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1997 J. Phys.: Condens. Matter 9 1267

(http://iopscience.iop.org/0953-8984/9/6/013)

J. Phys.: Condens. Matter **9** (1997) 1267–1279. Printed in the UK PII: S0953-8984(97)73186-2

Particle size dependence of zero-field microwave absorption in powdered Bi–Sr–Ca–Cu–O superconductors

C Topaçlı† and E A Harris‡

† Department of Physics, Hacettepe University, Beytepe 06532, Turkey
‡ Department of Physics, University of Sheffield, Sheffield S3 7RH, UK

Received 28 March 1996, in final form 11 October 1996

Abstract. The non-resonant magnetically modulated microwave response measurements of powdered Bi–Sr–Ca–Cu–O samples using the conventional EPR spectrometer are presented. After cooling in a near zero magnetic field, all samples exhibited a sharp (about 12–37 μT) microwave absorption with applied magnetic field, superimposed on the widely observed and well explained broader minimum. The width of the absorption maximum is found to be dependent on the particle size. It becomes broader with decreasing particle size. The effects of particle size and field history on the peak are given in detail and some possible mechanisms to account for the observations are presented.

1. Introduction

The high-T_c superconductors exhibit interesting microwave absorption effects. There are several distinct features in the field-dependent microwave absorption that have been generally observed. One of these is strongest at temperatures immediately below T_c and occurs in all superconductors. It is observed at moderate magnetic field strengths and is attributed to the field-dependent resistivity felt by induced microwave currents near the critical point [1–3] and used as a T_c indicator. The other contribution has a very intense absorption minimum centred at zero field. Its field derivative has a peak-to-peak width in the range 0.1–10 mT, depending on the composition and granularity of material [4–7]. This signal has been attributed to the damped motion of fluxons within Josephson junctions where the effective viscosity is weak and the fluxons are highly mobile when a field is applied; microwave power is absorbed as fluxons are driven through the junction regions by microwave currents. Absorption through this mechanism can be intense for the high-T_c cuprate superconductors because of the large number of tunnelling junctions between the grains of these materials.

The other widely observed feature in the field-dependent microwave absorption is the hysteretic behaviour [8–11]. It has been attributed to a consequence of reversible and irreversible processes associated with a superconducting critical state with pinning and depinning of fluxons during a modulation cycle [11]. Later the concept of pinning and depinning of fluxons was given a better interpretation in terms of fluxon motion in an anharmonic pinning potential by Kessler *et al* [11(b)]. A model based on a loss mechanism in intergranular Josephson junctions has been proposed [12] and it was shown that it can account for both the hysterestic behaviour and the broad minimum.

Another feature observed in ceramics and also in crystal samples in field-dependent microwave absorption is a noise-like structure which is stronger than the instrumental noise.

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Its intensity is found to increase with decreasing temperature and to decrease with increasing field. It is also attributed to the flux jumps and phase slips in the complicated network of Josephson junctions [7].

The other feature is a very narrow absorption maximum centred at exactly zero field. A maximum was observed close to T_c at 91 K in single-crystal specimens of $YBa_2Cu_3O_y$ [13]. When the temperature decreased to below 89.2 K, it is replaced by a minimum. A similar maximum close to T_c was observed in high-T_c thin films [11]. This weak and rather narrow maximum appears to be superimposed on the widely seen minimum at lower temperatures. Some small but distinct zero-field absorption maxima have also been observed in ceramic superconductor samples [14–16]. They have been seen in $YBa_2Cu_3O_y$ and $ErBa_2Cu_3O_y$ fine powdered samples and studied in detail by Harris *et al* [17].

In this paper, measurements on a similar absorption signal which has been observed at low temperatures at exactly zero field in finely powdered $PbBi_2Sr_2Ca_2Cu_3O_y$ and $Bi_3Sr_2CaCu_2O_y$ samples using EPR instrumentation will be presented. It is an absorption maximum which is very narrow with a peak-to-peak width of the field derivative as small as 12 μT in ungraded $PbBi_2Sr_2Ca_2Cu_3O_y$ which is narrower than in $YBa_2Cu_3O_y$ [17] and differs from the broader absorption minima which have been seen widely by others. The width of this peak shows a particle size dependency.

2. Experimental methods

All our experiments have been carried out using a conventional EPR (Varian 4502) spectrometer operating in the X band. Temperature control is achieved by the use of an Oxford Instruments ERS9 helium gas flow cryostat. The stability of the temperature is obtained with a digital temperature controller using the thermocouple EMF to calculate the temperature of the helium flow and to adjust the heater current to maintain the set temperature. By the use of a flow controller and a digital temperature controller, the temperature of the helium flow can be maintained to within 0.5 K of the set temperature between 5 K and 300 K. A second thermocouple, which is in contact with the sample, placed inside the sample tube, was used to measure and control the real sample temperature. In all experiments, a microwave frequency close to 9.1 Ghz with 100 KHz field modulation and phase-sensitive detector were used to obtain the output as normally proportional to the field derivative of the microwave absorption. A low-frequency 40 Hz field sweep was also available, enabling narrow features to be displayed and saved on a digital storage oscilloscope.

The materials studied were $PbBi_2Sr_2Ca_2Cu_3O_y$ and $Bi_2Sr_2CaCu_2O_y$ supplied in powdered form by Merc Ltd of Poole, UK. As supplied, the particle size distribution ranged up to 50 μm with a mean value of about 10 μm. In order to investigate the effects of different particle sizes, a graded set of samples was prepared using a differential sedimentation method as described in [17]. The samples produced by this method had a graded range of particle sizes up to known maxima: less than 20 μm, less than 10 μm and less than 5 μm.

In most experiments the powdered material was suspended in xylene. This allowed us to eliminate any possible effects of conduction between the particles. Before cooling the sample, shaking the liquid to increase their separation was found to have no effect on any of results reported here except the broad minimum.

3. Results

The results of a typical non-resonant microwave absorption experiment on a sample of $PbBi_2Sr_2Ca_2O_y$ ground into a powder containing a range of particle sizes is shown in figure 1. This spectrum was obtained after cooling the sample in zero magnetic field from above T_c to about 6 K, but with no shielding of the magnetic field of the Earth; a low modulation (10 μT) was used and an applied field was slowly swept over a small range ($\pm$1.5 mT) on either side of zero field. The intrinsic noise level of the equipment was much less than the structure observed which consists of a random pattern of spikes giving a noise-like appearance. As seen from the figure, there are three different signals superimposed on each other and centred at exactly zero field: a very narrow maximum (Max 1), a slightly wider minimum (Min) and a broader maximum (Max 2). The sign of the signals were checked by comparing the shape with the EPR from the DPPH standard sample in the same cavity which is known as a g marker and gives a microwave absorption maximum. So, the sharp feature (Max 1) at exactly zero field is found to correspond to a maximum in the microwave absorption.

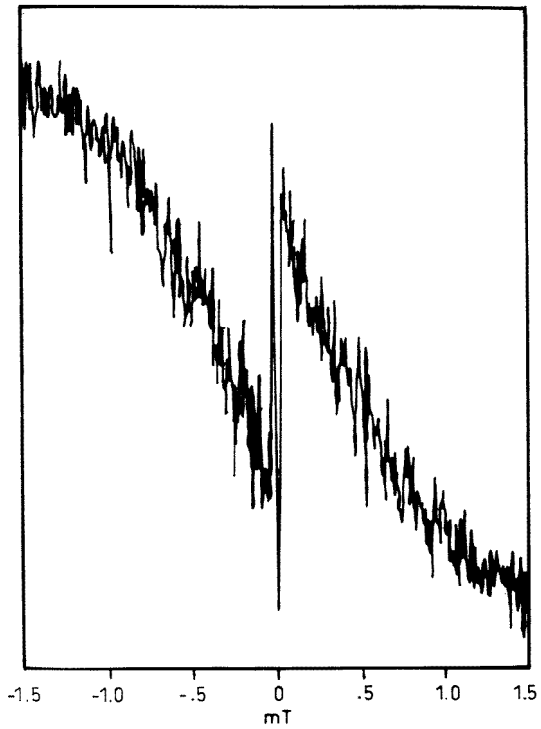

Figure 1. Typical non-resonant field derivative microwave absorption signal for an ungraded sample of $PbBi_2Sr_2Ca_2Cu_3O_y$ measured at 6 K with a modulation amplitude of 10 μT (a.u., arbitrary units). The signal shows three striking features (a broad maximum Max 2, a minimum Min and a very maximum Max 1) all of them superimposed with reproducible complex structure.

Another microwave absorption feature (Max 2) is broader with a 3 mT peak-to-peak width and is observed very strongly in ungraded material. This Max 2 was not seen in graded (less than 20 μm less than 10 μm and less than 5 μm) samples and it was again absent when the ungraded powder sample was suspended in the xylene to separate the

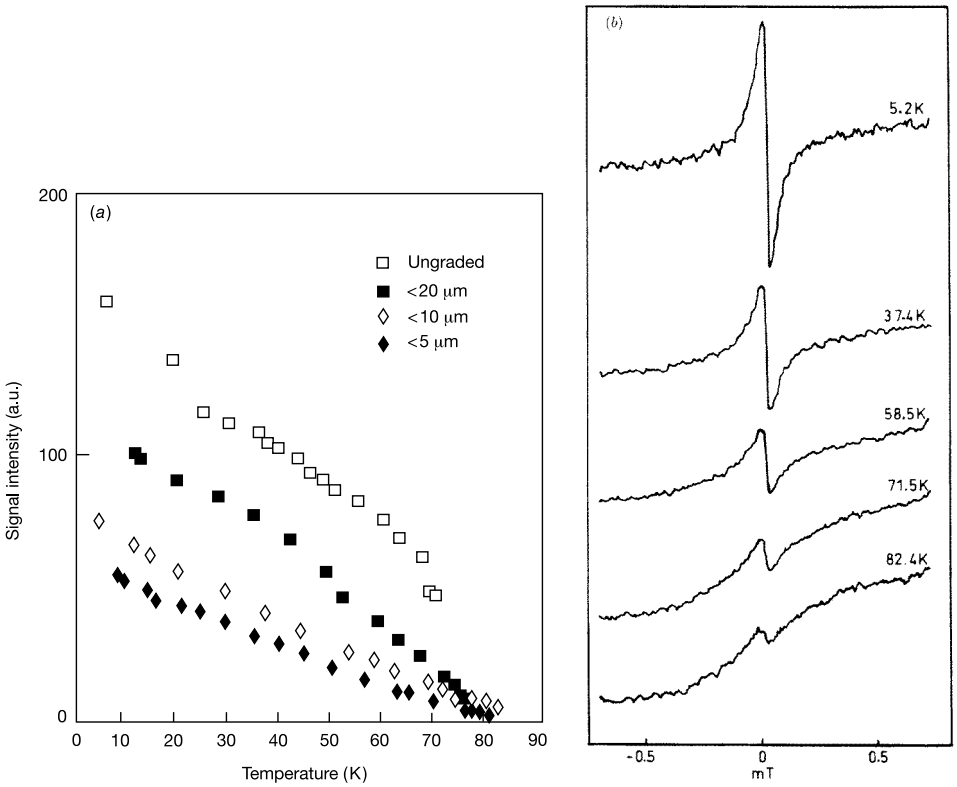

Figure 2. (a) Temperature dependence of Max 1 for the $PbBi_2Sr_2Ca_2Cu_3O_y$ samples for equal masses of different particle sizes. All measurements in the figure have been performed using the storage oscilloscope. (b) Field derivative of microwave absorption Max 1 in the $PbBi_2Sr_2Ca_2Cu_3O_y$ samples with particle size less than 10 μm at different temperatures.

particles. The intensity of Max 2 decreases with increasing temperature and is replaced by a minimum at 107 K. The intensity of this minimum increases initially and then decreases with increasing temperature; finally it disappears at a temperature close to the T_c-value of 117 K.

The other features shown in figure 1 are the noise-like structure and absorption minimum (Min) which is superimposed on Max 1 with 0.18 mT peak-to-peak width in this sample. The noise-like appearance of the signal in the low-field region is a structure composed of many narrow irregular components and is reproducible over small field sweeps. The structure is stronger in ungraded samples than in graded samples. The intensity of the spikes in the structure decreases with increasing applied field and temperature.

The observed minimum has properties in general agreement with those reported by others [11, 18]. For example, its width varies over the range 0.18–2.6 mT and depends on the grain structure and particle size. It decreases with increasing particle size. Its intensity depends on temperature and it disappears at 50 K in an ungraded sample but persists to nearly the T_c-value in graded samples.

The intensity of Max 1 decreases with decreasing particle size and with increasing temperature as shown in figure 2(a). In the ungraded sample the signal could only be measured up to 70 K because it is masked by Max 2 which becomes very dominant at this

temperature. It was observed to higher temperatures in graded samples but disappears at a temperature significantly below T_c. The shape of Max 1 for the particle size less than 10 μm at different temperature is shown in figure 2(b). The sample was cooled in zero field to the temperatures at which the spectra were taken; then the field was swept in the ± 0.5 mT range through the zero field with a low modulation amplitude (10 μT). This sharp Max 1 was observed clearly in all graded samples, but Max 2 could not be observed in these samples.

The width of Max 1 is found to be dependent on the particle size in $PbBi_2Sr_2Ca_2Cu_3O_y$ and $Bi_2Sr_2CaCu_2O_y$. As shown in figure 3, the width of the signal decreases with increasing particle size. While it is 12 μT for the ungraded sample, the widths are 25 μT, 31 μT and 37 μT in particle sizes less than 20 μm, less than 10 μm and less than 5 μm, respectively. The width of the signal was measured using the storage oscilloscope to increase the accuracy of the measurement. The modulation amplitude was kept fixed (10 μT) in all measurements to avoid broadening of the signal due to the modulation field. No particle size dependence was reported for $YBa_2Cu_3O_y$ and $ErBa_2Cu_3O_y$ [17].

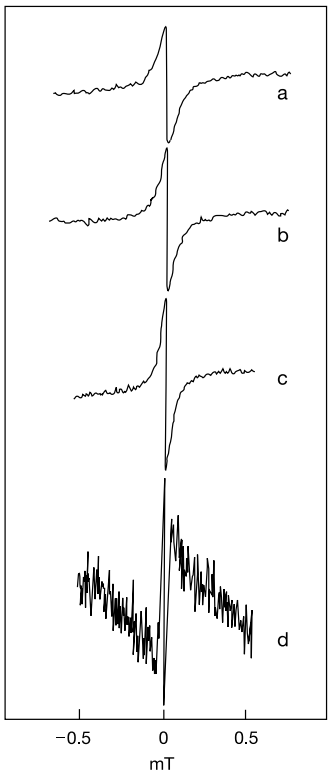

Figure 3. Field derivative of Max 1 for different sizes at 13 K: (a) ungraded; (b) less than 20 μm; (c) less than 10 μm; (d) less than 5 μm. The spectra are recorded with a low modulation amplitude (10 μT) after the samples had been cooled in zero field.

When the sample was not cooled in zero field, the signal size was found to be much reduced, as shown in figure 4(a). The data were obtained on the oscilloscope after cooling in a field through T_c to 15 K. In ungraded material the signal disappears at cooling fields greater than 0.5 mT, whereas for the smallest (less than 5 μm) particle sizes the reduction in the signal size was much less marked and it could be seen after cooling in fields of several

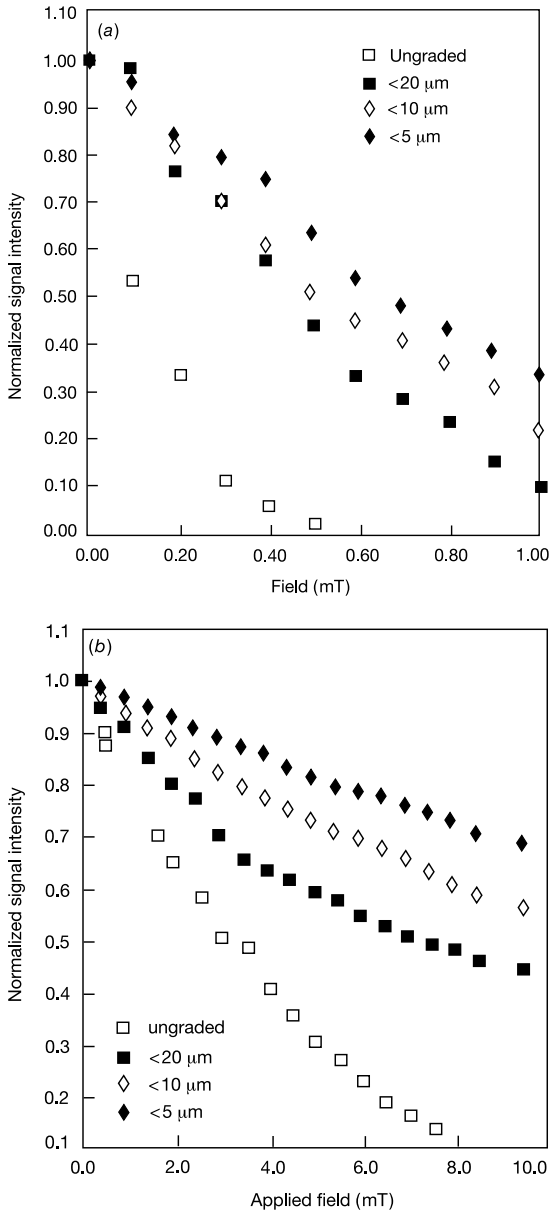

Figure 4. (*a*) The peak-to-peak intensity of the Max 1 signal in $PbBi_2Sr_2Ca_2Cu_3O_y$ as a function of applied field in which the sample was cooled through T_c to 15 K. (*b*) Reduction in the peak-to-peak intensity of Max 1 in $PbBi_2Sr_2Ca_2Cu_3O_y$ with application of a magnetic field at 15 K after the sample had been cooled in zero field.

milliteslas. As seen from the figure, the peak (Max 1) disappears at higher cooling fields for small particle sizes.

A similar reduction in peak-to-peak intensity of the zero field absorption was observed, when a field was applied after zero-field cooling (see figure 4(b)). It was observed clearly that this effect also depends on the particle size. The data in figure 4(b) were obtained by

first cooling the sample in zero field to 15 K. A field was applied for a few seconds and then reduced to zero, after which the peak-to-peak height of Max 1 was measured from the oscilloscope trace, using a low-amplitude 40 Hz field sweep. The reduction in the peak-to-peak intensity was irreversible and the signal could only be restored by heating above T_c and recooling in zero field. Comparing figures 4(a) and 4(b), it is clear that the application of an external field during cooling of the sample has a much greater effect on the absorption peak than the same field applied at low temperatures.

The peak-to-peak intensity of Max 1 is reduced more at higher temperatures than at lower temperatures in the same field as shown in figure 5(a). The data shown in the figure are obtained in the same way as for figure 4 at two different temperatures for different particle sizes. While the applied field required to halve the signal intensity after cooling in zero field if 3 mT at 15 K, the required cooling field is found to be much less (1.5 mT) at the same temperature for the ungraded sample. When the particle size is decreased, these field values are found to increase.

A similar reduction in the peak-to-peak intensity of Max 1 was observed when the same experiment as that for figure 4(b) was repeated at higher temperatures. In figure 5(b), data obtained at two different temperatures are shown for the ungraded and less than 10 μm particle sizes. As the applied field required to halve the signal intensity was 12 mT at 15 K, it was found to be 9.0 mT at 25 K for the particle size less than 10 μm. These field values were found to be lower with increasing particle size and temperature.

4. The results for $Bi_2Sr_2CaCu_2O_y$

Similar experiments were performed on $Bi_2Sr_2CaCu_2O_y$ ungraded and graded (less than 20 μm, less than 10 μm and less than 5 μm) samples. The broad Max 2 signal observed for ungraded $PbBi_2Sr_2Ca_2Cu_3O_y$ was not seen at all in these samples. When the results of $Bi_2Sr_2CaCu_2O_y$ were compared with the results of $PbBi_2Sr_2Ca_2Cu_3O_y$, the absence of the broad Max 2 signal is an important difference. The other features observed for $PbBi_2Sr_2Ca_2Cu_2O_y$ were also seen for $Bi_2Sr_2CaCu_2O_y$. There is a broad minimum (Min) whose width increases with decreasing particle size and a sharp maximum (Max 1) at zero field. Another difference in the $Bi_2Sr_2CaCu_2O_y$ samples was the induced sharp minimum observed at fields in which the sample was cooled. Max 1 could not be seen in the ungraded sample where it is hidden by this stronger sharp induced minimum which has been discussed in detail in [19]. When the sample was cooled while the field repeatedly swept through zero field at a low frequency of 40 Hz to eliminate the induced minimum, it was possible to see Max 1 for larger particle sizes but, in this case, the narrow Max 1 was distorted and broadened by this sweep field and modulation field. In graded samples, Max 1 has a similar width and properties to that in $PbBi_2Sr_2Ca_2Cu_3O_y$.

The cooling field and the applied field dependences of the peak-to-peak intensity of Max 1 in $Bi_2Sr_2CaCu_2O_y$ are shown in figure 6 for different particle sizes at 5 K. Comparing figures 4–6, it is clear that this maximum is more permanent in $Bi_2Sr_2CaCu_2O_y$ than in $PbBi_2Sr_2Ca_2O_y$ and is quenched at higher fields.

5. Discussion

In order to distinguish between effects due to microwave currents within individual particles and currents passing between different particles, the $PbBi_2Sr_2Ca_2Cu_3O_y$ sample was suspended in xylene. First, the intensity ratio of the Max 1 signal to the broad Min

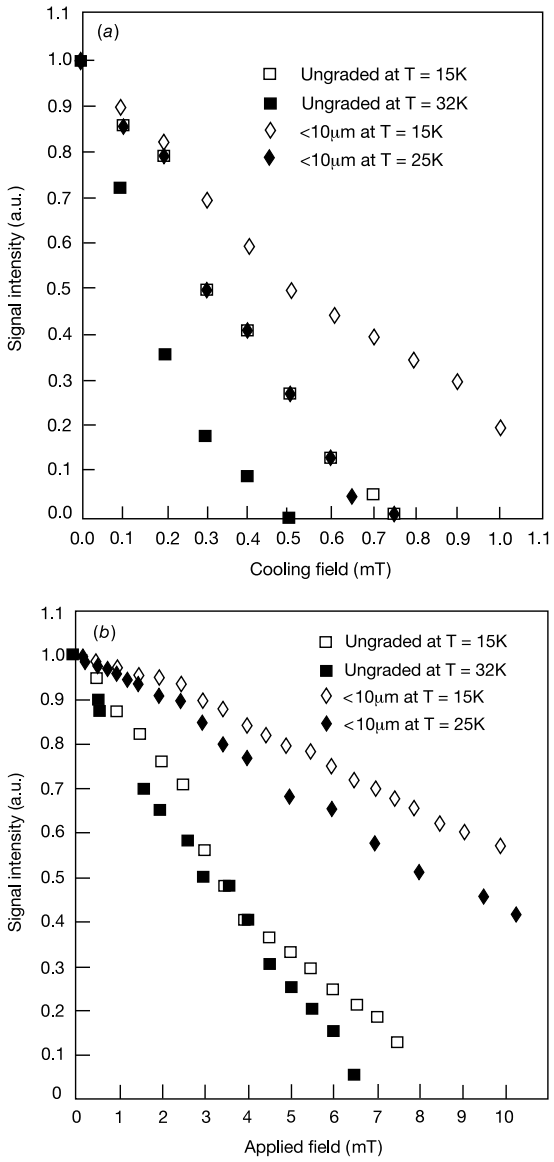

Figure 5. Reduction in the peak-to-peak intensity of Max 1 in $PbBi_2Sr_2Ca_2Cu_3O_y$ with ungraded and less than 10 μm particle sizes at different two temperatures (a.u., arbitrary units): (*a*) when the sample is cooled in different fields; (*b*) with application field after the samples had been cooled in zero field.

was found to be 2.0 ± 0.2 at 5 K, while all the particles were at the bottom of the sample tube. Then a small amount of xylene was put into the sample tube. The sample tube was shaken well to separate the particles and cooled very quickly through T_c to the same temperature (5 K). In this case, the intensity ratio of the Max 1 to the Min was found to be 11.0 ± 0.2. In the later case, the intensities of both Max 1 and Min were found to decrease. The ratio of the reduction in the peak-to-peak intensity of Max 1 was found to be 2.2 ± 0.2 while the ratio of the reduction in the peak-to-peak intensity of Min was found to

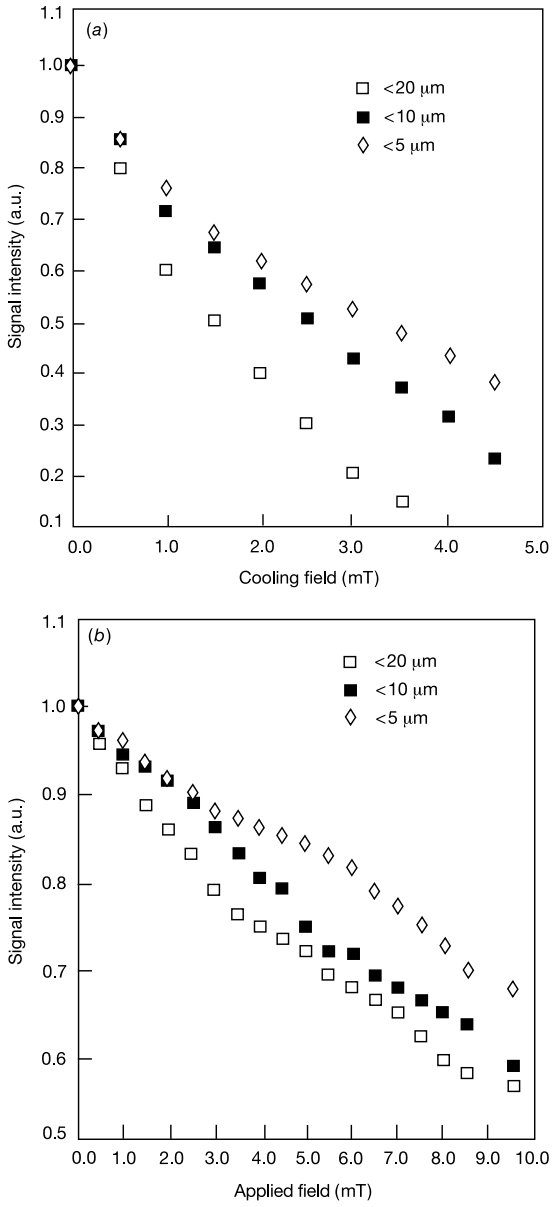

Figure 6. Reduction in the peak-to-peak intensity of $Bi_2Sr_2CaCu_2O_y$ at 5 K (a.u., arbitrary units): (*a*) after cooling in different fields; (*b*) with application of a magnetic field after cooling in zero field.

be 14.0 ± 0.2. The reduction in the intensity of the Max 1 signal in the xylene is attributed to the reduction in the sample amount in the middle of the cavity. When the xylene is added to the sample tube, it separates particles and raises some of the particles from the bottom of the tube. This reduces the sample amount contributing to the absorption and also reduces the sensitivity of the cavity. So a similar reduction in the intensity of the Min signal is expected, but it is seen that the intensity of the Min signal is reduced much more

than expected. This strongly suggests that the absorption Min signal is contributed by the currents flowing between particles and also the most important result that Max 1 is not in any way affected by these currents.

The broad Max 2, which is observed only in the ungraded $PbBi_2Sr_2Ca_2Cu_3O_y$ powder sample, and the sharp Max 1, which is seen in all graded powder samples, have not been reported widely in the literature. The Max 2 signal also disappeared when the ungraded sample was suspended in the xylene to separate the particles. This strongly suggests that the contribution to this signal comes from the larger particles and the currents which flow between the particles. A possible mechanism for Max 2 could be the dissipation by the normal current component driven by the out-of-phase electric field associated with supercurrents in loops containing weak links, which are coherent in the zero field. This mechanism would be affected by particle dilution and could account for Max 2.

The Max 1 signal has been attributed to a quadruple phase slips in loops containing weak links by Harris *et al* [17]. They have also mentioned another mechanism for the zero-field microwave absorption which has been given a similar mechanism by Afanas'ev *et al* [20]. They considered an ensemble of loops of various areas with a distribution of flux; so the net current will be composed of interfering terms. Only in the case of zero field will the currents be coherent. They assumed that the particles have surface currents which flow along the paths with an area in which there are Josephson weak links. They argue that the Josephson current flowing along such a path in a static field superimposed on an alternating field induces a maximum power dissipation at zero field.

The two main quantities of interest are the size and width of the peak. Harris *et al* found that the predicted intensity of the absorption peak should be small compared with the experimental peak. Additionally, the linewidth is related to the field which produces one flux quantum in average cross-sectional area ϕ_0/S, where S is the area of the loop and ϕ_0 the quantum of flux. We have found that the width of the peak gets smaller with increasing particle size, as predicted in this model. Afanas'ev *et al* also reported a similar width dependence on the particle size and a similar decrease in the intensity of the peak with applied field measurements for $YBa_2Cu_3O_y$, as we have shown here for $PbBi_2Sr_2Ca_2Cu_3O_y$ and $Bi_2Sr_2CaCu_2O_y$. These results encourage the view that this mechanism could account for the zero-field absorption maximum.

Nebendahl *et al* [21] have also reported a zero-field absorption maximum for $YBa_2Cu_3O_y$. They have attributed this maximum to a splitting of the characteristic 'diffraction'-like pattern. Their idea is based on a drastic change in the critical current versus field curves depending on the orientation of the measuring field which was either parallel or perpendicular to the junction plane. This explanation could also account for zero-field absorption, since the Josephson junction width could depend on the particle size. This could be examined by taking splitting as in the diffraction pattern into account in the model developed in [22]. We shall not give details of the model here, since the model and the modulation, particle size and temperature dependences of characteristic low-field hysteresis were discussed in detail in [22]. As is well known, the low-field microwave absorption in the intergranular junctions has a field-sweep independent (reversible) part, and a part (irreversible) which changes sign when the field sweep direction is reversed. The latter part yields a hysteresis, while the first part give the derivative (reversible) signal.

In order to obtain the field-sweep independent part, we have to assume a field dependence of the critical current of the Josephson junction. The most elementary approach is to take the envelope of the familiar diffraction pattern which seems reasonable for the granular

powder sample, since they include many Josephson junctions:

$$I_c = I_{c0}\frac{\sin(\pi H/H_0)}{\pi H/H_0} \tag{1}$$

where H_0 is the field for which the first flux has penetrated into the junction. The filed H_0 depends on the size of the junction, i.e. $H_0 = \Phi_0/S$, where Φ_0 is the flux quantum and S is the effective area of the junction exposed to the external magnetic field [12]. The effective field dependence could be taken as the envelope of equation (1), when some distribution in the junction size is assumed. However, as noticed by Nedendahl *et al* [21] and analysed in detail by Miller *et al* [23, 24], the critical current versus field curve of a junction cooled in low fields with the field perpendicular to the junction plane shows a drastic change depending on the orientation of the measuring field which was either parallel or perpendicular to the junction plane. As a consequence of their discussion, a splitting of the characteristic 'diffraction'-like pattern can occur at zero field. When such a splitting is taken into account in the developed model [22], the obtained microwave absorption signal (just the derivative part of whole low-field microwave absorption) is shown in figure 7. For comparison, we have included a split diffraction pattern, which yields the obtained broad minimum and narrow maximum signals, in the inset of figure 7.

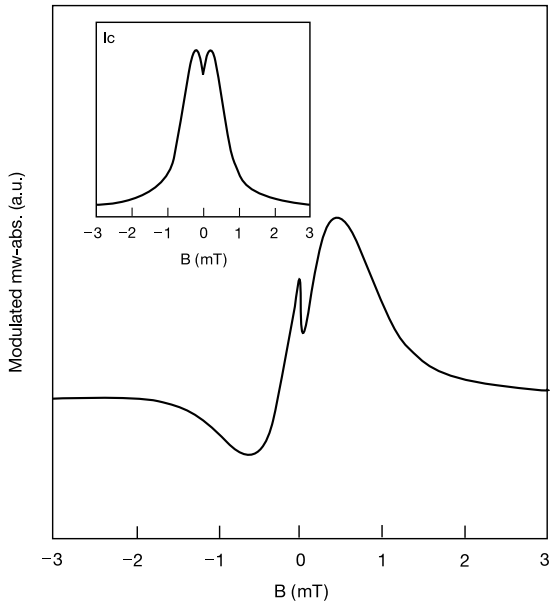

Figure 7. Modulated microwave absorption signal of the granular sample obtained with the modification of the model described in [22] (a.u., arbitrary units). The inset demonstrates the alteration of the diffraction pattern discussed in the text.

The splitting of the 'diffraction' pattern of a single junction due to vortex pinning for a field perpendicular to the junction plane as discussed in [23, 24] could depend on the junction size. In a granular superconductor the junction size is determined by the grain size. If we assume that the grain size depends on the particle size, it is possible to obtain the particle size dependence (or grain size dependence) of the zero-field line (Max 1) shown in figure 3. It is expected that this assumption is applicable at least for small particle sizes, although we are aware that it is questionable for larger particle sizes. In figure 8, we have

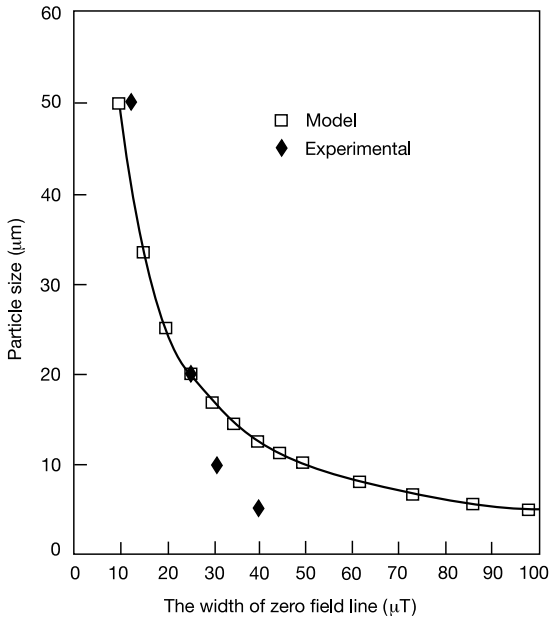

Figure 8. Particle size dependence of the zero-field microwave absorption. Using the experimental data regenerated from figure 3, the solid curve obtained from the model shows the grain-size-dependent splitting in the Josephson junction critical current.

given the calculated width of the zero-field line (Max 1) according to Josephson junction size (solid curve). The experimental results for the particle size are also shown in the figure. The width of the zero-field line becomes less when the grain size is increased, as seen from the theoretical curve. The experimental data also show similar behaviour, but there is a small discrepancy at very small particle sizes. The results demonstrate that the grain size does not get smaller as much as the particle size does, but a detectable change in the grain size is still shown when the particle size decreased. The results also show that the grain size depends on particle size somehow, but it is not linear.

In conclusion, the microwave absorption at low-fields, and especially the absorption maximum observed at zero field, can be explained when the proposed alteration of the critical current of Josephson junctions is taken into account.

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