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High Pressure Studies of the Superconducting Transition in $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10}$*

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Abstract

The superconducting transition for a bismuth–lead superconductor has been investigated at helium pressures to 500 MPa. The form of the resistance transition is similar at all pressures and shows a marked discontinuity at ~107·5 K for the material investigated. The characteristic temperature defining the T_c transition initially falls at 100 MPa and then increases at 200 MPa. Somewhere above 200 MPa the normal resistance increases more rapidly and also irreversibly. Repeated cycling above 200 MPa generates a small resistance in the sample at temperatures below the onset temperature. This is apparently caused by pressure-induced slip or fracture. The normal resistance around 120 K increases systematically with pressure and temperature.

1. Experimental Features

The resistance of $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10}$ has been measured around 110 K at isostatic pressures between 5 and 500 MPa. For each experiment the system was taken to the desired pressure using He gas and then released to atmospheric pressure at the conclusion of each experiment. Pressures were measured using a manganin gauge. The temperature of the sample was measured using a copper–constantan thermocouple inside the pressure chamber. The pressure dependence of the thermocouple temperature coefficient was determined by correlating readings for a platinum resistance thermometer inserted in the outside of the pressure vessel during immersion in both liquid nitrogen and liquid oxygen. A linear pressure coefficient of $0\cdot003\,\mathrm{K\,MPa^{-1}}$ was obtained. This correction is of the same order of magnitude as the variation in transition temperatures and is believed to account for the discrepancies in the data published for YBCO (August 1988).

2. Experimental Detail

Sample Preparation

Starting materials consisting of Bi_2O_3, PbO, $SrCO_3$, $CaCO_3$ and CuO, and corresponding to the composition $Bi_{1\cdot6}Pb_{0\cdot4}Sr_2Ca_{2\cdot5}Cu_{3\cdot5}O_x$, were mixed

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mechanically and reacted first at 800° amd then at 830° for 14 hours, with intermediate regrinding. The resulting powder was formed into 2 mm thick discs by uniaxial pressing at 2 tonnes per square centimetre, and sintered at 860°C in air for 60 hours. The sample was cooled at 90° per hour and annealed at 400°C in flowing oxygen for 2 hours. X-ray diffraction measurements showed that the material consisted almost entirely of the 2:2:2:3 phase ($T_c \approx 107$ K), together with a few per cent of the 2:2:1:2 ($T_c \approx 85$ K) phase and a trace of Ca_2PbO_4.

The superconductor was prepared as a square prism, 2 mm on an edge and approximately 15 mm in length. Several loops of tinned copper conductor were attached at the ends and at three intermediate locations. These were tack soldered and a thermocouple soldered to the middle location. Spaces between the wire loops were filled with silver dag and the surface coated with high resistance varnish.

Experimental Apparatus

The apparatus consisted of a cylindrical stainless steel sample chamber, a 730 mm connecting length of pressure tubing (16 mm O.D.×3·2 mm I.D.), and a pressure head incorporating ten electrically insulated cones for individual wire connections to the ambient pressure environment. The vertically mounted pressure chamber could be conveniently immersed in a dewar containing liquid nitrogen or liquid oxygen for boiling point calibrations. The rate of warming during experiments could be influenced by raising or lowering a deep dewar containing some liquid nitrogen.

The pressure head was connected by high pressure tubing to a helium pumping system consisting of a 2 kbar diaphragm compressor and a 12 kbar capacity intensifier (1 kbar ≡ 10^2 MPa). Pressure was measured using a manganin gauge calibrated against several melting points for mercury (Molinar *et al.* 1980).

Sample temperature was measured with a copper–constantan thermocouple located near the surface of the sample and close to its midpoint. The sample was wrapped in teflon tape and placed in a snug-fitting copper tube in the bore of the sample chamber. The distal ends of the thermocouple wires were soldered to beryllium copper cones in the pressure head. A four probe 50 ohm platinum resistance thermometer was inserted in the centre of the base of the pressure chamber and exposed to atmospheric pressure. Its centre was located approximately 30 mm below the centre of the sample contained in the high pressure chamber.

Measurements and Temperature Calibration

The T_c transitions were observed using a four probe measurement of resistance. The 10 mA current was switched off between measurements and the voltage was measured with the current flowing in either direction. The pressure vessel was cooled by immersion in liquid nitrogen and was then allowed to warm at a rate of one degree every six minutes.

Calibration of the thermocouple was carried out over the range of pressures at the boiling points of liquid nitrogen and liquid oxygen which provided steady thermal conditions. The thermocouple e.m.f. was compared under these conditions with temperatures indicated by the platinum resistance thermometer. The latter was calibrated in turn against the boiling point of oxygen at known atmospheric pressure (Kemp 1977), at the NaCl–ice eutectic (−21·13±0·02°C) and at the triple point for water (0·01°C). The NaCl–ice eutectic was determined using a platinum resistance thermometer (E106) supplied by the National Measurement Laboratory (CSIRO). With the aid of standard curves, these three points were fitted to fourth-order polynomials. The resulting expressions are accurate to ±0·2 K.

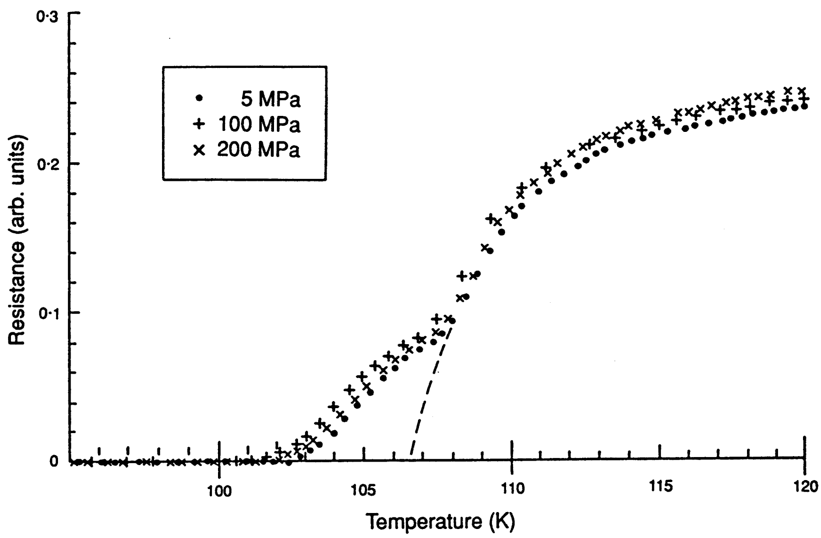

Fig. 1. Resistive transition at 5, 100 and 200 MPa. The dashed line corresponds to a typical result at zero applied pressure.

3. Analysis

The form of the resistance transition shown in Fig. 1 is common for the results at all pressures. The discontinuity at ~107·5 K is interesting because measurements on identically prepared materials in the absence of applied pressure normally show a smooth transition to zero resistance at ~106·5 K (R. Driver and C. Andrikidis, unpublished results). The low temperature tail could therefore be the result of pressure induced microstructural modifications which impair the continuity of the 106 K superconducting phase. It should be noted that the lead doped 2:2:2:3 phase crystallises from a melt and, as Asano *et al.* (1988) have observed, the resulting porous microstructure can be markedly altered by an intermediate cold pressing. Alternatively, the low temperature tail may reflect the presence of a second superconducting phase.

It is difficult to find a common method of relating the experimental curves to an absolute temperature. The 107·5 K discontinuity and low temperature tails make it difficult to locate temperatures where resistance begins. The inflection points (where $d^2R/dT^2 = 0$) were calculated by integrating a second order expression for the first derivative. This method uses five consectutive data points and gives satisfactory results except at the actual inflection points.

Another method (consistent with the inflection determinations but more reliable) is to determine the linear component of the resistance variation above (and also below) T_c, and to draw rays through the baseline intercept with slopes of 0·1 and 0·9 times the resistance slope. Two characteristic temperatures are thus defined where the rays meet the resistance curves. Because the form of the curve is the same at all pressures, this procedure was accepted as the most satisfactory.

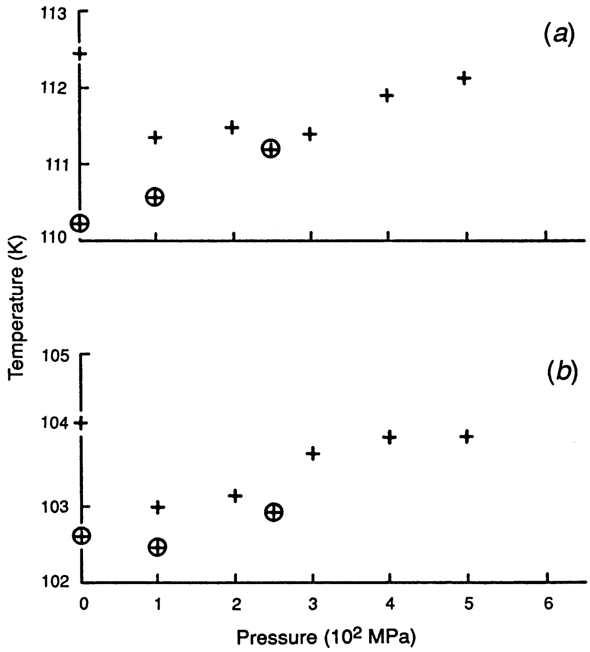

Fig. 2. Changes in characteristic temperature with pressure for (*a*) the higher and (*b*) the lower characteristic temperatures. The lower points (with circles) in each diagram were obtained in repeat experiments after progressive cycling to 500 MPa.

4. Results

The changes in characteristic temperatures with pressure are shown in Fig. 2. The first three crosses in both parts (*a*) and (*b*) show that the characteristic temperatures initially fall at 100 MPa and then increase at 200 MPa (see also Fig. 1). The normal resistance around 120 K increases systematically with pressure and temperature. Somewhere above 200 MPa the normal resistance starts to increase more rapidly and also irreversibly. Because of this behaviour some lower pressure measurements were repeated after the sample had cycled to 500 MPa. These results revealed that the normal resistance remained high and that small residual resistances occurred at temperatures below the onset temperatures. For these repeat experiments the characteristic temperatures were lowered between about 0·5 and 2 K (see circled crosses in Fig. 2). The sample used in the experiment was removed and X-rayed, but no changes could be found in the spectrum or in line widths.

5. Explanation

The material $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10}$ forms a granular aggregate with many small superconducting regions separated by grain boundaries. The 107·5 K discontinuity may represent different regimes of planar and perpendicular conduction, with distinct intergrain boundaries between the superconducting regions. Above 200 MPa the superconducting regions either slip or fracture, increasing the extent or width of the grain boundaries. While the grains can be recompressed so that the material is superconducting the more extensive damage leaves a permanent low pressure resistance.

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