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$YBa_2Cu_3O_{7-\delta}$:Li SUPERCONDUCTOR SYSTEM INVESTIGATED BY DIRECT MICROWAVE ABSORPTION METHOD

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The influence of thermal neutrons irradiation at different fluencies on the superconductivity properties of Li (2 at. %) doped $YBa_2Cu_3O_{7-\delta}$ system has been investigated by direct microwave absorption method. We have noticed changes of the superconducting properties of the 123 phase that depend on the neutron fluency. By increasing fluency, initially, the phase structure supports a disordering, then a re-ordering accompanied by a significant increase of the average critical temperature T_c to 85.7 K and finally we observed a degradation of the 123 the phase accompanied by a diminishing of T_c. The intragranular dephasing magnetic field B_d, i.e. the direct microwave absorption (DMA) parameter associated with the magnetic field dependence of the critical current density exhibited an opposite tendency, initially decreasing and then increasing at a value close to those of unirradiated samples. We also noticed only on the irradiated samples the presence of a flatness of the DMA curves around 0.5 T, consistent with the expression of a peak effect.

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1. Introduction

In the past years, direct microwave absorption (DMA) [1], together with magnetically modulated microwave absorption (MAMMA) [2,3] remark themselves by simplicity and, at the same time, the amount of information they can provide concerning the high-Tc superconductors (HTcS).

The information provided by this method concerns the dependency of microwave absorption versus steady magnetic and, as a rule, are well illustrated by a characteristic incomplete inverse bell shape absorption curve whose minimum corresponds to zero value of dc-magnetic field (Fig. 1). For bulk samples, these curves show a fast rising (FR) section of the microwave absorption, usually within 0 to 40 mT magnetic field range, followed, at higher field values, by a slow rising (SR) section (Fig. 1). Both these sections are strongly related to the current lines inside the material, but having different sources.

As proved in ref. [1], the decoupling of the intergranular Josephson junctions (JJ's) by the external dc-magnetic field generates the FR section, while SR regain is related to the contribution of the intrinsic weak links from inside the grains. In this way, the asymptotical values of the DMA signals (considered in this work as conventional "zero" reference levels of the corresponding DMA responses) correspond to the maximum microwave absorption in sample. (Fig. 1) while the SR section can furnish information concerning the superconducting phase within the material itself.

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According to ref [1], the SR microwave absorption section can be very well described by a simple exponential law:

$$Y = |Y_0| exp\left(-\frac{B}{B_d}\right) \tag{1}$$

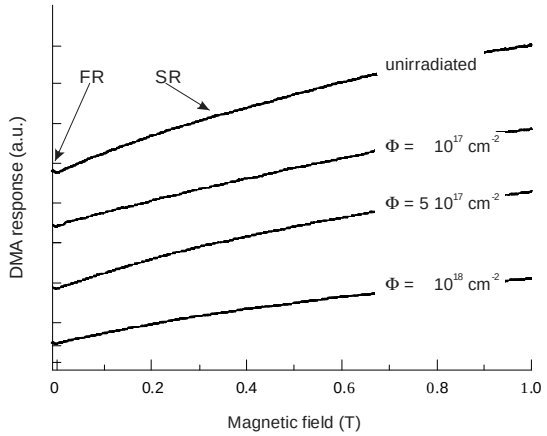

Fig. 1. The experimental DMA curves of the $YBa_2Cu_3O_{7-\delta}$:Li (2 at %) system before and after the thermal neutron irradiation. The first part of the absorption curve (FR), corresponds to a sharp increase of the microwave absorption while the second one (SR) illustrates a much slower recover. This section is very well described by a in the absence of a peck effect by simple exponential growth function.

where: Y represents the SR DMA signal, Y_0 is the maximum value of the SR DMA signal corresponding to zero-magnetic field and reflecting the amount of superconducting phase, B stands for the magnetic field, while B_d represents the intragranular dephasing magnetic field, tightly correlated with the intragranular critical current density [1].

Usually, the numerical value of the Y_0 parameter is obtained by a best fit method from the SR section of the DMA curve. It is worth to mention another important parameter of DMA curve, the zero magnetic field value of the FR section, Y_{0j}, directly connected to the intergranular JJ's number. Its value has can be calculated by subtracting the Y_0 amplitude from the experimental DMA curves in the region of zero dc-magnetic fields.

In this way, DMA represents a complementary method that allows the estimation of the intergranular Y_{0j} JJ's concentration, the extent Y_0 of superconducting phase, as well as the values of intragranular dephasing magnetic field, B_d. All these peculiarities makes DMA a simple, inexpensive and at the same time very efficient method of investigation of a great variety of high Tc superconducting materials.

Applied to neutrons irradiated samples, this method is very useful in characterising the influence of heavily ionising radiation onto the HT_cS. Consequently, in this paper we present the results of our studies by direct microwave absorption (DMA) on the neutron irradiated Li (2 at. %) doped $YBa_2Cu_3O_{7-\delta}$ system.

2. Materials and methods

Polycrystalline samples of the $YBa_2Cu_3O_{7-\delta}$:Li (2 at. %) system were prepared by the conventional solid-phase reaction from a mixture of high-purity Y_2O_3, CuO and $BaCO_3$. Li was introduced by adding of LiOH. Finally, appropriate amounts of these materials were calcinated at 930 ^oC for 20 hours in flowing oxygen. After that, the final compound was reground, pressed in pellets and again sintered under the same conditions. Previous investigations [4] showed that the

samples are single phase orthorhombic $YBa_2Cu_3O_{7-\delta}$, with an important amount of large (<100 μm) and well matched grains (like the bricks in a wall).

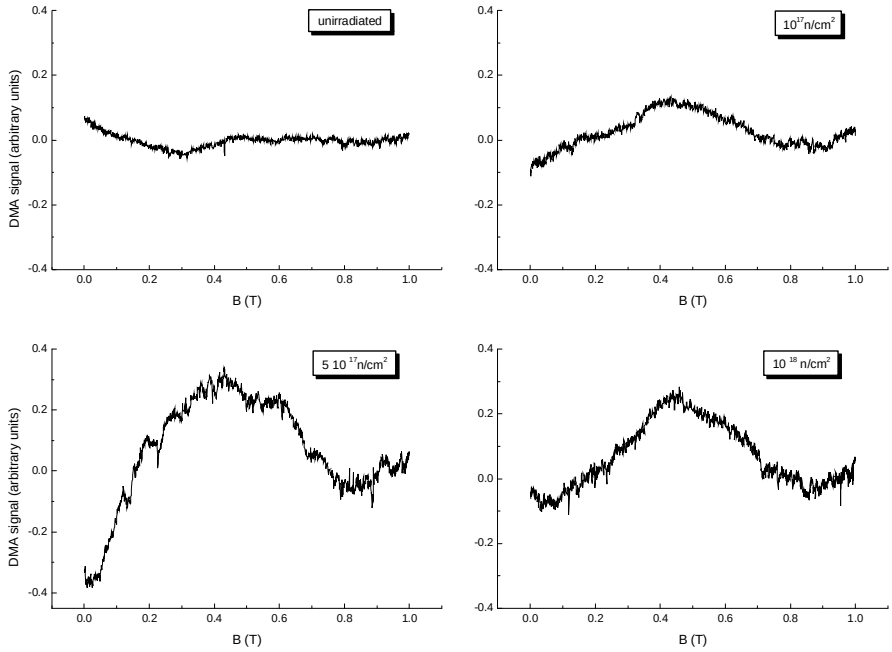

Fig. 2. The difference between observed SR DMA curves and simple exponential growth curve described by eq. (1) for both unirradiated and thermal neutron irradiated $YBa_2Cu_3O_{7-\delta}$:Li (2 at. %) samples.

The irradiation with thermal neutrons was performed at the VVRS nuclear reactor of the National Institute for Physics and Nuclear Engineering-Bucharest at 10^{17} cm^{-2}, 5×10^{17} cm^{-2} and 10^{18} cm^{-2} fluencies. During irradiation, the sample temperature was maintained below 40 ^oC by using an appropriate cooling system.

The direct microwave absorption investigations were carried out at 77 K by means of an on-line, X-band (9 GHz) home-made spectrometer, at an incident microwave power of the 3 mW and by sweeping slowly the steady magnetic field from -15 mT to 1 T in 2 min. The magnetic field induction has been monitored by the means of a digital teslameter with an average precision of 0.1 mT. During the measurements, samples has been placed in the maximum of the microwave magnetic field. Further, the signal amplitudes were reported at the same amplification and at the same sample quantity, *i.e.*10 mg. More details concerning experimental set-up, spectrometer sensitivity and measurements reproducibility have been previously described in ref. [1-3,5].

3. Results and discussion

For a better illustration of our experimental results, in Fig. 1 we have reproduced all experimental DMA curves, while Table 1 contains the numerical values, as determined using a best-fit method, the corresponding parameters, *i.e.* zero-magnetic field FR (Y_{0j}) and SR (Y_0) amplitudes, as well as the intragranular dephasing magnetic field B_d. At the same time we have figured the numerical values of the thermal neutron fluencies, taking into account that between these values and the concentration of the induced defects there is a positive correlation.

Table 1. The DMA and MAMMA [6] parameters of the $YBa_2Cu_3O_{7-\delta}$:Li (2 at. %) samples before and after the thermal neutron irradiation. One can note a similar dependency of both DMA and T_c^{mw} MAMMA parameters.

Sample	Thermal neutrons fluency	DMA		MAMMA		
		Y_{0j} (a.u.)	Y_0 (a.u.)	B_d	T_c^{mw} (K)	ΔT_c^{mw} (K)
$YBa_2Cu_2O_{7-\delta}$ (2 at. % Li)	unirradiated	0.07	9.0	1.02 ± 0.05	84.3	> 3.0
	10^{17} cm^{-2}	0.08	11.9	0.79 ± 0.03	83.1	< 3.0
	5×10^{17} cm^{-2}	0.10	13.1	0.96 ± 0.03	85.7	2.3
	10^{18} cm^{-2}	0.05	17.1	0.96 ± 0.03	81.3	4.0

- a.u. stands for arbitrary units.

At a careful analysis of all DMA curves we have observed around 0.5 T, for all irradiate samples the same peculiarity consisting of a small deviation from the exponential law (1) characterizing the SR section of the curves. In order to evidence clearly this effect, we subtracted from the experimental curves the exponential ones as determined from the fit of the experimental data at magnetic fields lower than 0.15 T and higher than about 0.7 T (Fig. 2). At the same time, we have observed that the area under this peach reaches a maximum for a thermal neutron fluency of 5×10^{17} cm^{-2}.

On the other hand, we observed an inverse variation with the thermal neutron fluency of the dephasing magnetic field (B_d)which decreases after 10^{17} cm^{-2} fluency, when 123-phase was disordered and recovers almost at the initial value for 5×10^{17} cm^{-2} arguing for an almost complete restoration of the 123-phase (Table 1). At the same time, the density of intergranular JJ's maintains a very low and nearly insensitive value to irradiation, in total agreement with the MAMMA investigations [6].

In this way, the experimental data concerning both SR peak area and Y_0 parameters support the initial assumption made on the basis of MAMMA results that certain initially non-superconducting intergranular zones were activated at T > 77 K by irradiation. Some of such zones, with T_c closed to 77 K, can act as efficient intragranular pinning centres if the steady magnetic field normalises them [7], determining, in this way, the slow growth of intragranular dephasing magnetic field B_d after the highest fluencies (Table 1).

It must pointed out that the SR peaks reflect the usual peak effect described in ref. [8-16]. Such peaks mark the role of the surface as a source of pre-irradiative defects that constitute accumulation centres for the point defects generated during irradiation. By increasing the magnetic field, the small zones containing clusters of point defects coalesce together and become pinning centres.

4. Concluding remarks

Li (2 at. %) doped $YBa_2Cu_3O_{7-6}$ have been irradiated at room temperature with thermal and epithermal neutrons at fluencies varying between 10^{17} cm^{-2} and 10^{18} cm^{-2} and then investigated by means of direct microwave absorption method.

Depending on the neutron fluency the superconducting properties of the 123 phase significantly changed. By increasing the fluency, the 123 phase structure supports initially a disordering, then a re-ordering accompanied by a significant increase of the average critical temperature which reached a maximum at 5×10^{17} cm^{-2} which was followed again by a degradation.

The intragranular dephasing magnetic field B_d, the DMA parameter associated with the magnetic field dependence of the critical current density, reflects the evolution of the structure,

with a minimum for the same value of neutron fluency followed by an increase at higher fluencies. Previous microwave magnetically modulated absorption confirmed these findings. Another interesting peculiarity that is manifested only in irradiated samples and consists of the presence of a flatness of the DMA curves around 0.5 T was interpreted by us as a peak effect.

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