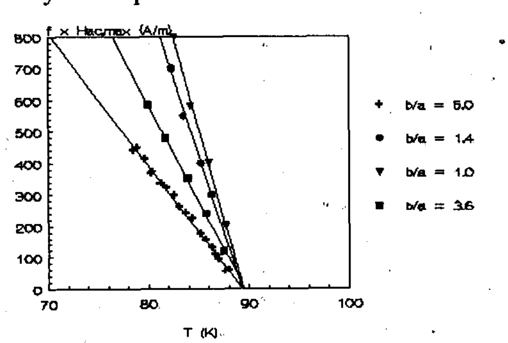

Figure 2 : The variation of f x $H_{ac,max}$ with the ratio b/a for long orthorombic $YBa_2Cu_3O_{7-x}$ bulk samples.

We shaped our samples as long orthorombic forms with cross section a x b and b = ± 1mm in all of our samples. As is very well known, the intergranular part of the susceptibility of long orthorombic samples is very sensitive to the b/a ratio [5,7]. This is best illustrated by the decreasing of the ac field at the intergranular χ'' maximum, $H_{ac,max}$, for rising b/a (figure 2). However, the $H_{ac,max}$ variation, and even the f x $H_{ac,max}$ variation is much less than linear with a, leading to a $J_{c,Bean}$ (eq. 2) increasing with rising b/a (figure 3).

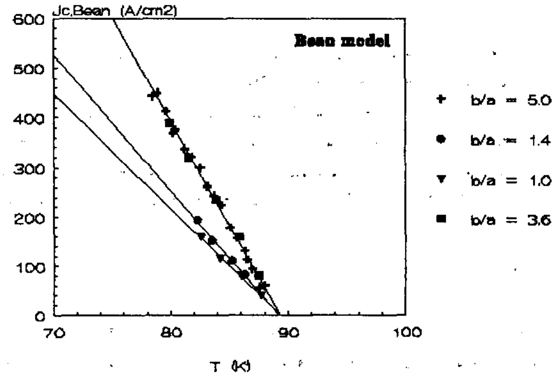

Figure 3 : $J_{c,Bean}$ for long orthorombic bulk $YBa_2Cu_3O_{7-x}$ samples with different b/a ratio.

The Kim model, on the contrary, does lead to b/a dependent k, H_0 and b/a dependent $J_{c,Kim}(H_i)$, but we find the $J_{c,Kim}$ value averaged over the changing local fields H_i in the sample, to be *b/a independent*. In other words

$$\overline{J_{c,Kim}} \approx [\, J_{c,Kim}(H_i{=}0) + J_{c,Kim}(H_i{=}H_{ac,max})\,] / 2 \quad (3)$$

seems shape independent (figure 4). As expected from our discussion of equations 1 and 2, the noise on the $J_{c,Kim}$ values is much larger than the noise on the $J_{c,Bean}$ values.

The results of figure 3 and 4 show that the real $J_c(H_i)$ dependence favorises the Kim model over the Bean model, as do the measurements of higher ac harmonics [8]. However, we remarked from the analysis of our different measurements that the H_0 parameter in equation (1) increases for rising b/a. For the highest b/a value (b/a=5) H_0 is even much larger than the maximum local field H_i, making equation (1) and (2), and the J_c values derived from them, equivalent. Figure 3 and 4 show indeed exactly the same results for b/a=5. In other words, for high b/a the Kim model evolves into the Bean model, meaning that the $J_c(H_i)$ dependence must be small.

Therefore, if J_c is aimed at, we prefer to work with thin slab samples i.e. long orthorombic samples with high b/a. Then, the easier Bean formula (equation 2) is correct and we can benefit from the lower noise in the derivation.

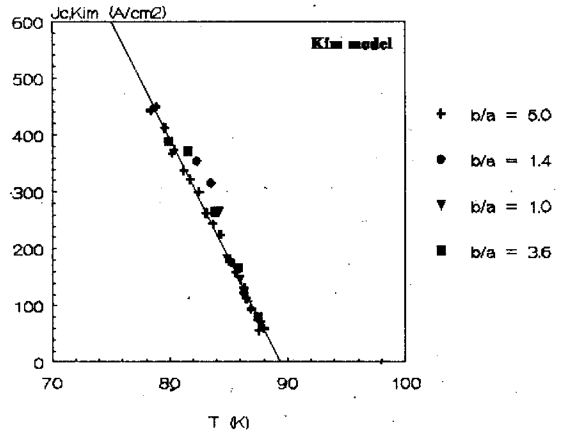

Figure 4 : Averaged $J_{c,Kim}$ for long orthorombic bulk $YBa_2Cu_3O_{7-x}$ samples with different b/a ratio.

2.3 Comparison to other J_c determinations

We compared the J_c values derived from our ac measurements on thin slab samples with J_c values determined by 4 point transport measurements at zero dc field, performed on the same bulk materials but the samples now in long orthorombic shape with b/a = 1. Figure 5 compares both J_c values for 3 different bulk materials prepared with a different heat treatment. From these and similar experimental results on even more $YBa_2Cu_3O_{7-x}$ bulk material, it is clear that the directly measured transport values of J_c coincide with the J_c values derived from our ac measurements. This is not very surprising since the transport J_c is also a field averaged critical current in the critical state, as is the averaged $J_{c,Kim}$ derived from the χ measurements. As our results showed $J_{c,Kim}$ to be b/a independent (figure 4), even the difference in shape of the samples is not important.

In figure 5c we also included a J_c value derived from magnetic SQUID measurements at very low fields. Also this value matches with the other measurements, as is expected from the correlation between magnetisation and ac susceptibility [4,9]. For SQUID measurements at high dc fields the derived J_c values are normaly much higher. Since high dc fields render the intergrain weak links unable to carry any more current, these high J_c values correspond to the J_c values of the grains and not the intergrain J_c.

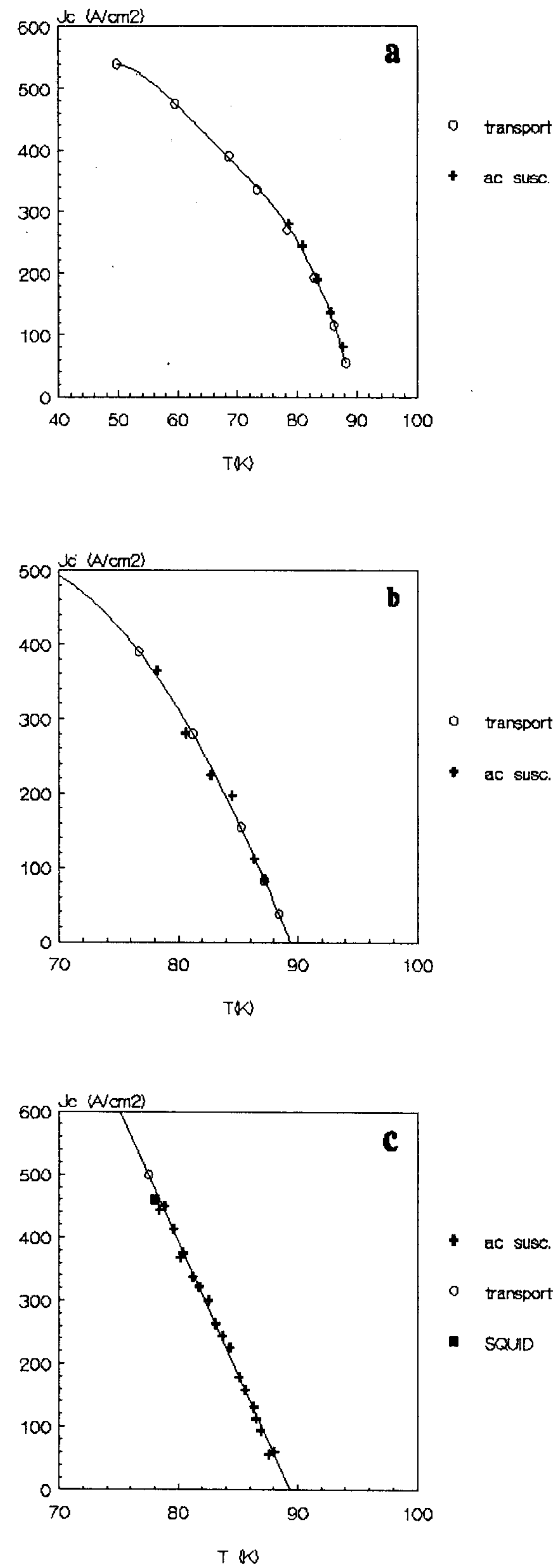

Figure 5 : Comparison of J_c determined by χ measurements and by transport and magnetic SQUID measurements. This for three different bulk samples.

3. Conclusion

Our experiments show that an ac susceptometer is a suitable tool to determine even quantitatively the quality of bulk high T_c material. We find the field averaged J_c values derived using the Kim critical state model to be independent of the cross section of a long orthorombic sample and to coincide with the J_c values determined from transport measurements or magnetic SQUID measurements at low fields. To facilitate the J_c derivation and to minimize the noise of the derived J_c values, χ measurements on thin slab shaped samples are recommended.

Acknowledgements

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