# Interaction of AC magnetic field with the vortex system in highly anisotropic Bi-2223 superconductor; intercalation effects. Comparison with Bi-2212

D. Shaltiel [a,*], H.-A. Krug von Nidda [b], A. Loidl [b], C.T. Lin [c], B. Liang [c], T. Kurz [b], B. Bogoslavsky [a]

[a] *The Racah Institute of Physics, The Hebrew University, Givat Ram, Jerusalem 91904, Israel*
[b] *Experimental Physics V, Electronic Correlations and Magnetism, Institute of Physics, University of Augsburg, D-86135 Augsburg, Germany*
[c] *Max Planck Institute for Solid State Research, Heisenbergstrasse 1, D-70569 Stuttgart, Germany*

* Corresponding author. Tel.: +97226715829; fax: +97226584437.
*E-mail address:* shaltiel@vms.huji.ac.il (D. Shaltiel).

## 1. Introduction

Great efforts have been made recently to grow high quality triple layer $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ (Bi-2223) single crystals, $T_c = 110$ K, in order to study their intrinsic properties and compare them with those of the double layer $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi-2212), $T_c = 90$ K, and the mono layer $Bi_2Sr_2CuO_{6+\delta}$ (Bi-2201), $T_c = 10$ K. The best available single crystals were grown using the travelling solvent zone technique, with a steep temperature gradient along the melting zone, using a very slow grow rate and long annealing time [1,2]. Large single crystals were obtained that show an onset $T_c = 110$ K. However the transition width was much larger than in Bi-2212. Intergrowth phases (<2%) of Bi-2212 and Bi-2201 were observed that can explain the larger transition width in this compound. Several properties of the Bi-2223 such as the pseudo-gap temperature and resistivity anisotropy were investigated as a function of oxygen doping in these crystals [1–3].

The effect of an ac magnetic field on the vortex system in high-$T_c$ superconductors has been demonstrated by the so called "shaking effect" which showed that the application of an ac magnetic field parallel to the *a–b* plane in the high-$T_c$ superconductors thermalizes the vortex system [4,5].

The interaction of the AC field with the vortex system was studied in high anisotropy superconductors such as Bi-2212 by the "Induced Microwave Dissipation by the AC Magnetic field" (IMDACMF) technique [6,7]. In Bi-2212 the AC magnetic field induces two different signals that result from either *changes in the microwave dissipation* due to changes in the thermally activated flux flow (TAFF) resistivity induced by the AC magnetic field [8,9], or from *microwave dissipation* induced by the interaction of the AC field with the vortex system [6]. The IMDACMF is a contact-less technique, very sensitive and simple to operate. It can measure the superconducting transition in any material either in powders or solids. In single crystals it can investigate the magnetic anisotropy of this transition. Its unique advantage is the investigation of dynamic effects of the interaction of AC magnetic fields with PV and JV in highly anisotropic superconductors where non linear effects were observed [6,7].

As the anisotropic unit cell structure of the tri-layer Bi-2223 is larger than the bi-layer Bi-2212 structure, the effect of the increase of the distance between the conducting layers on the AC interaction with the vortex system can be studied from the changes of the response of the AC field on the microwave absorption, where it is expected that the general behaviour will be similar. Indeed strong signals as strong as in Bi-2212 were observed. However it was found that the intergrowth layers affect the crystal homogeneity and that only a small portion of the crystal shows superconducting properties related to Bi-2223 with a $T_c$ of 110 K. The major part of the crystal exhibits superimposed transitions of superconducting material whose $T_c$'s range from 100 K to 80 K. Nevertheless the results enabled the study of the interaction of the AC field with its vortex system and the effect of the intergrowth layers on its superconducting properties. For the measurements we used a Bruker ELEXSYS spectrometer working at X-band frequency (9.36 GHz). The microwave response of the sample is detected by a lock-in amplifier at the modulation frequency of 100 kHz. A He-continuous flow cryostat ESR 900 (Oxford Instruments) allowed for temperature regulation of the sample in the temperature range $4.2 < T < 300$ K.

## 2. Experimental and results

The Bi-2223 single crystals used in this experiment were grown using an improved travelling solvent floating-zone technique described in Ref. [1] and were characterized by various methods. The crystals used in the present experiment were small platelets of about $1 \times 1 \times 0.2\ mm^3$ whose large facet was a (0 0 1) crystallographic plane, determined by Laue X-ray back-reflection method. The microwave experimental technique is described elsewhere [6]. Note that the sample is exposed to a collinear DC and 100 kHz AC magnetic field. The crystal could be rotated around an axis within the *a–b* plane that allowed the orientation of the magnetic fields parallel or perpendicular to the

$a$–$b$ plane. The transverse microwave magnetic field was always parallel to the $a$–$b$ plane.

Fig. 1 shows the temperature dependence, 115 K > $T$ > 60 K, of the signal intensity at various angles, $\theta$, ranging from $B \| c$ to $B \perp c$ for a Bi-2223 crystal ($\theta$ is the angle between the $c$-axis and the magnetic field). Two sets of signals are observed: a narrow one with low-intensity at temperature 110 K > $T$ > 105 K, shown also as an inset in the figure, and a broad one with high intensity at temperature 100 K > $T$ > 80 K. The low-intensity signals have a bell shape, their intensity reach maximum for $B \| c$, and it decreases with changing the angle being almost zero at $B \| a$–$b$; this behaviour is similar to the shape and angular behaviour observed just below $T_c$ in other high-$T_c$ superconductors such as YBCO [8,9] and Bi-2212 [9]. The intensity of the lower temperature broad signals also varies with angle; their intensity reaches again a maximum at $B \| c$ and decreases towards $B \| a$–$b$ where the intensity is minima. An additional anomaly signal is observed around 80 K.

Fig. 2a and b show the evaluated maximum intensity observed at each orientation as a function of angle for the 110 K > $T$ > 105 K and the 100 K > $T$ > 80 K signals, respectively. The estimated temperature for maximum intensity at these two regions for all angles is 108 K and 90 K, respectively. The intensities are derived from Fig. 1 by subtracting for each angle from the intensity at 108.2 K and 90 K the corresponding intensity at $B \| a$–$b$. They fit closely a $\cos\theta$ function for both 110 K > $T$ > 105 K and 100 K > $T$ > 80 K signals. As discussed below it indicates that the angular dependence at the two temperature regions result from the variation in the TAFF resistivity.

Fig. 3 shows the intensity for $B \| c$ and $B \| a$–$b$ as a function of temperature for a DC field of 1 mT and an AC field of 0.9 mT peak to peak, from temperature above $T_c$ down to 4 K. The signals down to 80 K are the same as those shown in Fig. 1 for these two orientations. At $B \| c$ the low-intensity bell-shaped signal close to $T_c$ is followed by a

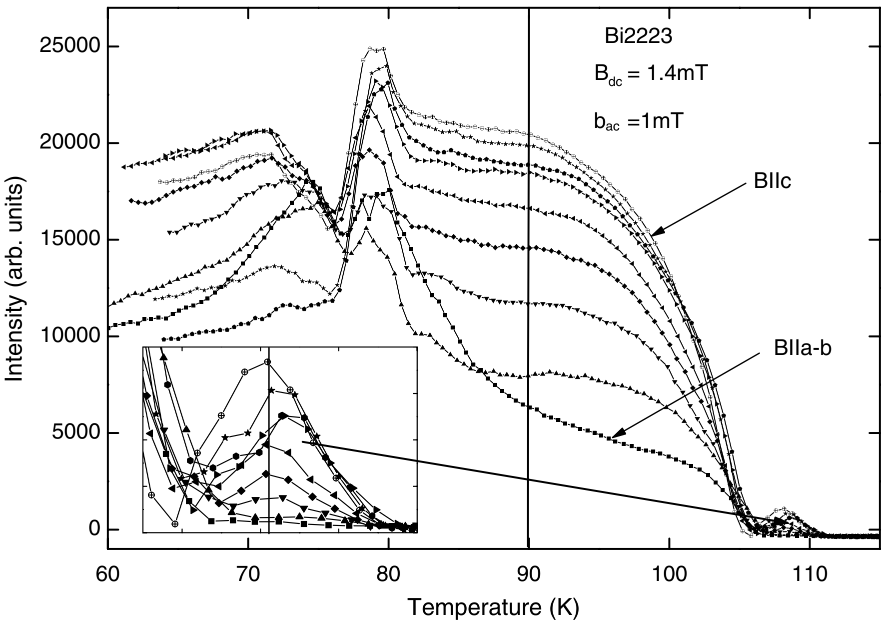


Fig. 1. Signal as a function of temperature 115 K > $T$ > 60 K for different orientations from $B \| c$ to $B \| a$–$b$. It shows: close to $T_c$ (110 > $T$ > 105) a low-intensity bell-shape with a narrow temperature-width signal (enlarged at the inset) and a broad temperature-width signal (100 K > $T$ > 80 K) with a much larger intensity. The intersection of the two vertical lines at $T$ = 108.2 K and $T$ = 90 K with the curves have been used to derive the TAFF intensities for the various angles in Fig. 2. At 80 K an additional signal is observed; it is not related to the dissipation due to the TAFF.

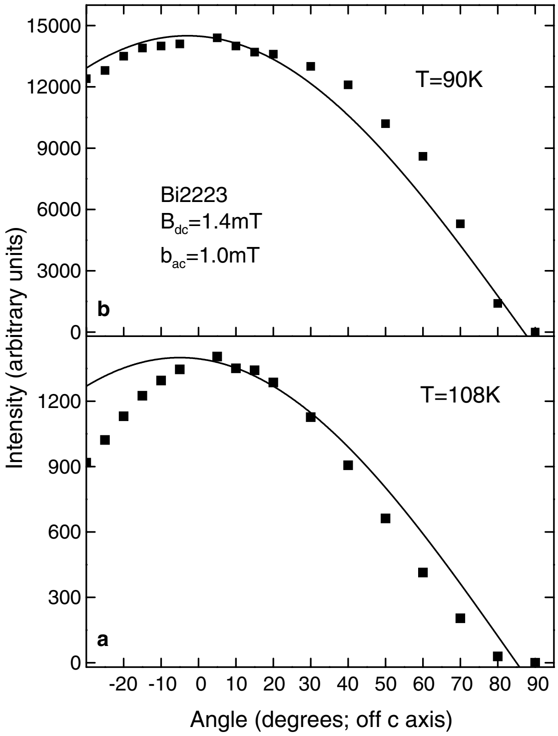


Fig. 2. Intensity of the TAFF signal as a function of angle, derived from Fig. 1 for the bell-shaped 110 K > $T$ > 105 K and the broad 100 K > $T$ > 80 K signals. (a) The intensity of the bell-shaped 110 K signals. (b) The intensity for the 100 K > $T$ > 80 K signal. The intensities were obtained by subtracting from the intensity, at $T = 108.2$ K and 90 K, respectively the intensity at $B \| a$–$b$. This procedure was necessary as the signal intensity for $B \| a$–$b$, expected to be equal to zero, contains additional contributions related to the convolution of the TAFF signal.

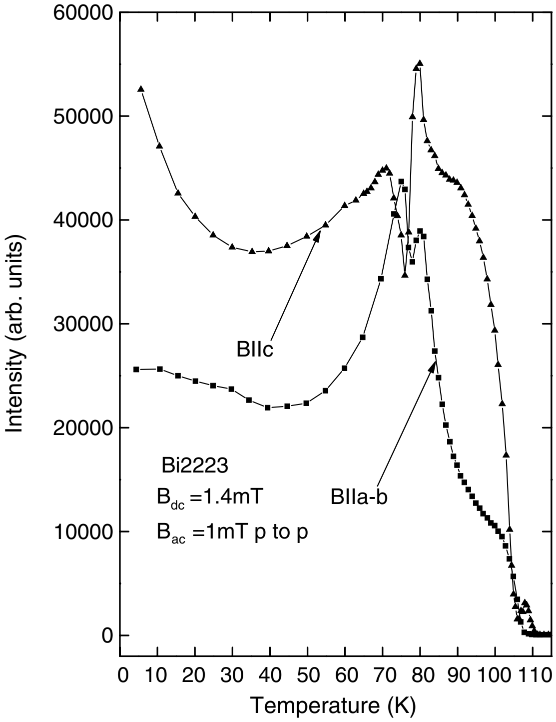


Fig. 3. Signal intensity and signal phase of the Bi-2223 as a function of temperature from above $T_c$ down to 4 K for $B \| a$–$b$ and $B \| c$.

sharp increase and a broad maximum down to 80 K, where the additional anomaly signal is observed. At $B \| a$–$b$ the intensity is zero just below $T_c$ and it increases in two steps down to about 80 K where again the additional anomaly signal is observed. Below about 75 K both orientations show a slight decrease in the intensity that increases again towards lower temperatures; the intensity for $B \| c$ is larger than for $B \| a$–$b$. The larger intensity of the signal below 75 K for $B \| c$ compared to $B \| a$–$b$ has an important consequence in the interpretation of the interaction of the AC field with the vortex system.

Fig. 4a and b show the signal intensity and signal phase (with respect to the AC phase) of the Bi-2223 crystal as a function of the DC magnetic field at a constant AC magnetic field of 0.9 mT (peak to peak) for $T = \sim 80$ K, 47 K and 4 K, for $B \| a$–$b$ and $B \| c$, respectively. Though the intensities for the different temperatures show different behaviour, the signal shape at the three temperatures is similar and their intensities do not differ greatly for both orientations. The same holds for the phases whose dependences as a function of DC field are almost the same. Here again the similarity for the two orientations has important consequence with regard to the interpretation of the AC field with the vortex system.

## 3. Discussion

To understand the origin of the signal intensities as a function of temperature, that range from $T_c = 110$ K down to 80 K (Fig. 1), and the angular behaviour of their maximum intensities (Fig. 2),

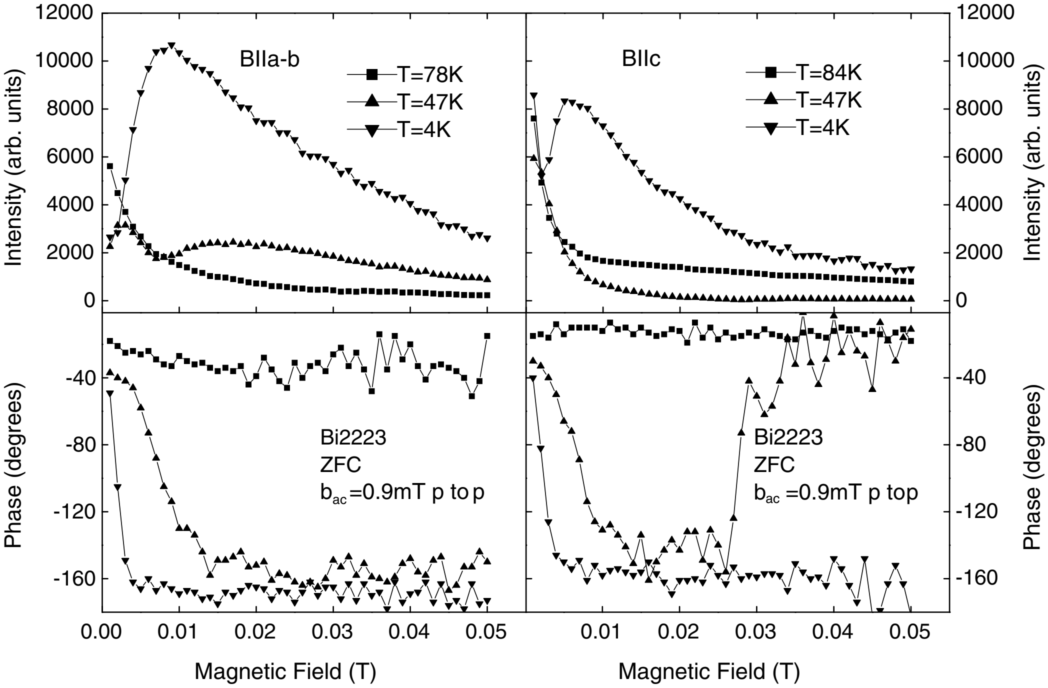


Fig. 4. Signal intensity and signal phase of ZFC Bi-2223 as a function of DC magnetic field at ~80 K, 47 K and 4 K, for an AC magnetic field of 0.9 mT (peak to peak) for $B\|a$–$b$ and $B\|c$. It shows similar behaviour of the signals' shape and small differences in intensity at $B\|c$ compared to $B\|a$–$b$. These results are unexpected when compared with those obtained in an optimally grown Bi-2212 crystal, that shows large intensity signals at $B\|a$–$b$ and zero intensity signals at $B\|c$ [6].

we refer to the Tinkham model on the TAFF resistivity [10] and the Ginzburg–Landau anisotropy theory [11]. The Tinkham model predicts a bell-shaped signal as a function of temperature for the microwave dissipation induced by the AC magnetic field just below $T_c$ [8,9]. Indeed the small-intensity signal just below 110 K fits this model. It has been shown that in anisotropic superconductors, the peak intensity of the bell-shaped signal, $S(\theta)$, as a function of the angle, $\theta$, between the $c$-direction and the magnetic field follows the Ginzburg–Landau mass-tensor formulation [11]

$$S(\theta)/S_{(B\|c)} = [\cos^2\theta + \varepsilon^{-2}\sin^2\theta]^{+1/2} \quad (1)$$

where $\varepsilon$ is the anisotropy constant. For $\varepsilon > 100$ the second term in (1) is negligible and can be omitted, hence the relative intensity $S(\theta)/S_{(B\|c)}$ follows a $\cos\theta$ behaviour. Therefore at large $\varepsilon$ value, it is practically not possible to derive the exact values of the anisotropy constant from the angular dependence of the peak intensity.

Fig. 2a shows that the peak intensity of the Bi-2223 bell-shaped signal close to $T_c$, obtained from Fig. 1 varies as $\cos\theta$. A $\cos\theta$ behaviour is also obtained from Fig. 1 by subtracting from the maximum intensity for each of the 100 K > $T$ > 80 K broad signals the intensity at the corresponding temperature at $B\|c$ (Fig. 2b). Thus the narrow and broad bell-shaped signals observed in Fig. 1 and the $\cos\theta$ angular behaviour of their maximum intensities shown in Fig. 2 can be assigned to the variation of TAFF resistivity. It indicates that the present Bi-2223 compound results from a convolution of superconducting compounds with different superconducting transition temperatures transitions. The 110 K > $T$ > 105 K signal can be assigned to stoichiometric Bi-2223 superconductor; its small-intensity indicates that it occupies only a small part of the crystal. The much larger intensity of the 100 K > $T$ > 80 K broad signal can be associated with a distribution of superconducting phases whose superconducting transition temperature ranges from 100 K down to 80 K, and they compose the larger part of the crystal. Hence the net signal results from a convolution of TAFF signals whose position varies due to their different $T_c$. The close to $\cos\theta$ dependence of the peak intensities as a function of angle indicates that their anisotropy $\varepsilon$ is larger than 100.

The conclusion that different parts of the crystal have different $T_c$'s whose values are lower than the $T_c$ of Bi-2223 except for a small part with the proper $T_c$ of 110 K, is unexpected as X-ray measurements show that more than 98–99% have the Bi-2223 structure [1]. To understand the mechanism that spreads the $T_c$'s towards lower values, we note, from the surface decoration and the TEM measurements [1], that Bi-2212 and Bi-2201 layers were intercalated into the Bi-2223 crystal during the growth process and small amounts of them remain even after annealing. Thus the intercalation of Bi-2212 and Bi-2201 affects the properties of the superconductor and causes an inhomogenous compound with different $T_c$'s lower than the stoichiometric 110 K compound. A consequence from these results is that the measured properties of the Bi-2223 crystals obtained so far may not show its intrinsic properties.

The origin of the 80 K anomaly signal as a function of temperature mentioned above, with only small variation in intensity and temperature for different magnetic field orientations is not yet clear. It was also detected in all other Bi-2223 crystals obtained from different sources. Its intensity decreases sharply with the DC magnetic field [12].

In addition to the TAFF signals that result from modulation of the TAFF resistivity by the AC magnetic field inducing changes in the microwave dissipation signals of strong intensity are observed at temperatures down to 4 K as shown in Figs. 3 and 4. Their behaviour is similar to the signals observed in Bi-2212 crystal, where it was shown that they originate from the interaction of the AC magnetic field with the vortex system, inducing dissipation of microwave power [6]. These AC induced dissipations differ from microwave losses as a function of DC field without the presence AC field reported in Ref. [13] for Bi-2212.

A better insight on the effect of the interaction of the AC field with the vortex system in Bi-2223 can be obtained by comparing the present results with those observed in the optimally-doped Bi-2212 crystal [9] where intercalation of other phases does not occur.

Fig. 5 shows the intensity as a function of temperature in Bi-2212 down to low temperatures for a magnetic field parallel and perpendicular to the $c$-axis. For $B \| a$–$b$ the signal results from the interaction of the AC field with the vortex system. It is characterized by a sharp increase of the intensity just below $T_c$ that increases even further down to 4 K. In contrast, in Bi-2223 the sharp increase is substituted by a broad increase that starts at 103 K (seven degrees below $T_c$) down to 80 K (Fig. 3); it confirms the conclusion deduced from the angular dependent results that indicate a spread in $T_c$'s down to 80 K. The signal below 75 K results from the interaction of the AC field with the vortex system.

For $B \| c$ a single narrow bell-shaped signal in Bi-2212 just below $T_c$ is observed, followed by an almost *zero intensity* signal at lower temperature down to 4 K. The intensity of this bell-shaped signal as function of angle varies as $\cos\theta$ indicating

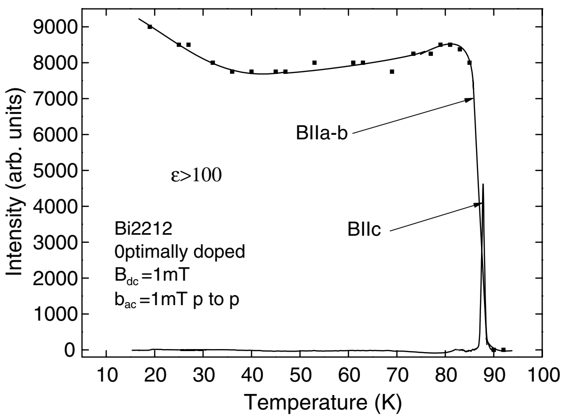


Fig. 5. Signal intensity of the Bi-2212 as a function of temperature from above $T_c$ to 4 K for $B \| a$–$b$ and $B \| c$. Note that for $B \| c$ below the TAFF signal, the intensity is practically zero compared to the large intensity observed in Bi-2223, shown in Fig. 3.

that it is due to the TAFF signal and that the anisotropy $\varepsilon$ is larger than 100 [9]. The zero signal intensity observed at lower temperatures implies that the AC field does not induce microwave interaction with the vortex system when the magnetic fields are perpendicular to the planes where the vortices form vertical stacks of pancake vortices situated on the Cu–O planes. The results in Bi-2223 for $B\|c$ defers in two aspects compared to those of Bi-2212: (1) Although the signal below $T_c$ in both compounds originates from the TAFF interaction, the narrow signal in Bi-2212 indicates a superconducting homogenous crystal with a single transition, compared to the spread in $T_c$'s in the present Bi-2223. (2) The Bi-2223 signal observed below 80 K indicates that the AC field induces microwave interaction with the vortex system for the magnetic field is parallel to the $c$-axis (Fig. 3), where only PV are formed. This is in contrast to the Bi-2212 results that indicate that the AC field does not induce microwave interaction with the vortex system at this orientation. Furthermore the signal intensity of the Bi-2223 at this orientation is even stronger than at $B\|a$–$b$, in contrast to the zero intensity observed in Bi-2212. Assuming, as in Bi-2212, that a necessary condition to obtain microwave dissipation is the presence of JV, the present results indicate that the intercalation affects the distribution of the two types of vortices and enables induced microwave interaction at orientations where only PV are supposed to be formed. It should be emphasised that X-ray analysis and the TAFF results, presented in this work, indicate that the Bi-2223 crystal has a single crystal structure with the $c$-axis perpendicular to the sample *facet* [1].

The interaction of the AC magnetic field with the vortex system in Bi-2223 can also be inferred from the induced microwave dissipation at constant AC field as a function of DC magnetic field for different temperatures. Fig. 4 shows the response as a function of dc field for three temperatures, 78 K, 47 K and 4 K for $B\|a$–$b$ and 84 K 47 K and 4K for $B\|c$. A detailed discussion of the magnetic field dependence and its interpretation is out of scope of the present work. Instead we refer to similar results obtained in Bi-2212 for $B\|a$–$b$ where the signal intensity and signal phase as a function of DC field for temperatures down to 4 K is discussed in detail [6]. Here we point out some features related to the present work. At 80 K and $B\|a$–$b$ the signal intensity decreases exponentially as a function of $B_{DC}$ and the phase is close to zero. The zero phases indicate interaction of the AC magnetic field with pancake vortices. At 4 K the signal has a bell-shape whose phase is close to $-180°$ here the AC field interacts mostly with JV. At the in-between temperature of 47 K the results indicate transition from interaction with one type to both types of vortices. The intensity as a function of DC field in Bi-2212 is maximum at magnetic fields parallel to the planes where mostly JV are formed and goes to zero at $B\|c$ where predominantly PV are formed. However in the present Bi-2223 crystal strong signals are observed at $B\|c$ (Fig. 4b), whose DC field dependence of the signal intensity and the signal phase are similar to those observed for $B\|a$–$b$ for all three temperatures. The occurrence of a strong signal parallel to the $c$-axis is not expected; in particular as the bell-shape signal intensity with the $-180°$ phase at 4 K indicates that it results from interaction with JV [6]. It leads to the same conclusion discussed above that the intercalation, that introduces imperfections, affects the distribution of the two types of vortices and enables induced microwave interaction at orientations where only PV are supposed to be formed.

## 4. Conclusions

This work presents results on the induced microwave dissipation by AC magnetic field in a superconducting single crystal Bi-2223. It was 98–99% pure with 2–1% were Bi-2212 and Bi-2201 phases intercalated in the crystal. The onset $T_c$ was 110 K. The observed microwave signals originate from two entirely different mechanisms.

One set of signals is due to modulation of the TAFF resistivity by the ac field that leads to changes in the microwave dissipation; it is always observed just below $T_c$ in any high-$T_c$ superconductor. The analysis of the TAFF signal shows that intercalation generates regions in the crystal with different $T_c$'s that are spread from 110 K to

80 K, and that only a small part of the crystal has indeed a $T_c$ of 110 K.

The other set of signals results from the interaction of the AC field with the vortex system that induces the microwave dissipation. A detailed description and some interpretation related to this interaction for Bi-2212 are given in Ref. [6]. In the present work the induced microwave dissipation by the AC magnetic field in Bi-2223 single crystals is reported for the first time. It is the third superconducting crystal in addition to Bi-2212 and the organic superconductor $\kappa(ET_2)Cu(NCS)_{2+}$ [14] where similar microwave dissipations were observed [6]. These three materials have in common a high anisotropy resulting from intrinsic Josephson coupling. This interaction was not observed in the optimally-doped low anisotropy $YBa_2Cu_3O_{7-\delta}$ where the chains participate efficiently in the charge transfer between the planes [15]. It indicates that Josephson coupling is a necessary condition to observe this signal. Hence intrinsic Josephson coupling plays a decisive role to induce microwave interaction by the AC magnetic field. This conclusion was derived from measurements in Bi-2212 that showed that the maximum intensity of signals, due to this interaction, occurs at magnetic fields parallel to the *a–b* plane, where mostly JV are formed. They go to zero for the magnetic fields parallel to the *c*-direction, where PV are formed. Assuming that the mechanism that induces the observed microwave dissipation as a function of temperature or DC magnetic field in Bi-2223 is similar to that observed in optimally-doped Bi-2212, it brings to the conclusion that unlike Bi-2212 the vortices in the present Bi-2223 do not arrange themselves as expected, namely mostly PV or JV are formed if the magnetic field is parallel or perpendicular to the *c*-axis, respectively. Thus the intercalation of the additional phases induces deviation from the intrinsic properties of the crystal as it affects the distribution of the two types of vortices enabling AC induced microwave interaction at orientations where only PV are supposed to be formed. An anomaly signal around 80 K, whose intensity decreases strongly with the dc field, has been observed in all measured Bi-2223 crystals. The origin of this signal is not clear yet. Additional efforts to grow single phase Bi-2223 crystals are necessary to investigate their intrinsic properties.

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