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MAGNETICALLY MODULATED MICROWAVE ABSORPTION INVESTIGATION OF $Bi_{1.8}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_x/(LiF)_y$ SUPERCONDUCTING SYSTEM

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The effect of LiF content on the 2223 phase of the high temperature superconducting system $Bi_{1.8}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_x/(LiF)_y$ (y = 0.02 to 0.15) was investigated by means of magnetically modulated microwave absorption (MAMMA). The experimental results confirmed a two fold increase of the superconducting phase content together with a slight rise (around 2 %) of the critical temperature for y=0.07. At the same time, for y between 0.05 and 0.12 the $-\Delta T_c^{mw}/\Delta B$ ratio was practically constant and equal to (18.5 ± 1.5) KT^{-1}, a typical value for 2223 superconducting phase.

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1. Introduction

The critical current density J_c represents one of the most important parameters for any potential applications of the high-temperature superconductor (HTS). At the same time, above 77 K the flux - pinning effect is weak. Consequently, by properly engineering the pinning centers, which includes and the doping with various atoms, it is possible to increase significantly the critical current density. Previous investigations [1, 2] have showed that Li substitution in Cu and LiF addition to $Bi_2Sr_2CaCu_2O_{8+\delta}$ (denoted by Bi-2212) superconducting system, lead to a significant grow of the critical current density by increasing of the Bi-2212 superconducting phase content. A similar result was noticed on $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ (denoted by Bi-2223) ceramics and tapes [3, 4]. On the other hand, the F-doping of (Bi,Pb)-Sr-Ca-Cu-O (BiPSCCO) superconductors, results in the enhancement of the flux pinning effect and the remarkable improving of the superconducting properties of the Bi-2223 phase [5].

By using the standard solid state reaction method, recently, batches of $Bi_{1.7}Pb_{0.4}Sr_{1.5}Ca_{2.5}Cu_{3.6}O_x/(LiF)_y$ superconducting system, y varying between 0.02 and 0.3, were prepared and investigated by various technique such as resistive, AC complex susceptibility and DC magnetization measurements [6, 7]. Due to the fact that the synthesis procedure led to critical temperatures of the Bi-2212 phase lowers than 77 K [7], there was no response from this phase above 77 K.

In this paper we present the results of the critical state study by an alternative method, Magnetically Modulated Microwave Absorption (MAMMA) [8, 9] carried out on batches of $Bi_{1.8}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_x/(LiF)_y$ (y between 0.02 and 0.15) system, close to above mentioned

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composition. The application of this method is of primary importance because whereas resistance measurements record the onset of the first superconducting path in the sample, microwave absorption technique provides a measure of the sample state, in the sense that all regions of the sample contribute to the observed response. Consequently, the microwave absorption, as measured by MAMMA, presents a peak at the so called average critical temperature T_c^{mw}. This temperature, which is lower than the T_c determined by resistance measurement, has the advantage of characterizing all regions of the sample and not only the first superconducting path as T_c does. Moreover, the features attributed to the Josephson Junctions (JJs) formed at interfaces between superconducting grains and clusters are observed below T_c^{mw} as a rising baseline.

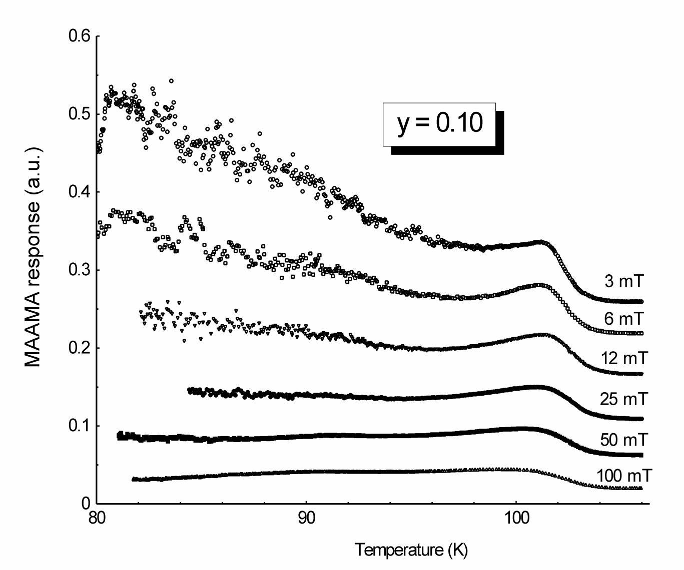

Fig. 1. MAMMA responses for different DC magnetic fields of the $Bi_{1.8}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_x$/ $(LiF)_y$; y =0.1.

2. Materials and Methods

To prepare samples with nominal $Bi_{1.8}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_x/(LiF)_y$ composition, high-purity $SrCO_3$, $BaCO_3$, $CaCO_3$, and CuO powders mixed in the ratio Sr:Ba:Ca:Cu = 1.8:0.2:2:3 were pressed in 9×9×3 mm^3 pellets at 0.75 GPa and calcinated in an alumina crucible at 924 ^{o}C for 85 hours. The pellets were ground in an agate mill and the upper procedure was repeated again twice at 947 ^{o}C and 950 ^{o}C. Bi_2O_3 and PbO powders were added in the obtained precursor and were mixed to obtain the ratio: Bi:Pb:Sr:Ba:Ca:Cu = 1.8:0.4:1.8:0.2:2:3. The obtained powder was calcinated at 820 ^{o}C for 20 hours in air. Small quantities of final calcinated mixture were ground again, sieved through a 40 μm mesh, thoroughly mixed with various amount of LiF, pressed in 3 x 3 x 10 mm^3 pellets at 0.75 GPa and sintered in air at 834 ± 2 ^{o}C for 300 h.

The MAMMA investigations were carried out in the X-band (9 GHz) at an incident microwave power of 3 mW and in 3, 6, 12, 25, 50 and 100 mT DC magnetic fields, superimposed over a 0.1 mT AC magnetic field (100 kHz). Resulting MAMMA responses were registered by slowly increasing the sample temperature (~ 1 K min^{-1}) from 77 K up to 110 K. The sample temperatures were measured with a precision of ± 0.2 K, while all other experimental errors were no greater than ± 5 %.

Investigated samples, cut from the initial pellets, were placed in the center of a TE_{011} resonant cavity, where the microwave magnetic component reached a maximum. Both DC and AC magnetic fields were perpendicular to the microwave one.

It must be pointed out that due to the fact that the microwave electric field vanishes at the sample position, the eddy currents induced by the microwave magnetic field are the primary source of losses.

Table 1. The experimental values of the MAMMA parameters of investigated samples.

	Sample					
	y = 0.02		y = 0.05		y = 0.07	
B (mT)	T_c^{mw} (K)	I (a.u.)	T_c^{mw} (K)	I (a.u.)	T_c^{mw} (K)	I (a.u.)
3	101.0	6.05	100.6	5.37	101.6	11.22
6	100.4	5.79	100.4	4.91	101.4	9.27
12	99.6	4.55	100.2	3.86	101.2	7.41
25	98.7	3.58	99.8	3.13	100.9	5.61
50	97.9	3.01	99.3	2.48	100.6	4.39
100	97.3	2.18	98.8	1.78	99.3	3.02
$-\frac{\Delta T_c^{mw}}{\Delta B}$	-		18.5 ± 1.5 (K T^{-1})			
	y=0.10		y=0.12		y=0.15	
B (mT)	T_c^{mw} (K)	I (a.u.)	T_c^{mw} (K)	I (a.u.)	T_c^{mw} (K)	I (a.u.)
3	101.26	7.70	100.60	4.34	99.60	1.29
6	101.20	6.17	100.55	3.48	99.55	1.00
12	101.10	5.00	100.42	2.82	99.50	0.86
25	100.80	4.08	100.17	2.30	99.38	0.73
50	100.30	3.33	99.80	1.88	99.06	0.59
100	99.30	2.48	99.90	1.40	98.20	0.47
$-\frac{\Delta T_c^{mw}}{\Delta B}$	18.5 ± 1.5 (K T^{-1})				-	

(a.u. stands for arbitrary units)

3. Results and discussion

Fig. 1 illustrates different MAMMA responses, corresponding to $y = 0.1$ sample and recorded for different DC magnetic fields varying between 3 mT and 100 mT. All MAMMA signals are reported for the same amplification and sample weight. As it can be seen from this figure, the baseline below T_c^{mw} monotonously goes down by increasing the DC magnetic field, signifying the decoupling of the superconducting grains. At the same time, the noise-like signal which appears at lower temperatures gradually vanishes. For 100 mT magnetic field, all grains are decoupled; this fact being testified by a total absence of this kind of signal. Simultaneously, the positions of MAMMA peaks are slowly shifted towards lower temperatures. All the other samples have shown similar features.

To characterize in a proper manner the physical properties of the investigated systems, we have calculated for each case the following experimental MAMMA parameters: average critical temperature T_c^{mw}, MAMMA peak amplitude I_{max}, as well as $-{\Delta T_c^{mw}}/{\Delta B}$ ratio. All these data are reproduced in Table 1.

By analyzing these data, we can make the following remarks:

Table 2. Matrix of the correlation coefficients of the MAMMA peak amplitudes vs. magnetic field for all investigated samples. All correlations are significant for $p < 0.005$.

2 %	2%					
5 %	0.9980	5%				
7 %	0.9885	0.9946	7%			
10 %	0.9777	0.9872	0.9978	10%		
12 %	0.9778	0.9872	0.9978	1	12%	
15 %	0.9639	0.9771	0.9927	0.9977	0.99768	15%

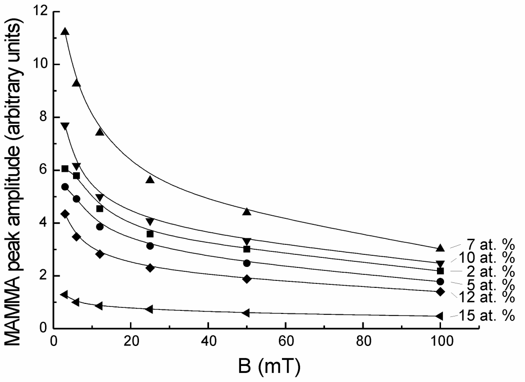

Fig. 2. MAMMA peak amplitudes vs. DC magnetic field for all investigated samples.

i.- The amplitudes of MAMMA peaks for all investigated samples depend on the DC magnetic field in a similar manner (Fig. 2). This peculiarity is sustained by the existence of a significant positive correlation ($r > 0.96$, $p < 0.005$) between the numerical values of the MAMMA peak amplitudes and the DC magnetic field for each sample (see Table 2). The result pleads additionally for a structural identity of the 2223 superconducting phase in all samples.

As well, it must be pointed out that the amount of this phase reaches a maximum at a LiF concentration of 7 at. %.

It is worth to mention that for all samples, excepting the extreme ones, the $-\Delta T_c^{mw}/\Delta B$ ratio was almost constant and equal to 18.5 ± 1.5 (K T^{-1}), the typical value for 2223 superconducting phase as it has been experimentally established in ref. [10, 11].

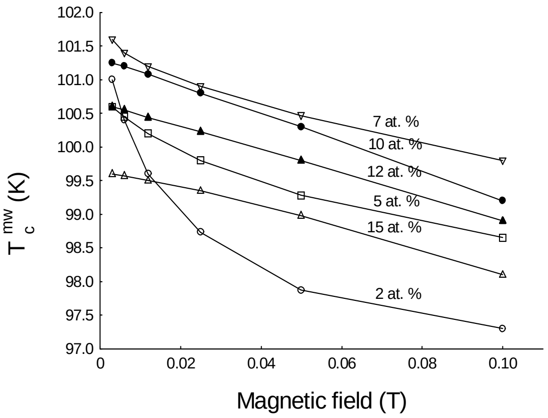

Fig. 3. Average critical temperature T_c^{mw} vs.DC magnetic field for all investigated samples. It can be remarked that the maximum critical temperature corresponds to the y= 7 at.% sample.

ii. – By increasing the LiF concentration, the critical temperature increases, reaching a maxim for *y* around 0.07, and than slowly decreases. This growth is more significant for low DC magnetic fields. This peculiarity, as well as the maximum reached by the 2223 superconducting phase quantity, can be explained as due to an optimum hole concentration.

4. Concluding remarks

High temperature superconducting system $Bi_{1.7}Pb_{0.4}Sr_{1.5}Ca_{2.5}Cu_{3.6}O_x/$ $(LiF)_y$ was investigated by magnetically modulated microwave absorption (MAMMA), evidencing the effect of LiF content on 2223 phase critical state. The experimental results show a significant growth (about two fold) of the 2223 superconducting phase quantity accompanied by a slightly increase (around 2 %) of the critical temperature for $y \sim 0.07$. Moreover, for *y* between 0.05 and 0.12, we have noticed that the ratio $-{}^{\Delta T_c^{mw}}\!\big/_{\Delta B}$ is practically constant and equal to (18.5 ± 1.5) KT^{-1}, typical value for the 2223 superconducting phase.

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