INDUCTANCE PROBE EXAMINATION OF A SUPERCONDUCTING Bi–Pb–Sr–Ca–Cu–O CERAMIC

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الخلاصـــة :

تم تصنيع واختبار المادة مفرطة الموصلية ذات التركيب الكيميائي $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_x$ بطريقة فاحص المحاثة الموصوفة حديثا [مرجع ١] . وأظهرت الطريقة المذكورة أن درجة الحرارة الحرجة (T_c) تتراوح مابين (١٠٥ - ١١٠) كلفن ووُجدت أنها متوافقة مع ماسبق نشره من أبحاث مختلفة [مرجع ٤] ؛ حيث تنخفض المحاثة بإضطراد من (٢٠٠) كلفن قبل أن تصل إلى درجة مفرطة الموصلية . وأوضح الانخفاض في المحاثة أن (٢٥٪) من حجم العينة يصبح مفرط الموصلية مقارنة بما يحدث في نظام YBCO ونظام Tl–Ba–Ca–Cu–O .

دلت قياسات المحاثة المغناطيسية (عند ٧٧ كلفن) تحت درجة الحرارة الحرجة (T_c) أن قيمة الحقل الحرج الأول (١٧) جاوس (H_{c_1} = 17 gauss) وقيمة الحقل الحرج الثاني (٤) تسله (H_{c_2} = 4 T) ؛ وأنها تتفق مع ماسبقٍ نشره من أبحاث مختلفة .

ABSTRACT

The superconducting material with the nominal composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_x$ has been synthesized and examined by the inductance probe method recently described by the authors [1]. The inductance data shows T_c to be in the range 105 K − 110 K, in good agreement with previously reported results [4]; the inductance falls steadily from about 200 K before the main superconducting transition is reached. The observed drop in inductance indicated that 25% of the volume of the material is superconducting, in contrast to the behavior of YBCO and Tl − Ba − Ca − Cu − O systems. Inductance−field measurements, at 77 K, below T_c show that the lower critical field $H_{c_1} = 17$ gauss, and the upper critical field $H_{c_2} = 4$ T, in good agreement with published data [11].

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1. INTRODUCTION

In three previous papers we have outlined the use of inductance probe methods to study the superconducting properties of high-T_c superconductors [1–3]. At the superconducting transition temperature, magnetic field flux is expelled from the sample, according to the Meissner effect [4]. The principle of the method is to use a small coil which can be filled with a superconducting material, and then cool the sample below its transition temperature T_c. The transition to a superconducting state is associated with a sharp drop in the inductance of the coil. This provides sufficient information to determine T_c, to examine the width of the superconducting transition and to estimate the percentage of the superconducting volume of material. Estimates of the lower critical field H_{c_1} can also be made by observing the change in the slope of the inductance (of the pre-cooled coil) with an externally applied d.c. magnetic field.

In the previous work attention was concentrated on the behavior of $YBa_2Cu_3O_{(7-x)}$ [1] and $Tl_2Ba_2Ca_2Cu_3O_{(10+x)}$ [2] compositions. In the present study we report the results of such a study on sintered pellets of Bi–Pb–Sr–Ca–Cu–O compositions.

Bulk superconductivity in the Bi–Sr–Ca–Cu–O system was first discovered by Maeda *et al.* [5] who observed stable and reproducible superconductivity with two phases characterized by the transition temperatures of 85 K (low-T_c phase) and 108 K (high-T_c phase). Shortly afterwards the 85 K and 108 K superconducting phases in the Bi–Pb–Sr–Ca–Cu–O system were discussed by Tarascon *et al.* [6] who reported the high-T_c phase has an excess Cu–O square sheet relative to the low-T_c phase in the stacking structure. Recently Sang-Geun Lee *et al.* [7] reported the upper and lower critical fields at 83.3 K for the $Bi_{1.4}Pb_{0.6}Ca_2Sr_2Cu_3O_y$ superconductor with T_c-108 K (see Table 1 for more details).

2. EXPERIMENTAL PROCEDURES

A specimen with a nominal composition of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_x$ was prepared in the conventional manner using high purity powders of Bi_2O_3, PbO, $SrCO_3$, $CaCO_3$, and CuO. The mixtures were calcined at 810°C for 19 hours in air. The calcined material was reground and pressed into pellet form, as small discs of diameter about 6 mm. The pellet was sintered at 850°C for 168 hours and annealed at 850°C for 24 hours followed by furnace cooling to room temperature in air. Phase analysis of the products was carried out by x-ray diffraction using a Hagg–Guinier camera with strictly monochromatic radiation and KC1 as the internal standard.

Table 1. Comparison of Present H_{c_1} Data with Previously Reported Values in Bi–Pb–Sr–Ca–Cu–O Sintered Pellets as Determined from Magnetization Measurements.

Author	Chemical formula	H_{c_1} gauss	Temperature of measurement (K)
Present work	$Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_x$	15–20	77
Sang [7]	$Bi_{1.4}Pb_{0.6}Ca_2Sr_2Cu_3O_y$	45	83.3
Joo-II [8]	$Bi_{1.7}Pb_{0.3}Sr_2Ca_2Cu_3O_y$	60	79
Murayama [11]	$Bi_{1.5}Pb_{0.5}Sr_2Ca_2Cu_3O_y$	6	77
Kimihito [12]	$Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_y$	8–16	77–105

The phase analysis of the specimen on which measurements are reported here was the high-T_c phase. The microstructure of the specimen was examined by scanning electron microscopy. Figure 1 shows the grain structure is indistinct and appears to be a fiber-like structure superimposed on a fused matrix.

All the measurements were made using the inductance probe method to monitor the inductance (L) of the coil with the superconductor inserted [1]. The variation of the inductance was measured as function of either temperature with zero externally applied magnetic field or in the presence of an applied magnetic field at a given temperature. A special coil having a room temperature inductance of 22.7 mH was constructed. This had a bore of volume about 160 mm^3 so only about 6% of the bore's volume was filled with superconducting material. Temperatures were recorded by Cu/constantan thermocouple placed in contact with the specimen inside the bore of the coil. All the measurements were made at a frequency of 10 kHz.

3. RESULTS AND DISCUSSION

3.1. Determination of T_c and Percentage of Superconducting Volume

The superconducting behavior of the specimen was first explored by observing the temperature variation of inductance in the absence of any applied magnetic field. Figure 2 shows a linear fall in the inductance (L) from room temperature (293 K) to 200 K and less pronounced though still significant from 200 K to just above 108 K. Just below 108 K there is a sudden sharp drop in the inductance (L) and below about 100 K the inductance remains constant to within the experimental error. The onset temperature of the superconducting transition agrees well with the value 107 K reported initially by Maeda *et al.* [5] from resistance *versus* temperature measurements and with the more recent data of Lee *et al.* [8] who studied the temperature dependence of electrical resistivity of a $Bi_{1.4}Pb_{0.6}Ca_2Sr_2Cu_3O_y$.

Following the method described in detail in [1] we have used the magnitude of the drop in inductance on cooling below the transition temperature T_c to estimate the fraction of superconducting material (S) present in the specimen. Direct substitution of the inductance at room temperature (L_{RT}) and the inductance at low temperature (L_{LT}) values into the relation

$$S = \frac{L_{RT} - L_{LT}}{P \times L_{RT}} \times 100 \qquad (1)$$

gives the percentage as $S = 25\%$. The packing fraction, (P), was found by weighing the pellet introduced into the coil and using the expression

$$P = \frac{m}{\rho l A}, \qquad (2)$$

where m is the mass of the pellet, ρ the x-ray density of the material (taken as 4.8 g/cm^3 according to Jain *et al.* [9] and Yusheng *et al.* [10]). A and l are the cross-sectional area and the length of the bore of the coil respectively. The S value is considerably higher than any previously observed value for Y–Ba–Cu–O superconducting ceramics [1], which typically gave S values of about 15%, and less than Tl–Ba–Ca–Cu–O superconducting ceramics [2], which typically gave an S value of about 55%. For well-prepared high-T_c samples the percentage of superconducting volume reaches 100%.

3.2. Estimation of Critical Fields

Most of the reported H_{c_1} values in the literature have been made by magnetization measurements; a selection of the results is given in Table 1 [7, 8, 11, 12]. From this it can be seen that the reported values of H_{c_1} vary over a considerable range; the highest value is 60 gauss [8] and the lowest value is 6 gauss [11]. In this study, the critical field behavior was examined by observing the change in inductance (at a fixed temperature) caused by the application of an external magnetic field provided by a Helmholtz pair. This normally produced a drop in inductance (if the sample is superconducting). The sample temperature was kept constant, *e.g.* at 77 K, and the inductance was monitored as the magnetic field was increased from zero upwards. The results obtained using Helmholtz pair are shown in Figure 3. At 77 K the lower critical field, (H_{c_1}), value is small, being about 17 gauss. The main features revealed are that there is a substantial rise at low field followed by a very small rise to the maximum field (180 gauss). At this maximum field the inductance is still significantly less than the room temperature value. It must be remembered here that the room temperature value of the inductance of the coil with superconductor at zero field is 22.7 mH. At 77 K the slope above 20 gauss is 6.67×10^{-5} mH gauss^{-1}. Assuming that a linear extrapolation can be made, the upper critical field H_{c_2} may be estimated as the values of field at which the normal coil inductance would be observed; this extrapolation yield value of 4 T at 77 K. This may be compared with the value quoted by Sang-Geun Lee *et al.* [7] of 60 T at 83.3 K for sintered pellets.

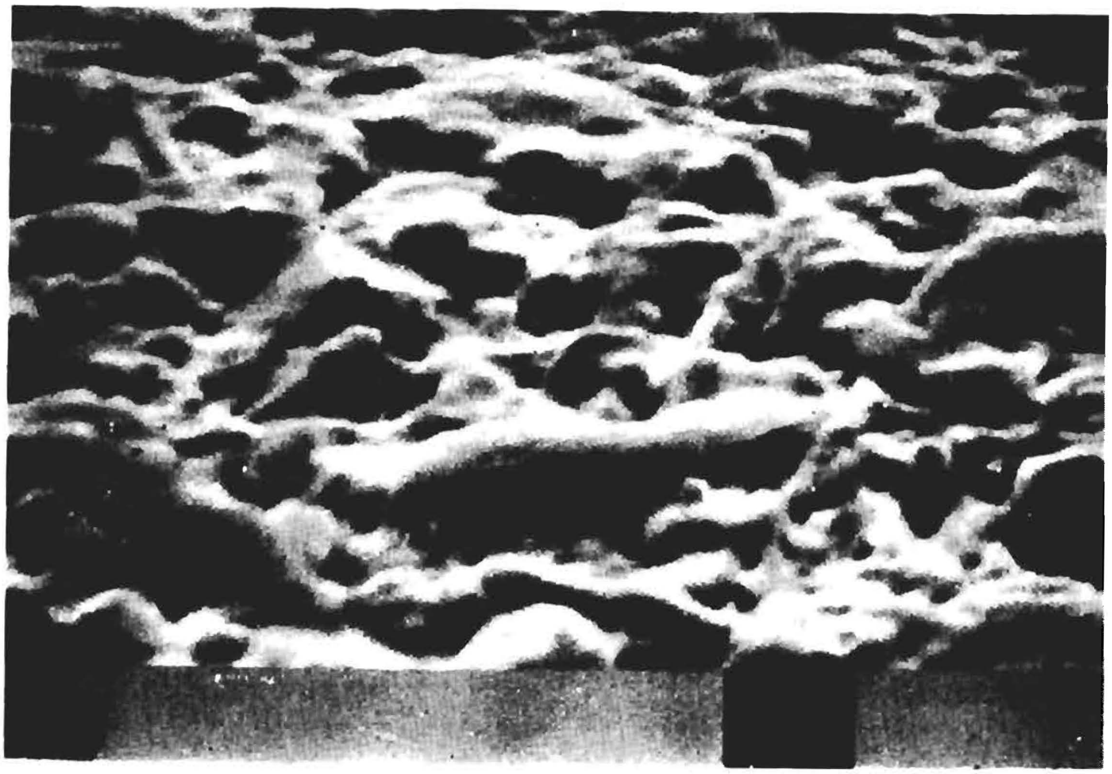

Figure 1. SEM Micrograph of Bi–Pb–Sr–Ca–Cu–O Sintered Pellet.

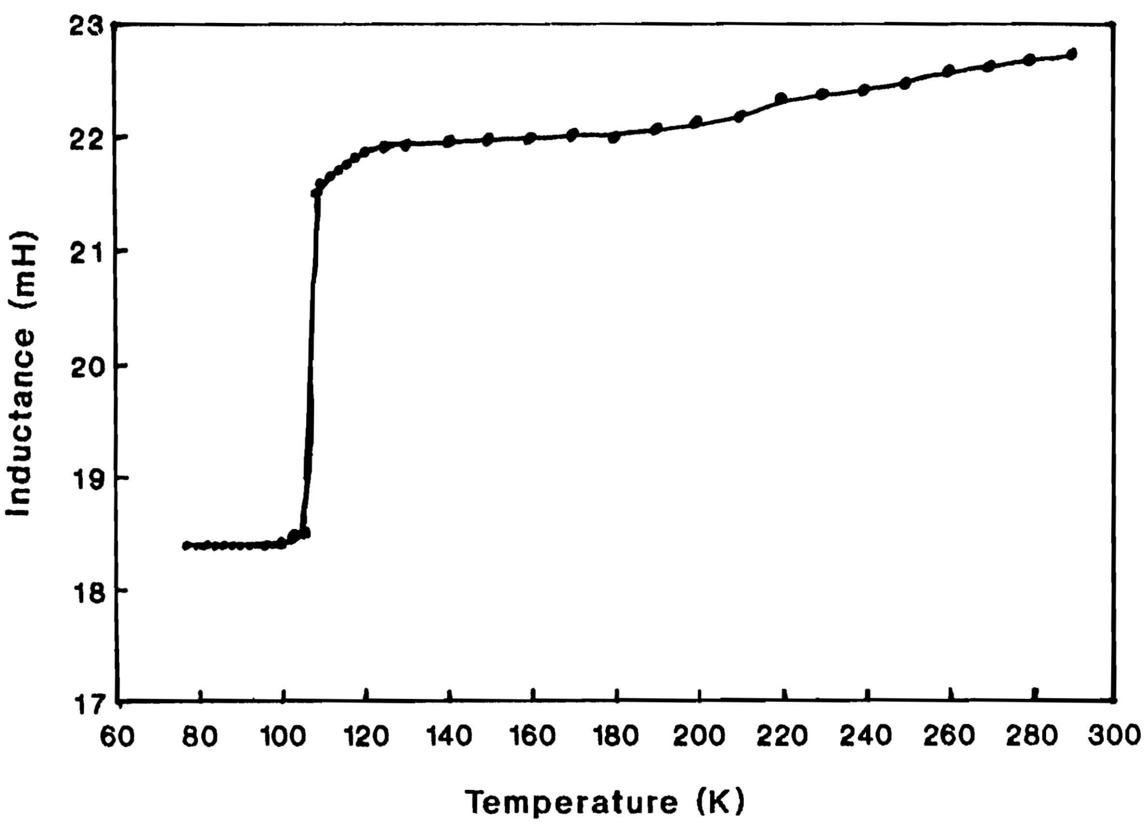

Figure 2. Variation of Inductance with Temperature for Bi–Pb–Sr–Ca–Cu–O Sintered Pellet.

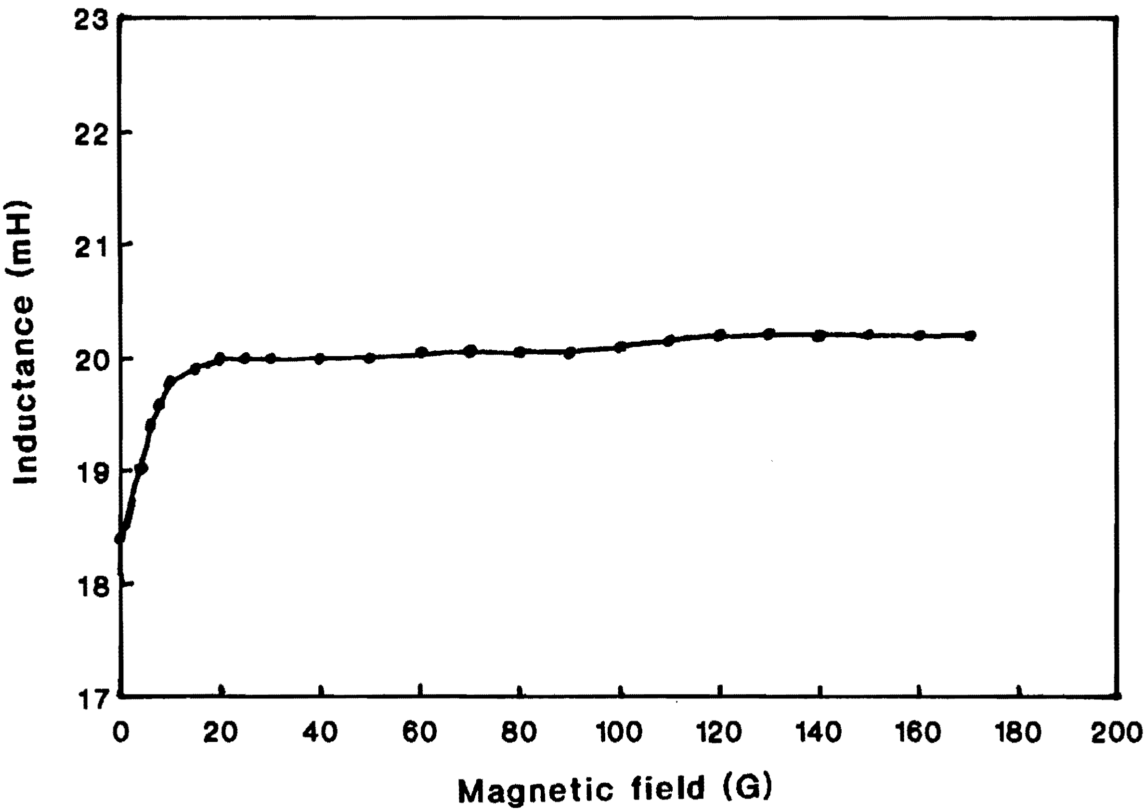

Figure 3. Variation of Inductance with Magnetic Field for Bi–Pb–Sr–Ca–Cu–O Sintered Pellet at 77 K.

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