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Microwave Absorption Study on (Bi, Pb)–Sr–Ca–Cu–O Granular Superconductors

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(Bi, Pb)–Sr–Ca–Cu–O is considered as a system of 2201, 2212 and 2223 superconductors embedded in the insulating matrix. The size of the grains depends on the time of recrystallization. These types of ceramics exhibit a two-step transition to superconducting state. Because electrical properties depend among other on the Josephson coupling between grains, the magnetically modulated microwave absorption study was undertaken. Magnetically modulated microwave absorption signal was observed to arise just as temperature had been lowered below T_1. The shape of this signal was studied to recognize the second temperature T_2. Some strong oscillations appear on magnetically modulated microwave absorption at lower temperatures, which might be related to local percolation breakdown in superconducting network.

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1. Introduction

Microwave absorption experiments are usually carried out in standard EPR spectrometer with magnetic field modulation. Therefore, the microwave absorption effect is referred to as magnetically modulated microwave absorption (MMMA). The use of the second magnetic modulation makes the observed signal to be the first derivative $\mathrm{d}P/\mathrm{d}B$ of absorbed power P [1]. In this paper we present measurement of MMMA in (Bi, Pb)–Sr–Ca–Cu–O as a function of temperature, magnetic field and microwave power. As a result of annealing, oxide superconductors belonging to the bismuth family crystallize forming a granular superconductor. Granular superconductors are materials containing superconducting grains embedded in insulating matrix. We show the influence of magnetic field and microwave power on superconducting properties of (Bi, Pb)–Sr–Ca–Cu–O.

2. Experiment

The samples were obtained by the solid state crystallization. The crystallization was carried out at 840°C. At this temperature, 2201, 2212 and 2223 phases crystallize [2]. The samples were annealed for the time intervals between 2 min and 20 h. The measurements of resistivity as a function of temperature are published elsewhere [3]. Magnetically modulated microwave absorption measurements were performed with a standard X-band EPR spectrometer RADIOPAN SE/X 2543 with TM_{102} cavity and a 100 kHz magnetic field modulation B_{m} [4]. The signals were recorded during a continuous change of temperature with the rate of 2 K/min. An Oxford ESR 900 flow helium cryostat was used for low-temperature measurements.

3. Results

The sample is a granular superconductor and exhibits a two-step transition to superconducting state [5] (Fig. 1). At T_1 most of the isolated granules transit

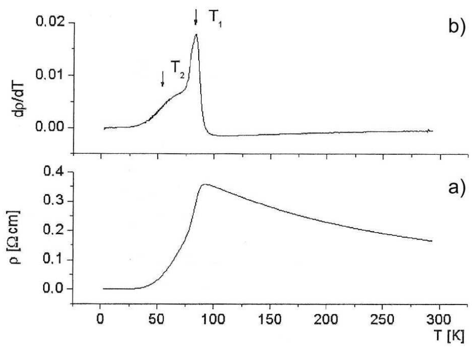

Fig. 1. An example of the superconducting transition in granular superconductor material (a), as well as an illustration of determination of two critical temperatures (b).

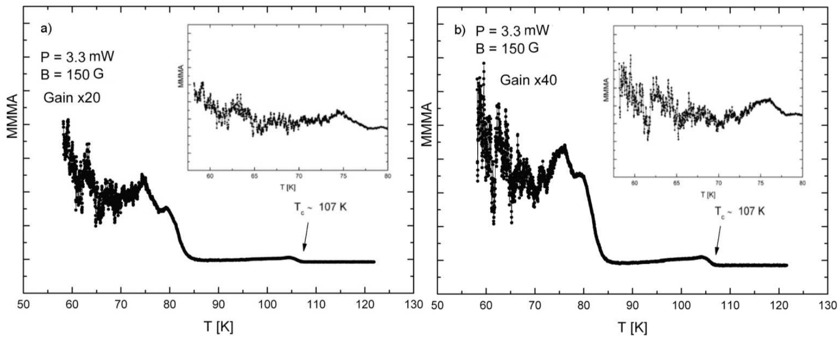

Fig. 2. MMMA vs. temperature; dependence on DC magnetic field of the sample annealed at 840°C for 20 h. The insets show the MMMA signal between 67.5 and 80 K. The magnetic field was applied in the direction perpendicular to the sample surface (a) $B = 150$ G and (b) $B = 250$ G.

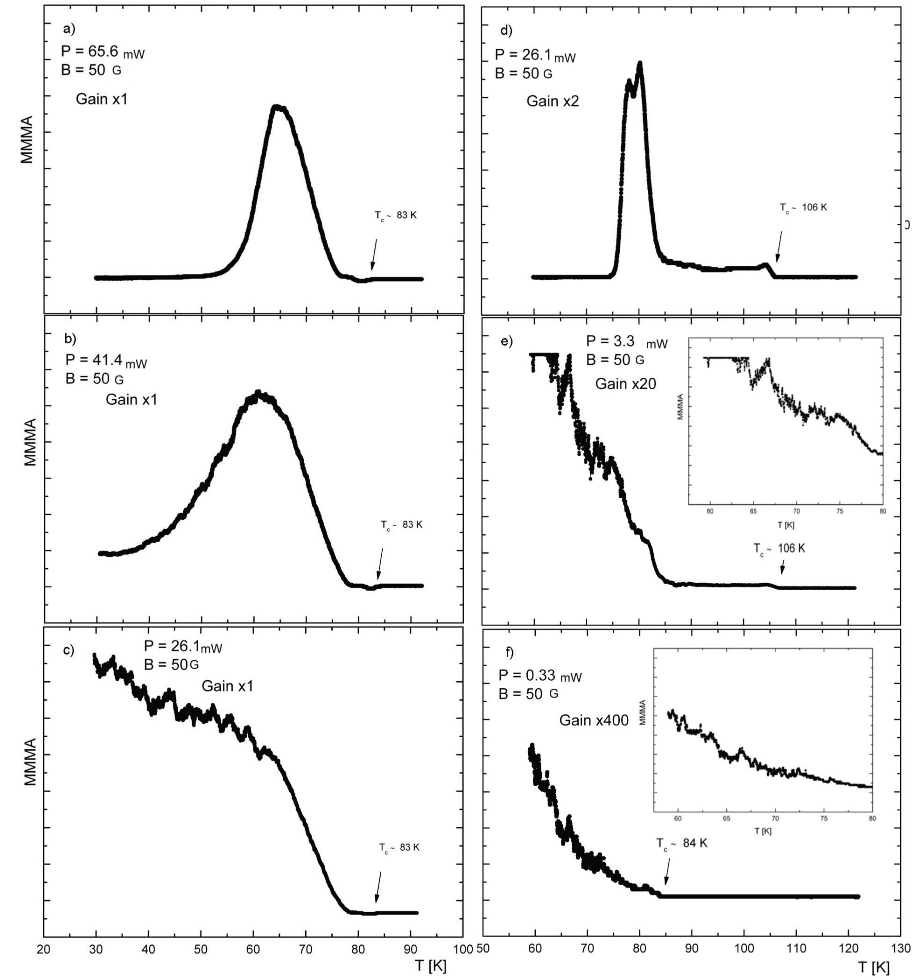

Fig. 3. MMMA vs. temperature; dependence on microwave power for the sample annealed at 840°C for 2 min ((a), (b), (c)) and 20 h ((d), (e), (f)). The insets show the MMMA signal between 67.5 and 80 K.

to superconducting state. At T_2 grains couple into the long-range order. We show MMMA signals for the samples annealed at 840°C for 2 min and 20 h. These samples were selected for MMMA measurements, because their microstructure depends strongly on the time of the annealing [2]. From conductivity measurements we know that $T_c^{\text{onset}} \approx 86$ K, $T_1 \approx 82$ K, $T_2 \approx 56$ K and $T_c^{\text{onset}} \approx 108$ K, $T_1 \approx 105$ K, $T_2 \approx 94$ K, respectively, for 2 min and 20 h annealed samples. MMMA signal was observed to arise just as temperature was lowered below T_1. The shape of this signal has been studied to recognize the second temperature T_2. The T_1 temperature for both samples does not depend on external magnetic field and microwave power.

Figure 2 shows the MMMA vs. temperature, dependence on microwave power. The measurements were performed with the 50 G magnetic field applied in the direction perpendicular to the sample surface. Some strong oscillations appear on MMMA at lower temperatures, which might be related to local percolation breakdown in superconducting network. The temperature at which oscillations start is located around 65 K and 75 K for samples annealed for 2 min and 20 h, respectively. This temperature does not depend on the applied magnetic field. The examples of magnetic field dependence on MMMA are shown in Fig. 2. These oscillations disappear for higher microwave power (Fig. 3a, d), 41.4 mW and 26.1 mW, respectively, for 2 min and 20 h annealed samples. Also the slopes of MMMA signals are very sensitive to microwave power. It requires further studies to solve this problem.

4. Conclusion

The MMMA method applied to (Bi, Pb)–Sr–Ca–Cu–O ceramics proves to be a very sensitive, complementary method to study inhomogeneous superconductors. For the sampled annealed for a long time this method, contrary to conductivity measurements, showed separately a weak transition to superconducting 2223 phase and a strong one at 2212 T_{c} (Fig. 3d and e). Therefore, the T_2 temperature derived from conductivity measurements does not apply to this sample. The oscillations on MMMA signal appear at temperatures above $T_{\mathrm{c}}(R = 0)$ of the bulk sample, thus we suggest they might be related to local percolation breakdown in superconducting network.

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