Microwave and ac power absorption and ac susceptibility measurements of the high-T_c superconducting Tl-Ba-Ca-Cu-O system near zero field

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The low-field dependence of electrical power absorption and ac susceptibility–temperature measurements are performed on three ceramic superconductors of the $Tl_2Ba_2Ca_{L-1}Cu_LO_{4+2L}$ family using modulated and direct microwave and ac methods. Below a critical temperature all samples exhibit a large increase in ac and microwave absorption near zero field, a Meissner effect, and a diamagnetic shielding phenomenon. The microwave signal recorded with a modulation technique has three components, out of which, one with hysteretic character is mostly the product of the normal cores of intragrain fluxons. The observed deviations of the signal from the real absorption behavior are explained. The measured ac susceptibility reflects mainly the moving magnetic flux vortices induced by intergrain Meissner and diamagnetic shielding currents. Our results suggest that the variation of magnetic field induces at low and microwave frequencies the same flux exclusion and penetration processes characterizable macroscopically mostly through the change in diamagnetic surface impedance.

INTRODUCTION

Among many other high-T_c superconductors, a thallium-based family has been discovered.[1] The common feature of all these compounds found so far is the existence of planes formed by copper and oxygen atoms. Although much experimental and theoretical research has been devoted to these materials, no clear understanding of the superconducting mechanism has yet emerged.

Superconductive properties can be usefully studied at microwave frequencies.[2-18] All high-T_c superconducting oxides investigated have exhibited a derivative signal with giant peak at very small external magnetic fields. dc and ac magnetization measurements also permit determination of many of the key parameters like penetration depths and coherence lengths.[19-26] Magnetization with hysteretic behavior has been observed in the V-Ba-Cu-O compounds.[27-37] In this paper we present some results of the observed hysteretic ac field absorption and ac magnetization of the Tl-Ba-Ca-Cu-O family, in low magnetic fields.

EXPERIMENTAL DETAILS

We studied four sintered ceramics with the following nominal composition and diamagnetic onset temperature: (a) $Tl_1Ca_1Ba_1Cu_1O_{4.5}$, $T_c=100$ K; (b) $Tl_2Ca_1Ba_2Cu_2O_8$, $T_c=120$ K; (c) $Tl_2Ca_2Ba_2Cu_3O_{10}$, $T_c=111$ K; and (d) $Y_1Ba_2Cu_3O_7$, $T_c=94$ K. The number of Cu atoms in the chemical formula of the Tl family indicates the number of CuO_2 layers in the primitive unit cell.[38,39] The samples were prepared by conventional methods. All compounds have disordered grain structures with 5–10-μm grain size.

By modulated microwave measurements the sample (typically 20 mg), introduced in a quartz dewar, was placed in the cavity of an X-band EPR spectrometer, at the maximum of magnetic field component. This component is perpendicular to the dc magnetic field H_0 produced by a commercial (JEOL) electromagnetic.

In order to obtain very low and negative field intensities with this magnet, four coils out of the total of eight were connected in one direction and three coils in the opposite direction. The remaining coil was biased by an external constant-current source. Magnetic field modulation technique at 100 kHz was applied.

The H_0 field was linearly varying from a negative amplitude ($-\max H_0$) to a symmetrical positive amplitude ($+\max H_0$) and back to $-\max H_0$. At signal recordings the field sweep time was 5 min. After the microwave current rectification the remaining 100 kHz frequency signal was amplified and phase-sensitively detected. Consequently the output of the installation was in close connection with the field derivative of the absorbed microwave magnetic energy.

For direct observation of microwave absorption we used the above-mentioned installation but without modulation, monitoring directly the variation of microwave cavity reflected power by recording the leakage current (a measure of imbalance of the microwave bridge) as a function of field. For low-frequency ac field absorption and transmission measurements a transformer with superconducting ceramic core was used. All absorption investigations were made at liquid-nitrogen temperature.

For ac susceptibility–temperature measurements a commercial rf bridge with a usual pickup coil was employed. Using a bridge we measured the inductivity before and after the insertion of the sample plate (with $1\times5\times7$ mm^3 typical dimensions) into the solenoid. The temperature varying between 293 and 77 K was determined by a Fe-Constantan thermocouple in contact with the sample.

No correction for demagnetization and for porosity of the sample was done. No field dependent absorption was observed in the normal state of compounds.

MODULATION AMPLITUDE DEPENDENCE OF MICROWAVE SIGNAL

Like the La, Y, and Eu based superconducting oxides,[6] all specimens that we investigated exhibit large increase in absorption on application of a low magnetic field. A typical derivative spectrum for the (c) sample is shown in Fig. 1. On inspection we see that the structure of this signal permits the identification of at least three distinct components: (A) a field independent base-line shift (B) an almost linear part, and (C) a curve very similar to a resonance line with considerable asymmetry. Because these components exhibit different dependences from modulation field amplitude (Fig. 2, Fig. 3), the resulting signal at different modulation intensities takes apparently different shapes (Fig. 4).

For usual amplitudes the C component is approximately 100 times greater than the two others, so the contribution of the A and B components can be neglected by first approximation. But for small modulation intensities A and B are non-negligibles. Not only the C component's peak height but also the distance of this peak from the zero-field crossing (ΔH_{op}) increase withe the ac modulation field (Fig. 5).

INFLUENCE OF FIELD SCAN AMPLITUDE ON THE MICROWAVE SIGNAL

The samples were cooled to liquid-nitrogen temperature in the "zero field" of the electromagnet. Next the field was scanned a few times through the positive peak and negative peak in forward and backward directions. Only after that was the derivative of microwave signal recorded. In all samples investigated the peak position ΔH_{op}, where the absorption changes most rapidly, shifts to higher fields if the amplitude of the dc magnetic field scan ($\max H_0$) increases (Fig. 6). There is a residual linewidth with decreasing scan amplitude. The data demonstrate that the linewidth (ΔH_{op}) depends strongly on the sample composition, basically on the number of Cu-O planes in unit cell. Not only the peak position but also the peak height as well depends on the scan amplitude and sample composition (Fig. 7).

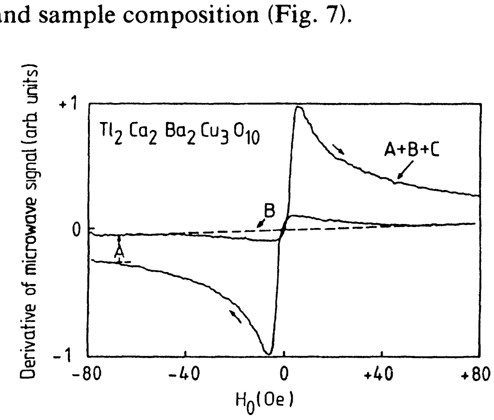

FIG. 1. Representative microwave signal related to field derivative of absorption in $Tl_2Ca_2Ba_2Cu_3O_{10}$ using $h_m = 1$ Oe modulation amplitude, at $T = 77$ K, by forward and backward field scan.

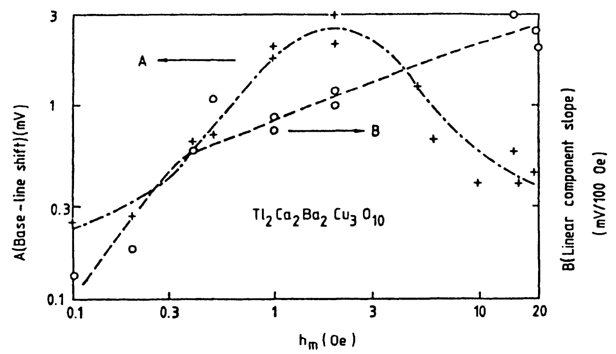

FIG. 2. Modulation amplitude dependence of the base-line shift (A) and mostly linear (B) component of the derivative signal from $Tl_2Ca_2Ba_2Cu_3O_{10}$. Lines are guides for the eye.

MICROWAVE ABSORPTION SIGNAL

All our microwave measurements discussed until now were excuted with modulation and phase-sensitive detection technique; consequently the derivatives of the microwave signals were plotted. We recorded the microwave absorption signal also directly (without modulation) with respect to the magnetic field strength (Fig. 8).

The microwave magnetic absorption curves like the derivative microwave signals for all our samples present, in a certain field interval, a memory effect with pronounced hysteretic behavior in forward and reverse magnetic field scan. While usually the phase-sensitive detection in the presence of field modulation does not yield directly the field derivative of the absorption when the absorption is hysteretic[11] we shall compare two signals obtained with and without modulation for the same sample.

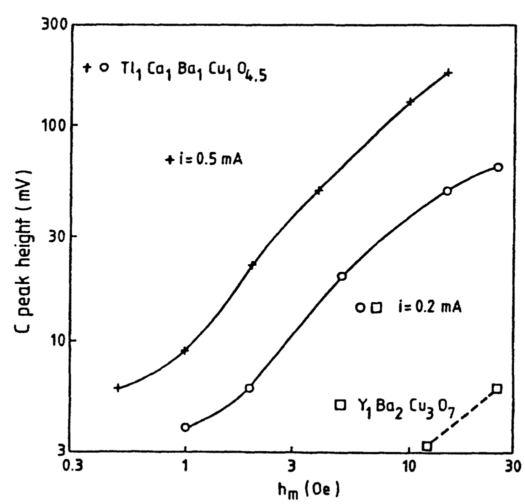

FIG. 3. Modulation amplitude dependence of the peak height of derivative microwave signal from $Tl_1Ca_1Ba_1Cu_1O_{4.5}$ and from $Y_1Ba_2Cu_3O_7$ by two microwave intensities ($i = 0.5$ and 0.2 mA).

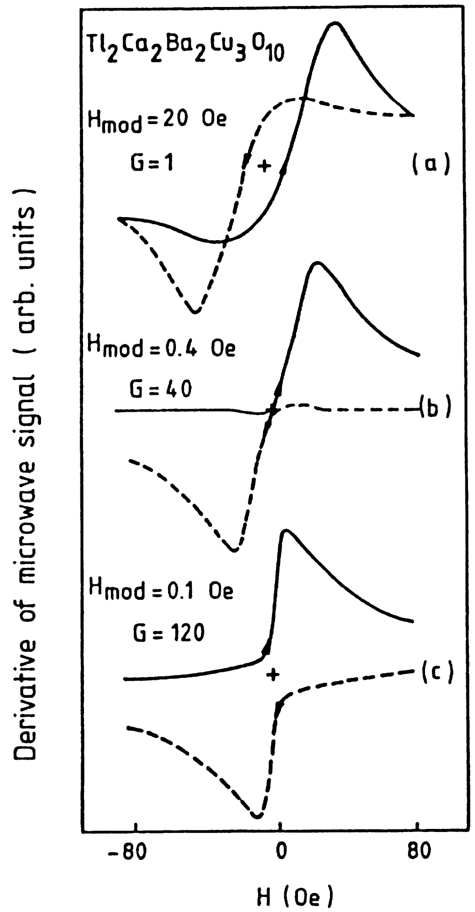

FIG. 4. Derivative of microwave signal from $Tl_2Ca_2Ba_2Cu_3O_{10}$ for different modulation amplitude: (*a*) $h_m = 20$ Oe receiver relative gain $G = 1$; (*b*) $h_m = 0.4$ Oe, $G = 40$ and (*c*) $h_m = 0.1$ Oe, $G = 120$.

Because by direct absorption measurements our experimental method was not able to reveal the change in fluxons polarity by magnetic field polarity changes, we redesigned the (*a*) absorption curve from the Fig. 8 using negative signal values for negative magnetic fields to indicate the polarity of fluxons involved. We plotted this

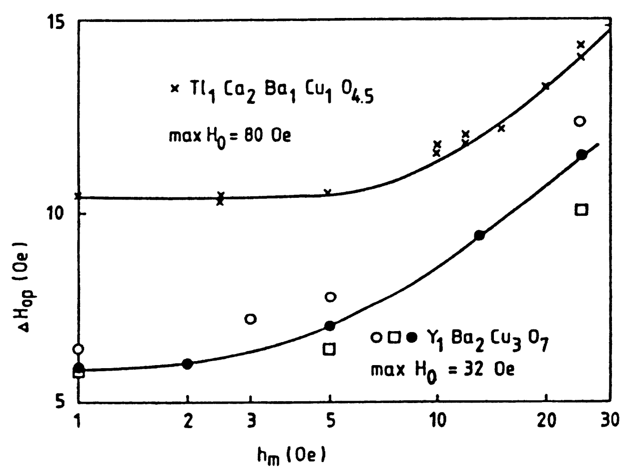

FIG. 5. ΔH_{op} half linewidth (the field where the peak of derivative signal appears) vs modulation amplitude for $Tl_1Ca_1Ba_1Cu_1O_{4.5}$ and for three different Y based samples.

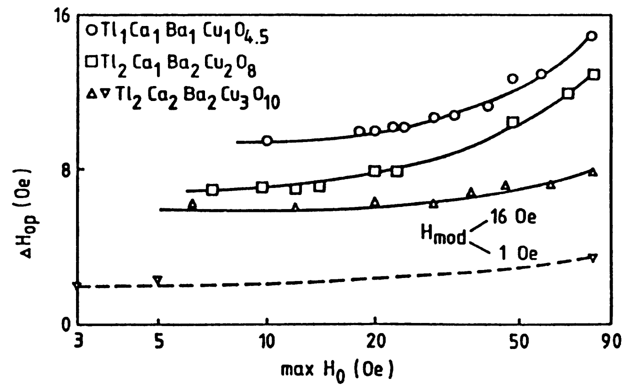

FIG. 6. The ΔH_{op} half linewidth vs scan field amplitude (max H_0) observed in three Tl compounds, for $h_m = 16$ Oe and in $Tl_2Ca_2Ba_2Cu_3O_{10}$ for $h_m = 1$ Oe too.

curve together with the derivative signal obtained with lockin phase technique under the same instrumental conditions using a modulation field of only ~ 10 mOe (Fig. 9). Absorption curves above and under the horizontal axis of Fig. 9 mean absorption in connection with positive and negative polarity fluxons. On the same figure we plotted also the derivative of the absorption signal obtained by graphical differentiation of the absorption curve (*c*) using positive values by forward and negative values by reverse magnetic field scan.

One can see that for decreasing fields the experimental (*b*) and the graphically calculated derivative curves (*c*)

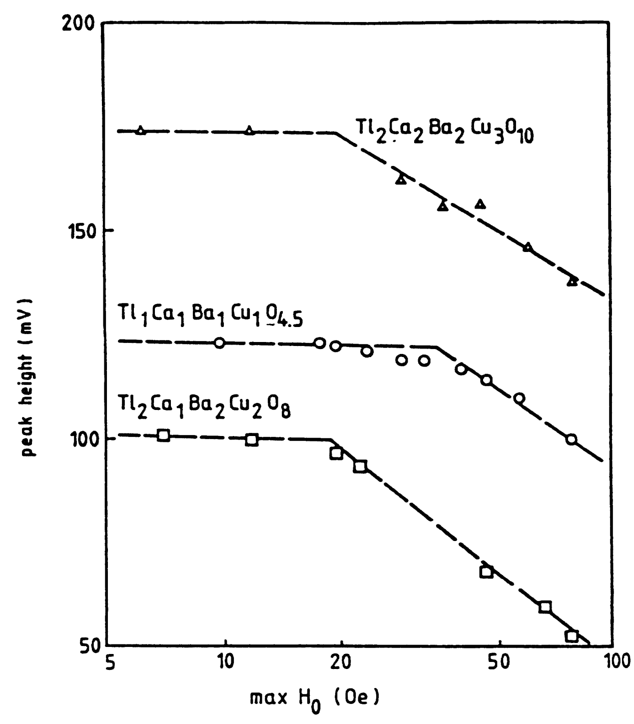

FIG. 7. The peak height vs scanning dc field amplitude (max H_0) for three Tl compounds at $h_m = 16$ Oe. The straight lines are guides for the eye.

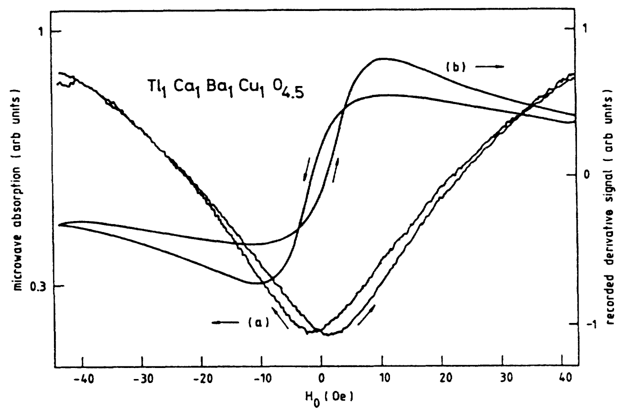

FIG. 8. Magnetic field dependence of microwave absorption signal (*a*) and the field derivative of microwave signal obtained with modulation ($h_m = 5$ Oe) and phase sensitive detection (*b*) in $Tl_1Ca_1Ba_1Cu_1O_{4.5}$.

coincide well enough, but for increasing fields, the experimental one containing the signal component called B too, is greater than (*c*). We neglect now the component A because, being independent of H_0, it does not change the form of curves.

The component B after us appears in the process of modulation in the following way. If the small modulating field (h_m) is superposed on the relative large direct field (H_0), as h_m varies from zero to $+h_{\max}$, the absorption first traces out the same curve as plotted with the direct field alone. As h_m decreases from $+h_{\max}$ to $-h_{\max}$, the absorption follows the upper branch of a minor hysteresis loop, and the slope of the curve decreases. When h_m increases from $-h_{\max}$ to $+h_{\max}$, the absorption follows the downer branch of the minor loop and the direct component of the absorption increases. Finally, the system attains a steady state in which the slope of the curve decreases while the direct component of the absorption increases.

We suppose that the difference between the measured and calculated derivative curves in the case of small enough modulation intensities, is that the component B is proportional with the reversible (incremental) diamagnetic permeability which progressively increases with increase of H_0. Extracting the components B from the (*b*) derivative curve plotted after phase-sensitive detection, the real derivative curve of absorption may be reconstructed. The component $B = (d)$ in a restricted field interval may be roughly approximated as linear function of H_0.

By normal modulation intensities the derivative signal is more distorted and therefore cannot be used directly to reconstruct the derivative curve of absorption. The dc signal component (A) appears like a transient response (step) with approximately $-\frac{1}{3}A$ amplitude by turning out and with $-\frac{2}{3}A$ amplitude by turning on the field sweep.

For large modulation amplitudes the sign of the magnetic field change is determined mostly by the ac modulation field and the resulting signal is a composite of signals from opposite sweep directions. Thus the dependence of the signals form versus modulation amplitude may be understood.

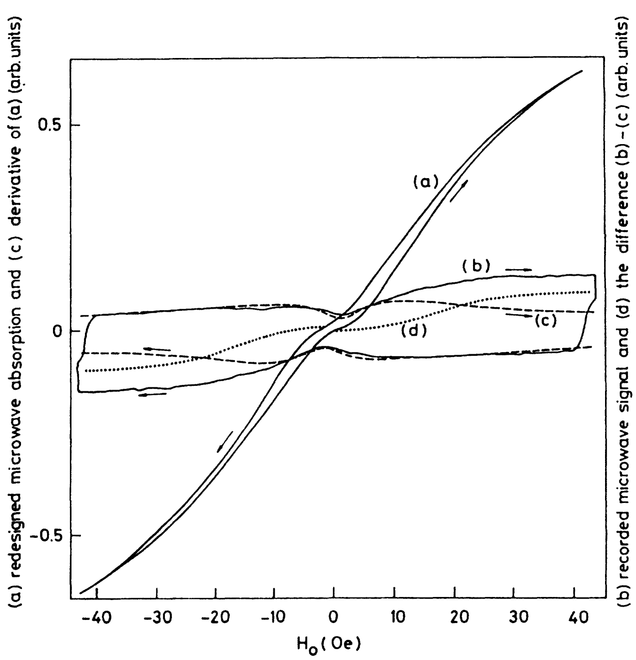

FIG. 9. Magnetic field dependence of (*a*) the redesigned microwave absorption signal, (*b*) the recorded derivative signal obtained with weak modulation ($h_m = 0.01$ Oe) and phase-sensitive detection, (*c*) the derivative of (*a*) obtained by graphical differentiation, and (*d*) the difference between (*b*) and (*c*).

POWER ABSORPTION AND TRANSMISSION AT LOW-FREQUENCY FIELDS

To clear up the role of microwaves in magnetic energy absorption of high-T_c superconductors, a second independent low-frequency routine was used. An elementary transformer having two coils wound around a superconductor core was constructed. A sine-wave 100-kHz frequency field was created by driving the primary winding with an appropriate supply. The secondary voltage was measured either with a digital multimeter (DMM) or after amplification and lockin phase detection was recorded as a function of the external magnetic field H_0.

Typical results for the Y and Tl based samples illustrated in Figs. 10 and 11 indicate the equivalence of the used two methods. All samples exhibit hysteretic behavior but interestingly the loops by the Tl based *c* sample close at zero field too (Fig. 12).

The frequency-dependent measurements (80 Hz, 360 Hz, 100 kHz, 10 GHz) reveal only slight changes in signal shape. This fact could be the consequence of their common origin, the diamagnetic behavior of the samples.

TEMPERATURE DEPENDENCE OF ac SUSCEPTIBILITY

The ac magnetization measurements are used as an independent third routine for material characterization. The temperature dependence of the real part of the com-

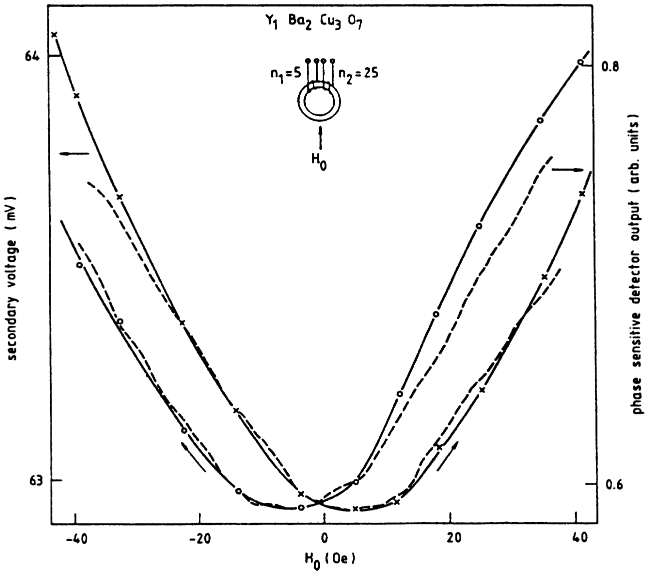

FIG. 10. Magnetic field dependence of secondary voltage of the $Y_1Ba_2Cu_3O_7$ superconductor core transformer measured with DMM and recorded after phase-sensitive detection. Both methods give the same result. Two, about $H_0=0$ symmetrically placed minima appear.

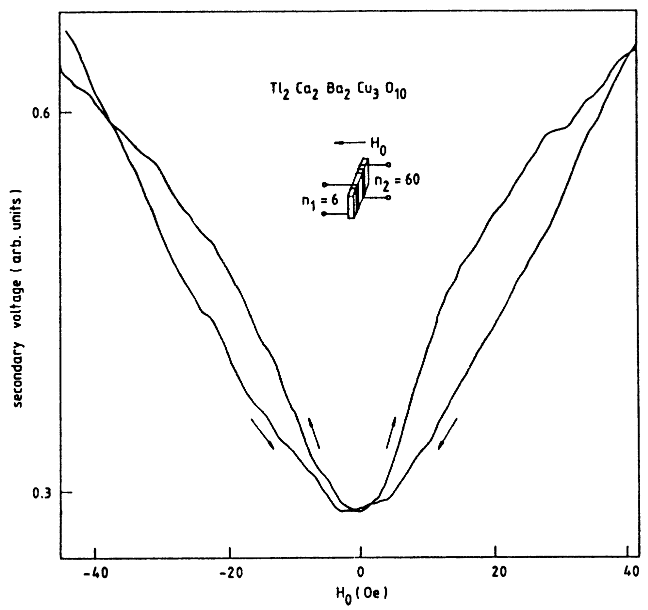

FIG. 12. The secondary voltage vs applied dc magnetic field recorded with phase-lock technique by the superconductor $Tl_2Ca_2Ba_2Cu_3O_{10}$ core transformer. For both scanning directions the minimum appears at $H_0=0$ (birdlike shape).

plex ac susceptibility was first determined by cooling the sample from room temperature to 77 K and after that by warming up to room temperature. The obtained data (Fig. 13) show the consequence of a partial flux exclusion (Meissner effect) and diamagnetic shielding below the critical temperature. With the magnitude of diamagnetic susceptibility the screened volume increases with decreasing temperature. As the temperature increases $-4\pi\chi$ decreases in magnitude. In all these experiments the curves, obtained by cooling down and by warming up conditions, are very different. This particularly is important since the presence of irreversibility would imply the existence of surface barriers of flux trapping.

We observed in all samples the magnetic field dependence of ac susceptibility (Fig. 14). The magnetic field decreases the volume from which the magnetic flux is ex-

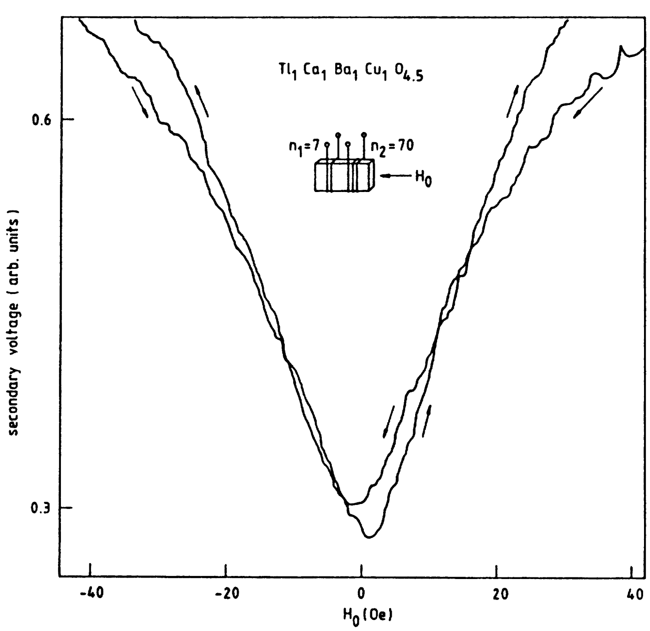

FIG. 11. Secondary voltage vs applied dc magnetic field recorded after phase-sensitive detection for the superconductor $Tl_1Ca_1Ba_1Cu_1O_{4.5}$ core transformer.

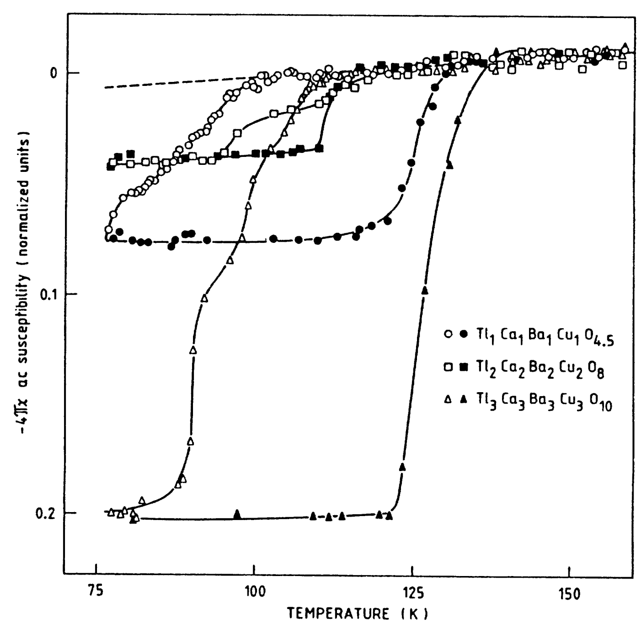

FIG. 13. Temperature dependence of the ac susceptibility in three Tl based compounds. Open symbols indicate decreasing solid symbols increasing temperatures. The magnetic field exclusion starts at 100 K (○), 120 K (□), and 111 K (△).

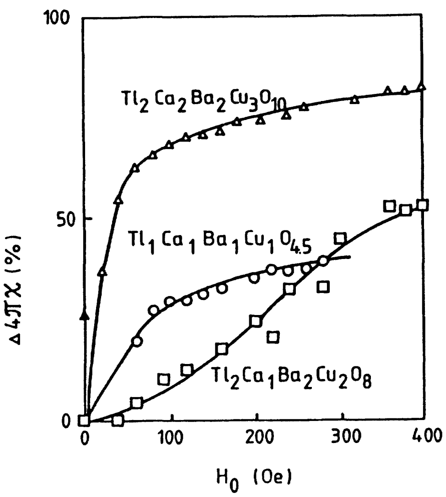

FIG. 14. Magnetic field strength dependence of the ac susceptibility at 77 K in three Tl based compounds. The solid triangle represents the remanent susceptibility modification for one of the samples. The comparison of Figs. 14, 13, and 7 indicates a connection between the field dependence of ac susceptibility and field amplitude dependence of microwave signal.

cluded. We also found that the susceptibility and magnetic penetration depth given in the topology of our experiment by $\lambda \cong (1-4\pi\chi)\frac{1}{2}d$, where d is the plate thickness, depend strongly on the composition, the crystal lattice structure, and the preparation conditions too. Repeating the thermal cycle many times, the measured parameters may reflect a modification on the structure of the samples.

BULK AND POWDER PROPERTIES

Comparing the results of measurements executed on the bulk sample and after that on the same sample grinded into powder, two kinds of behavior can be distinguished. Grinding destroys most of the intergrain connections and intergrain currents. Consequently the ac susceptibility decreases at least 10 times. The microwave

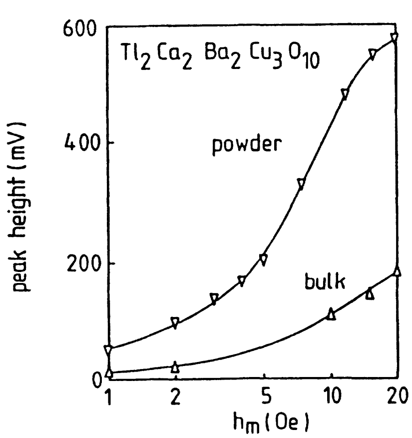

FIG. 15. Magnetic field dependence of microwave derivative signals peak height in bulk ceramic (△) and in powder (▽) $Tl_2Ca_2Ba_2Cu_3O_{10}$.

absorption data obtained with modulation and phase-sensitive detection for the bulk and powder sample are shown in Fig. 15. It can be clearly seen that the signal for powder is about 3 times greater.

These data demonstrate unambiguously that the diamagnetic Meissner effect and shielding is in connection with the coupling of the grains while microwave signal is due mostly to the grain surfaces. The supposition that there are two parts of materials which coexist in these superconductors, superconducting grains and boundary materials, is supported by our experimental results too.

DISCUSSION

By our absorption experiments the results obtained by both methods point out the usefulness of the magnetosurface impedance notion (Z) introduced by A. M. Portis *et al.*[40] We demonstrate in this paper that the absorption arises not only from microwave but from low-frequency ac fields too. So the surface impedance introduced originally for microwaves may be used generally, from low to microwave frequencies.

At the superconductor core transformer the two ac circuits are inductively coupled through the surface impedance. In this case Z may be called as the complex impedance of mutual induction $Z = iX = i\omega M$ where M is the mutual inductance of the two circuits. While the magnetic field produced flux exclusion causes a change in M, the amount of coupling between the two coils can be varied by application of an external dc magnetic field (parametric excitation).[41]

At microwave measurements the Q factor of the cavity proportional with the real part of complex impedance was monitored. The combination of results from microwave absorption, ac excitation, and susceptibility measurements confirms that, conforming to the expression of real and imaginary parts of the magnetosurface impedance, the principal macroscopic parameters responsible for field dependence are first the permeability and second the resistivity: $Z = f(H) = f(\mu, \rho^{1/2})$ with not equal importance. In the framework of the microscopic model corresponding to Z, ac magnetic field dependence occurs due to the changing number of normal electrons appearing in the pair breaking process at the surface of the sample or in the cores of moving flux vortices. Z is proportional to the number of fluxoids and this is proportional to the magnetic field in a hysteretic manner.

The derivative of microwave signal recorded after phase-sensitive detection is related to absorption and reveals at least three different components. The component called A may be attributed to the scanning fields. This field component $(\partial H_0/\partial t)$ may have also a contribution to the vortex displacements. Starting the field scan, not only the free but also the weakly pinned fluxons are involved. Stopping the field scan, the free fluxons continue their movement but without the weakly pinned part. Component B is a consequence of modulation. Extracting the components A and B from the recorded signal, we can obtain the derivative of absorbed microwave energy. This is demonstrated by graphical comparison of recordings obtained with different techniques. In general it is not impossible to deduce the absorption curve from correct results obtained with distortionary modulation technique even if this phenomenon is hysteretic.

Our data on thallium based superconductors with grain structure show sharp ac diamagnetic susceptibility drop with decreasing temperature. The diamagnetic susceptibility appears as a consequence of flux exclusion (Meissner effect) produced by a mostly intergrain current vortex structure. The vortices are in dynamic equilibrium configuration, and their density changes with the field strength. These intergrain currents flowing across weak-link boundaries persist up to 110–120 K and dominate the global diamagnetic behavior of the ceramics in dc and ac conditions. Above 110–120 K they start to decay and the shielding currents circulate predominantly in the individual grains. The exhibited thermal hysteresis of the susceptibility is due to the pinned flux. The amount of trapped flux may be correlated with the peak height of microwave derivative signal (Fig. 7 and Fig. 13) and depends on magnetic field (Fig. 14).

Grinding the ceramic in powder, the measured ac susceptibility becomes very small ($-4\pi\chi \cong 0.02$). This diminution of the real part of complex ac diamagnetic susceptibility indicates the partial disappearance of the surface intergrain screening currents.

On the contrary, the microwave signal of powders is greater than that of ceramics (Fig. 15). These facts could be interpreted too in the framework of the magnetosurface impedance of high-T_c superconductors with two coexisting parts, grains and boundary materials.

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