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The microwave absorption and flux trapping in highly oriented $YBa_2Cu_3O_{6.9}$ ceramics

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Abstract. Microwave absorption was measured in highly oriented ceramic samples of $YBa_2Cu_3O_{6.9}$ obtained by melt–quenching–melt growth solidification. Measurements on samples cooled down to the superconducting state in an external magnetic field permitted analysis of magnetic flux trapping. The character of the magnetic flux trapping has been found to be isotropic despite distinct structural anisotropy of the studied materials confirmed by the results of x-ray analysis. An attempt to interpret the results within the model of intergrain Josephson junctions has been made.

1. Introduction

Recently many authors have emphasized that the presence of intergrain or intragrain Josephson junctions in ceramic superconductors affects the properties of these materials [1]. Senoussi *et al* [2] proposed a description of the magnetic properties of ceramic superconductors within the model of a superconductor made of grains weakly coupled through Josephson junctions. The critical fields B_{c1j} and B_{c2j} have been studied and discussed by many authors [2–4]. The significant role of intergrain effects in ceramic superconductors has been confirmed by the results of critical current measurements [5]. Dulcic *et al* [6] discussed the influence of intergrain Josephson junctions in ceramics and of the junctions forming on defects in single crystals on the properties of high-temperature superconductors.

Because of the characteristic short coherence length ξ the presence of local inhomogeneities or defects leading to formation of weak links plays a much more important role in high-temperature superconductors than in classical ones. This fact has been confirmed by the model proposed by Deutcher and Müller [7] who analysed the superconductor–insulator–superconductor junction. The relation between the energy gap across the superconductor–insulator junction Δ_s and the energy gap characteristic for the whole sample is determined by the coherence length ξ, and the lattice parameter a.

According to Dulcic *et al* [6] for classical superconductors the energy gap at the link Δ_s affects the energy gap of the whole sample only in the close vicinity of T_c. When the barrier is very narrow its critical current takes a value close to the macroscopic one. Thus, in the proposed model the presence of defects and inhomogeneities will not exert significant influence on the properties of classical superconductors. In the case of high-temperature superconductors, as the coherence length is short the range of temperatures

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within which $\Delta_s(T)$ is much lower than $\Delta(T)$ is relatively wide. For $YBa_2Cu_3O_{6.9}$ it was assumed that $\xi_{ab}(0) = 34$ Å and $\xi_c(0) = 7$ Å. Under this assumption the temperature range within which $\Delta_s(T) \ll \Delta(T)$ is $(T_c - T)_{ab} \ll 1$ K and $(T_c - T)_c \ll 90$ K [6]. So the presence of intergrain and intragrain Josephson junctions essentially affects the properties of high-temperature superconductors over a wide temperature range.

The effects related to the presence of intergrain and intragrain Josephson junctions are particularly well manifested in the low-field microwave absorption [8,9]. Stankowski *et al* [10] considered a ceramic sample as a set of Josephson junctions interacting through the effect of superradiation. Under such an assumption the set of junctions could be treated as a single junction which permitted a phenomenological description of the line shape and the interaction of a superconducting sample with a microwave field. Blazey *et al* [11] explained the microwave absorption phenomenon in terms of dissipative fluxon motion in the intergrain region. Recently, Ramchandran *et al* [12] proposed a description of microwave absorption considering a set of resistively shunted Josephson junctions occurring in a ceramic sample. This permitted a qualitative description of most properties of microwave absorption observed in thin-film and monocrystal superconducting samples.

Measurements of microwave absorption have been widely applied in the investigation of superconductors in addition to the measurements of magnetic susceptibility and resistance. Microwave absorption has been used for the detection of transitions to the superconducting state in high-temperature superconductors [13, 14], classical, organic ones as well as fullerites [15]. This method has also been applied in high-pressure studies [16] and in the investigation of magnetic properties of superconductors [17]. Measurements of microwave absorption in superconductors cooled down to the superconducting state in a magnetic field permitted the analysis of magnetic flux trapping in these materials [18–20]. In our previous work [20] we presented the results of a ceramic $YBa_2Cu_3O_{6.9}$ sample with parameters $c = 11.67$ Å, $b = 3.82$ Å and $a = 3.89$ Å, characterized by high porosity of about 30% and a mean grain size of 10 μm. The sample was placed in a resonator of an EPR spectrometer. After cooling the sample in zero magnetic field the microwave absorption signal $\mathrm{d}P/\mathrm{d}B$ was symmetric and took a value of zero in zero magnetic field. When the superconducting sample was cooled in an external magnetic field of a few gauss, a distinct change in microwave absorption signal was observed, namely, the signal was shifted with respect to $B = 0$. This shift described as ΔB_{FC} corresponds to the mean value of the local magnetic field affecting the system of Josephson junctions and originating from the trapped magnetic flux. We have proposed an empirical formula describing the shift of the microwave absorption signal ΔB_{FC} as a function of the field B_{FC} in which the sample was cooled:

$$\Delta B_{FC} = \Delta B_{FC}^{max}[1 - \exp(-B_{FC}/B_0)]. \tag{1}$$

In this formula the process of flux trapping is characterized by two empirical parameters ΔB_{FC}^{max} and B_0 which stand for the maximum shift and coefficient of proportionality defining trapping effectiveness, respectively. This function has been found to describe the results obtained well.

In this paper we present the results of applying microwave absorption measurements in the investigation of magnetic flux trapping in oriented $YBa_2Cu_3O_{7-x}$ samples. In the investigations of critical fields and currents in single-crystal superconducting samples reported up to now, a strong anisotropy of these parameters was observed [21–24]. In the case of oriented ceramic samples of strong structural anisotropy the anisotropy of critical fields and currents was not so strong [25] because of the dominant role of intergrain effects.

These results have prompted us to study magnetic flux trapping in oriented $YBa_2Cu_3O_{7-x}$ samples, the aim of the study being to check the essential role of intergrain effects and to analyse the phenomena leading to microwave absorption. The results obtained allowed us to verify a new method of flux trapping investigation, where low-field microwave absorption has been applied.

2. Experimental method

The oriented ceramic $YBa_2Cu_3O_{7-x}$ samples were obtained by melt–quenching–melt textured solidification [25]. The precursor in the form of powder was obtained from the stoichiometric mixture of Y_2O_3, $BaCO_3$ and CuO through decarbonization at 860 °C. The product was homogenized and powdered in a platinum grinder at 1400 °C for 20 min in air atmosphere. Then the sample was ground, the obtained homogeneous powder was pressed into cylindric pellets, reheated for 20 min at 1150 °C, cooled to 1000 °C in 1.5 h and then from 1000 °C to 900 °C at a rate of 1 °C h^{-1}. The annealing was continued at 600 °C for 24 h in oxygen atmosphere. The sample's structure was controlled by a scanning electron microscope under magnifications of 260 to 2400. The orientation of grains as well as the parameters of an elementary cell were determined by x-ray diffraction analysis (XRD) on a Philips PW 1820 powder diffractometer using Cu Kα radiation. X-ray measurements in the plane of the basis of the cylindrical $YBa_2Cu_3O_{7-x}$ sample revealed only the presence of 001 type reflections.

This result permits the conclusion that the ceramic grains are arranged in parallel and the crystallographic axes of these grains are perpendicular to the base of the cylindrical sample, while the *a*–*b* planes of the grains are parallel to this base. The arrangement of grains was also observed under a scanning electron microscope [25]. The XRD investigation also permitted determination of the elementary cell parameters of the material: $a = 3.823$ Å, $b = 3.887$ Å and $c = 11.698$ Å. The oxygen stoichiometry was determined to be $YBa_2Cu_3O_{6.9}$ and the sample density to be 6080 kg m^{-3}.

The microwave absorption measurements of the $YBa_2Cu_3O_{6.9}$ sample were carried out on an S/EX Radiopan EPR spectrometer. The sample was placed in a TE_{102}-type microwave resonator at the maximum of the magnetic component of the microwave field. The sample was cooled by liquid nitrogen vapour. The sample temperature was measured by a copper–constantan thermocouple glued to the sample. All measurements were performed at 77 K. An external magnetic field generated in Helmholtz coils, which replaced the spectrometer magnet, was applied perpendicular to the magnetic component of the microwave field. The trapped flux of the magnetic field B_{FC} was parallel to the direction of the magnetic field produced in the Helmholtz coils. The value of the magnetic field intensity was controlled by the Hallotron milliteslometer within an accuracy of 0.1 G.

We also applied the second modulation of a frequency 100 kHz and amplitude of a few gauss. The residual laboratory magnetic field was compensated by an additional set of Helmholtz coils with an accuracy of 0.1 G.

3. Results and discussion

Changes in the shape of the microwave absorption were studied versus the magnetic field in which the oriented $YBa_2Cu_3O_{6.9}$ sample was cooled to the superconductivity state. For an appropriately high amplitude of second modulation the recorded signal was a derivative of microwave absorption with respect to the magnetic field dP/dB. After cooling the sample to the superconducting state in a magnetic field $B_{FC} < 0.1$ G (zero-field cooling) the dP/dB signal was symmetric and took a value of zero in zero magnetic field (figure 1(*a*)). When the sample was cooled to the superconductivity state (field cooling) the microwave absorption signal recorded in the still-present B_{FC} field was shifted with respect to the zero magnetic field by a value corresponding exactly to the B_{FC} field but in the opposite direction. This observation shows that on recording the dP/dB signal in the presence of the B_{FC} field, we have to apply an additional magnetic field opposite to the B_{FC} field in order to compensate for the effect of the B_{FC} field on the sample (figure 1(*b*)).

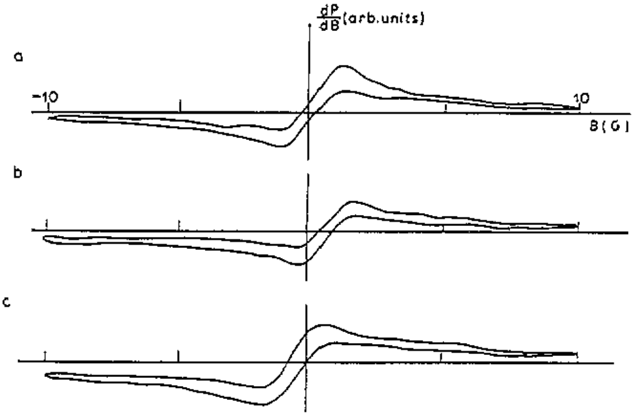

Figure 1. Microwave absorption signal dP/dB recorded after the sample has been cooled: (*a*) in zero magnetic field, (*b*) in non-zero magnetic field B_{FC} = 1 G not removed for signal recording, (*c*) in non-zero magnetic field B_{FC} = 1 G which was removed for signal recording.

The dP/dB signal recorded after the B_{FC} field has been removed is shifted with respect to zero magnetic field in the direction of the field (figure 1(*c*)).

Figures 2 and 3 illustrate dP/dB signals recorded after cooling the sample to the superconducting state in a B_{FC} field of different values. Figure 2 shows the signals recorded for the $YBa_2Cu_3O_{6.9}$ sample whose crystal axis *c* is parallel to the direction of B_{FC} while figure 3 shows the signals for the sample whose *c* axis is perpendicular to the B_{FC} field direction. Figure 4 shows the microwave absorption signals recorded for B_{FC} fields of different values in a sample obtained by grinding the textured ceramic to get grains of an average size of 0.07 mm.

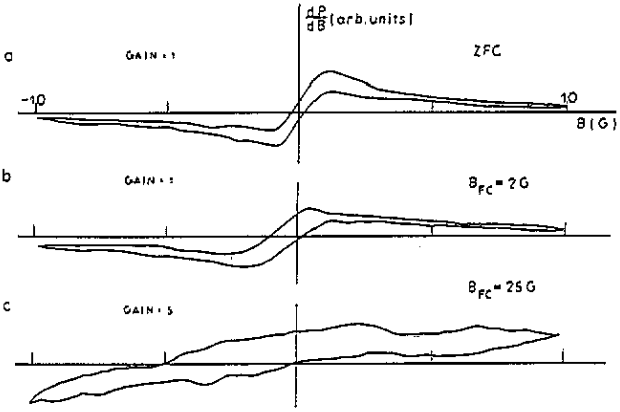

Figure 2. Microwave absorption signal recorded for cooling fields (*a*) 0 G, (*b*) 2 G and (*c*) 25 G for the crystallographic *c* axis of the sample parallel to the B_{FC} field.

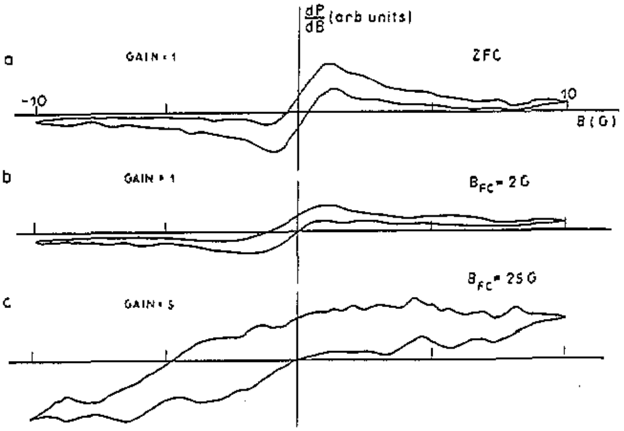

Figure 3. Microwave absorption signal recorded for cooling fields (*a*) 0 G, (*b*) 2 G and (*c*) 25 G for the crystallographic *c* axis of the sample perpendicular to this field.

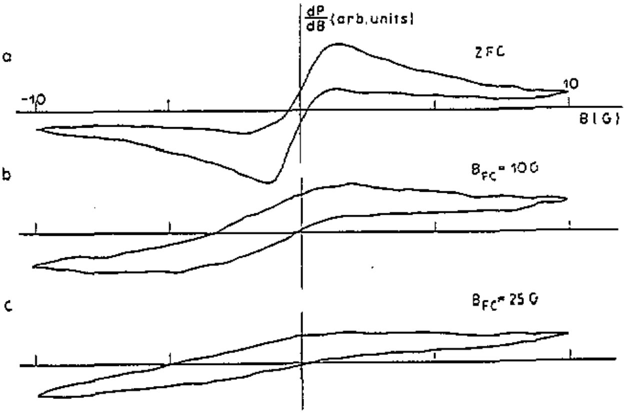

Figure 4. Microwave absorption signal recorded for cooling fields (*a*) 0 G, (*b*) 10 G and (*c*) 25 G in the powdered sample.

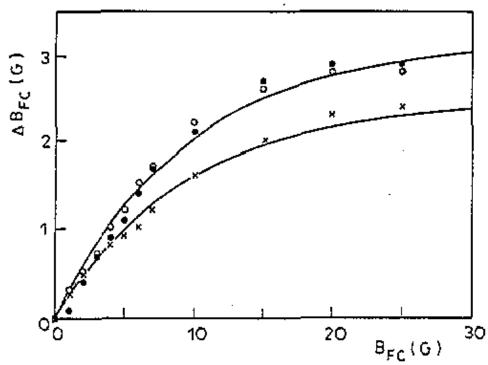

Figure 5. The shift of the microwave absorption signal ΔB_{FC} as a function of the cooling field B_{FC} for the *c* axis parallel to the B_{FC} field direction (●), perpendicular to it (○), and for the sample ground into powder (×). Full curves illustrate the function described by equation (4).

The dependence of the microwave absorption shift ΔB_{FC} on the field B_{FC} applied on cooling of the sample is shown in figure 5. The results presented in figure 5 were obtained for the sample whose *c* axis was parallel to the B_{FC} field direction, perpendicular to it and for the sample ground into powder. The dependence of the hysteresis width ΔB_{hyst} of the microwave absorption signal, defined as the distance in the scale of field between the points at which dP/dB becomes zero, on cooling fields was also measured (figure 6).

When a superconducting sample has been cooled in an external magnetic field B_{FC} of a

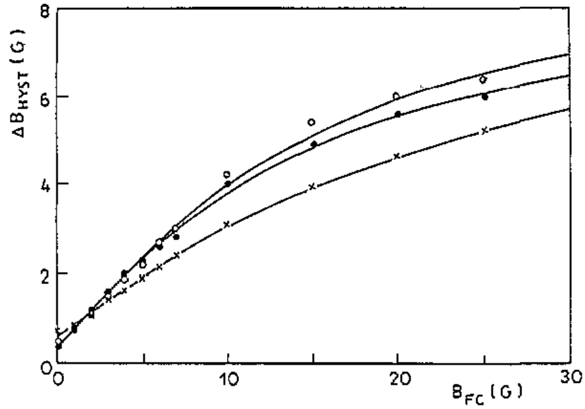

Figure 6. The hysteresis width as a function of cooling field B_{FC} for the c axis parallel to the B_{FC} field direction (●), perpendicular to it (○), and for the sample ground into powder (×). Full curves illustrate the function described by equation (5).

few gauss intensity, a clearly marked change in the microwave absorption signal $\mathrm{d}P/\mathrm{d}B$ is observed (figures 2–4). The whole signal is shifted with respect to zero magnetic field and broadened, and the width of the signal measured as the peak to peak distance increases. The signal whose derivative we observe is a superposition of all absorption signals due to groups of Josephson junctions in different local fields. During our experiment the distribution of the values of local field intensities changes because of flux trapping, and one can observe the changes in the line shape. The observed broadening of the $\mathrm{d}P/\mathrm{d}B$ signal is related to the fact that by freezing the sample in a greater magnetic field we get a greater distribution of the local field values. One can expect that for the highest cooling field of 20–25 G, the changes of line shape are due to some grains which begin to be penetrated. However, the intergrain flux trapping should give the anisotropic contribution to the shift of the $\mathrm{d}P/\mathrm{d}B$ signal, presented in figure 5. Such an effect has not been observed within the accuracy of our measurements. Also, the integral area of the absorption curve did not change as a function of the cooling field, as we reported previously [20]. This means that the total absorption remains the same up to 25 G, and it is difficult to see the intragrain penetration with the field and temperature conditions as applied in our experiments.

We suppose that one can expect intragrain penetration in the field range 50–200 G, corresponding to lower critical fields of monocrystals [21]. The variation of the background slope of the $\mathrm{d}P/\mathrm{d}B$ signal, which can be seen on figure 4, is due to the increase of the line width. When the maximum of the $\mathrm{d}P/\mathrm{d}B$ signal is shifted towards the higher field, for the highest cooling field, we observe only the central part of the $\mathrm{d}P/\mathrm{d}B$ signal. In consequence, the $\mathrm{d}P/\mathrm{d}B$ signal recorded for the highest cooling field seems to have an additional background slope.

The observed changes in the shape of the microwave absorption line depending on the field in which the sample was cooled are a consequence of magnetic flux trapping in a superconducting material. If by N_0 we denote the number of trapping centres in the sample and by N the number of trapped fluxons that comprise the populated trapping centres, then the increase in the number of trapped fluxons, $\mathrm{d}N$, will be proportional to the change in the value of the frozen field $\mathrm{d}B_{FC}$:

$$\mathrm{d}N \approx (N_0 - N)\mathrm{d}B_{FC}. \tag{2}$$

In this simple model we have assumed that one fluxon is trapped in one trapping centre. Introducing the coefficient of proportionality B_0 defining trapping effectiveness of a single centre, we get the equation:

$$\mathrm{d}N = (1/B_0)(N_0 - N)\mathrm{d}B_{FC} \tag{3}$$

whose solution is

$$N = N_0[1 - \exp(-B_{FC}/B_0)]. \tag{4}$$

Instead of the number of populated trapping centres, we can employ empirical values as $N_0\Phi_0/S_{eff} = \Delta B_{FC}^{max}$ where Φ_0 is a quantum of magnetic flux, S_{ef} is the effective area and ΔB_{FC} is the measured shift of the microwave absorption signal (figure 5). Then we can go back to equation (1) of this paper, first proposed in [20]. The experimental results shown in figure 5 are well described by the solution of (3), with the same parameter $B_0 = 10$ G for the powdered sample as well as for different orientations of highly oriented ceramics.

Assuming that the magnetic field flux penetrates the sample only via intergrain regions, the effective area can be expressed as $S_{eff} = SP$ where the sample porosity $P = 3\%$ and the sample surface area $S = 0.04$ cm^2. Given these values the number of trapping centres calculated from (1) is $N_0 = 2 \times 10^4$.

The hysteresis dependence on cooling field can be described by (1) with a small modification:

$$\Delta B_{hyst} = \Delta B_{hyst}^{max}(1 - \exp(-B_{FC}/B_0)) + \Delta B_{hyst}^{ZFC}. \tag{5}$$

The parameters ΔB_{hyst}, B_0 and ΔB_{hyst}^{ZFC}, are of the same order as parameters used in equations (1) and (4). However, it was difficult to fit all the experimental results with the same parameters. The empirical equation (5), written by analogy to (4), is difficult to interpret.

Anisotropic single crystals of $YBa_2Cu_3O_{7-x}$ have been the subject of many studies [21–24]. Measurements of the lower critical field at 77 K gave the values $H_{c1}(B\|c) = 200$ G and $H_{c1}(B \perp c) = 50$ G [21]. The values of the critical current in various $YBa_2Cu_3O_{7-x}$ single crystals differ by an order of magnitude depending on the crystal orientation [23, 24]. From these results one would expect a strong anisotropy of the properties of the oriented ceramic $YBa_2Cu_3O_{7-x}$ samples. On the other hand, the measurements of the critical current reported in [25] showed a very small anisotropy which was attributed to the dominant role of intergrain effects in ceramic samples.

It follows from the measurements reported in this work that the isotropic character of magnetic flux trapping in $YBa_2Cu_3O_{6.9}$ samples of clearly anisotropic structure proves the dominant role of intergrain effects in determining the sample properties in low magnetic fields. Up until now it had been difficult to distinguish between the intragrain and intergrain effects in the measurements of the low-field microwave absorption [6, 9, 20]. This work proves the dominant role of intergrain flux trapping in low magnetic fields. We have also verified microwave absorption as a method of investigating flux trapping.

4. Conclusions

The description of magnetic flux trapping in terms of population numbers of trapping centres is in good agreement with experimental results. In structurally anisotropic oriented ceramic samples of $YBa_2Cu_3O_{6.9}$ the magnetic flux trapping in low magnetic fields, studied by microwave absorption measurement, is clearly isotropic. Intergrain effects play a significant role in the mechanism of magnetic flux trapping in ceramic $YBa_2Cu_3O_{6.9}$ samples.

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