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## **The remanent magnetization of Bi-Ca-Sr-Cu-O ceramic rings in weak magnetic fields.**

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### **Abstract**

The isothermoremanent magnetic moment ( $M_{IR}$ ) of the Pb-doped Bi-Sr-Ca-Cu-O ceramic rings with different height-to-diameter ratios has been studied using a superconducting quantum interference device (SQUID) magnetometer. It is found that in low fields when the magnetic field starts to penetrate into the sample,  $M_{IR}$  is proportional to some power of magnetic field. The exponent lies between 2 and 3 (the magnitudes for the well known long cylinder and thin disk limits, respectively). The SQUID-magnetometer is created by the authors and has some peculiarities: pick-up coils are designed as the symmetric second-order gradiometer, and solenoid contains two coils.

### **Introduction**

The magnetic properties of materials such as magnetization curves, ac response, relaxation, flux penetration in superconductors have been studied using the specimens that are far from ideal geometry assumed by the existing theories for the evaluation of such experiments. The field distribution could be exactly accounted for only in the special case of ellipsoids with linear magnetic response by the introduction of a demagnetizing factor. Magnetization of the real II-type superconductors is highly nonlinear and hysteretic, and the demagnetizing effects are negligible only in the infinitely long cylinders (or long prisms with arbitrary cross sections) in the magnetic field parallel to the axis (the so-called parallel geometry). In the case of parallel geometry the

magnetic behaviour of the II-type superconductors is well described by the Bean model [1-3]. The usual Bean model predicts both the linear dependence of the initial penetration depth and the quadratic dependence of the penetrated flux on the magnetic field. Recently, the exact solutions to the magnetization curve of thin disks [4-7] and strips [7-10] were found for the field perpendicular to the surface (the so called perpendicular geometry). According to these solutions the dependence of the trapped magnetic moment on the magnetic field is expected to be cubic (instead of quadratic for long cylinders and prisms). It was experimentally proved for the strips of  $\text{YBa}_2\text{Cu}_3\text{O}_7$  thin films on the  $\text{SrTiO}_3$  substrate [11].

Magnetisation experiments are often performed using cylinders comparable in their heights and diameters in the field applied along the axis of symmetry. The good understanding of the magnetic behaviour of these objects is actual and provokes great interest. Although some attempts were made to solve the problem of field penetration into arbitrary cylinder in an axial magnetic field [10], it is not solved yet. Thus the systematic experimental data on the magnetic response should be very useful for the construction of theoretical models and their proof.

In the present paper the isothermoremanent magnetic moment  $M_{IR}$  of Bi-Ca-Sr-Cu-O ceramic cylinders with a co-axial hole (i.e. rings) is investigated in low magnetic fields for different height-to-diameter ratios.  $M_{IR}$  is found to be proportional to  $H^\alpha$ , where  $H$  is the applied field, and the exponent  $\alpha$  depends on the height-to-diameter ratio and lies between two and three. Superconducting rings were chosen as an object of our study because in our laboratory the dynamics of the vortex motion at **dc** and **ac** magnetic fields has been studied simultaneously in the HTSC rings in which the field penetration was recorded using the Hall probe located in the center of the ring [12].

## Experimental

Magnetic measurements were made using a superconducting quantum interference device magnetometer created in our laboratory. Since most details of our magnetometer are more or less adaptations of arrangement described in the literature [13], we will emphasize only those parts relevant to what we consider to be different in our magnetometer. We have chosen the scheme of the symmetric second order gradiometer [14], slightly modified to design pick-up coils of the flux transformer connected to the SQUID.

The pick-up system of our magnetometer have four single-coil sections of an

equal diameter of 12 mm wounded by a 0.1 mm niobium wire on a cylindrical high-purity copper tube which confines a vacuum chamber and forms with a sample enclosure a reentrant Dewar (the so-called anti-Dewar). The two interior sections are wound in the same direction opposite to the direction of the outward sections. The distances between the neighbouring sections are: 7.5, 6.8 and 7.5 mm consistently from one end to another. This arrangement is obtained by means of a standard symmetric second order gradiometer by separating the double-wound central coil into two equal parts and displacing them oppositely along the axis of symmetry for equal distances. This design of the flux transformer provides important advantages in continuous registration regime (as registration of a temperature dependence at constant magnetic field or magnetization curves at constant temperature). In these measurements the sample is positioned at the geometric center of a gradiometer. Anyone can see that the interior sections look like the Helmholtz coils, but a 6.8 mm distance between them slightly exceeds the radius  $R=6$  mm. It is well known that the electric current flowing in the Helmholtz coils creates the most homogeneous magnetic field, so that the pick-up coils of Helmholtz design are almost insensitive to small sample shifts inside the coils, accordingly to the reversibility theorem (which declares that  $\Phi/M=B_M/J$  where  $\Phi$  is a magnetic flux in some contour created by magnetic moment  $M$  placed at some point;  $B_M$  is the component of magnetic induction  $B$  along the  $M$  direction at the same point, and  $J$  is the electric current in the same contour which creates the field  $B$ ). Thus one can calculate the distribution of field caused by the current in the pick-up coils instead calculating the flux dependence on the sample position. Taking into account the field from the outward coils, we must enlarge slightly the distance between the interior coils to maintain the homogeneity of field distribution. The optimum distance between the interior coils depends on the distance between the outward coils. We have chosen the above sizes as a certain compromise between the small size of the registration part of the flux transformer and rather high sensitivity. The calculations show that the total flux in these pick-up coils varies less than to 1% if the sample shifts from the center within  $0.35 R$  (i.e.  $\sim 2$  mm). For comparison the signal change in the single coil according to the same shifts is about 15%. For smaller shifts the signal variations are proportional to fourth power of shift opposite to the second power for the case of the single sensitive coil.

However the main advantage is that in the symmetric second order gradiometer the magnetic flux from a sample holder and a thermocouple could be efficiently discriminated. It could be easily theoretically proved. If the homogeneously magnetized

infinite rod pierces all the coils of the second order gradiometer parallel to the axis of symmetry, the total magnetic flux obviously equals zero due to astatism of the pick-up system. And if we consider two parts of this rod separated by the plane of symmetry (perpendicular to the axis of the system), the total flux caused in the pick-up coils by the upper part, is evidently equal to that caused by the down part of the rod due to symmetry of the system, not only by the magnitude but also by the sign! Consequently, the fluxes from both parts are strictly equal to zero (because the sum of two equal parts is zero). It should be noted that for the full discrimination of the signal in this system there is no need for the coincidence of the rod and the axis of the system.

As a rule, the sample holder could be considered as a homogeneously magnetized semiinfinite rod placed by its end at the geometric center of the pick-up system. Therefore its flux should be discriminated. Often for the temperature control a thermocouple is used, and its end is located outside the pick-up coils and apart from the sample, and the distance is usually chosen as a compromise between rather small error in the temperature measurement and rather weak magnetic signal from the thermocouple. The same arguments as for the sample holder could be applied to the thermocouple placed by its end on the plane of symmetry. Therefore the thermocouple could be placed directly near the sample to measure temperature accurately, and without influence on the magnetic signal of the sample. In practice the discrimination of the magnetic flux from both the sample holder and the thermocouple is not full, because the real sample holder and thermocouple are slightly inhomogeneous, and their ends are usually positioned on the plane of symmetry with some error. Nevertheless these reasons are not significant and our pick-up system discriminates 25-30 times the interfering flux from the sample holder and the thermocouple compared to the widely used ordinary first-order gradiometer.

The solenoid of our SQUID-magnetometer consists of two parts (see Fig.1): an outer part is the enclosed superconducting solenoid and is used for investigations in fixed magnetic fields up to 300 Oe; an inner part is the ordinary one (open-circuited, but also wound by Nb-Ti superconducting wire) and allows us to measure continuously the magnetisation curves at field-scanning range of  $\pm 30$  Oe around the magnitude fixed by the enclosed solenoid.

The investigated samples were made from the Pb-doped Bi-Ca-Sr-Cu-O ceramic prepared using the mixture  $\text{Bi}_{1.8}\text{Pb}_{0.3}\text{Sr}_{1.9}\text{Ca}_2\text{Cu}_3\text{O}_m + 0.3\text{CaCuO}$  and annealed at  $850^\circ\text{C}$  in the atmosphere for 120 hours<sup>[15]</sup>. The ceramic had the next parameters:  $T_c=106$  K,  $j_c(77\text{ K})= 340$  A/cm<sup>2</sup>, the average granules size  $d\sim 10$   $\mu\text{m}$ . The specimen had

a shape of a cylinder with diameter 4 mm with a co-axial hole 2.5 mm in diameter, the height varied from 1.6 mm to 0.24 mm. The initial height of the sample was 1.6 mm. After a series of measurements in different fields the certain layers were ground from the ends of the cylinder so that its new height became little smaller. And after that a new series of measurements was provided. We failed to make the specimen thickness smaller than 0.24 mm due to the low firmness of a this thin ceramic disk.

To measure  $M_{IR}$  the specimen was first warmed to about 120 K and then cooled in zero magnetic field ( $<4 \cdot 10^{-4}$  Oe) down to 2.2 K. Such a low field magnitude was obtained by compensation of the remanent field. The superconducting lead shield discriminated the external field down to  $\sim 2 \cdot 10^{-2}$  Oe. Cooling the sample at the fixed position at the pick-up system center, we registered a clear diamagnetic signal at temperature  $T_c$  of superconducting transition. The remanent field magnitude was estimated by diamagnetic signal, and after heating again above  $T_c$  the corresponding compensative current (of about 25  $\mu$ A) was applied to enclosed solenoid and was trapped in it. Then the procedure was repeated several times with the compensative field to specify the current magnitude. When the diamagnetic signal became small enough and its magnitude did not decrease after the latest procedures, the compensative current was fixed and then was added everytime when any new value of the magnetic field was set. This permitted us to reduce the remanent field about 50 times.

Then at the fixed temperature of 2.2 K a certain magnitude of the field was applied and after 1 minute the field was switched off (the compensation remains).  $M_{IR}$  corresponding to this applied field was determined moving the sample through the pick-up coils as described e.g. in [16]. The described procedure was repeated many times so that the applied field magnitudes were monotonically increased (every new field was larger than the previous one). It was assumed that the previous field distribution inside the sample must be wiped out by the next field, because the larger is the field the deeper the vortexes penetrate into the superconductor. This suggestion was verified for few experimental points: after measuring  $M_{IR}$  at some applied field the sample was warmed above  $T_c$  and then it was cooled at zero field, and the isothermoremanent magnetisation measurement is repeated for the same applied field magnitude. These successive measurements gave the same magnitudes of  $M_{IR}$ , thus our assumption was confirmed.

## Discussion

The isothermoremanent magnetisation dependence on the applied magnetic field in the range of 1-10 Oe for different heights of rings is presented on a double logarithmic scale in Fig.1. We see that in this range the isothermoremanent magnetic moment is proportional to the applied field to the some power:  $M_{IR} \sim H^\alpha$ , where exponent  $\alpha$  is located between 2 and 3. For the fields below 1 Oe the experimental points are highly spread within this scales owing to small signal value and the correspondingly high relative error in data measurement, and are not shown. There were two main causes of these errors: ferromagnetic impurities introduced upon mechanical processing of the sample, and the "zero" field which an accidental magnitude of about  $H_A \sim 4 \cdot 10^{-4}$  Oe, which causes an error because of the additional magnetic moment  $M_A \sim H_A \cdot V$ , where  $V$  is the volume of the sphere which has approximately the same radius as the specimen. Estimations show that the observed spread in experimental points may be caused only by the field  $H_A$ . For the fields above 10 Oe the  $M_{IR}$  magnitude exceeds the dynamic range of our SQUID-magnetometer; and though we can measure the large magnitudes of magnetic moment, the measurement errors dramatically increase. So we do not present here the experimental data on the fields above 10 Oe.

The exponent  $\alpha$  dependence on the height-to-diagonal ratio  $h/\sqrt{h^2+d^2}$ , where  $h$  is the height and  $d$  is the diameter of the sample, is shown in the Fig.2. The open circles are the experimental data and the black circles are the known limits for the long cylinder ( $\alpha=2$ ) and for the thin disk ( $\alpha=3$ ) mentioned above. Evidently, the long sample limit should be the same for the solid cylinder and for the cylinder with co-axial hole because the penetration depth of the vortices is proportional to the field and in weak fields the central part of the cylinder is of no importance, and the end effects (where the vortices penetrate to the center even in weak fields) are negligible. The thin sample limit should be different for a solid disk and for the ring because the screening currents flow over the whole surface of the sample [7-9] for any weak external applied field and therefore the whole surface is significant. It is evident from the Fig.2 that for the rings with outer diameter 4 mm and the inner diameter 2.5 mm the thin limit differs from three and equals approximately 2.3.

## CONCLUSIONS

The isothermoremanent magnetic moment ( $M_{IR}$ ) of Bi-Ca-Sr-Cu-O ceramic cylindrical rings of different height-to-diameter ratio is investigated by a SQUID magnetometer in low magnetic fields. In the range of fields where the magnetic flux starts to penetrate into the sample,  $M_{IR}$  is found to be proportional to some power of the magnetizing field. The exponent value is found to be dependent on the thickness of the sample and to be between 2 and 3 - the two well-known limits for the long cylinder and thin disk, respectively. The exponent limit for a thin disk with a central hole (thin ring) seems to be about 2.3 which noticeably differs from the limit for a solid disk.

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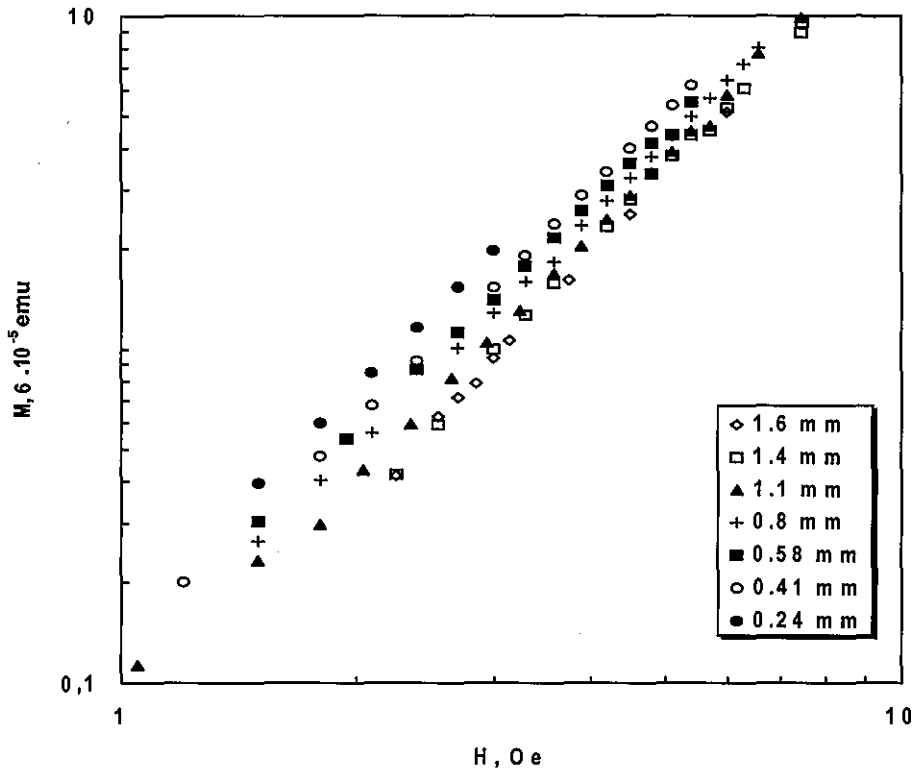


Fig. 1. The remanent magnetisation  $M_{IR}$  dependence on the applied field for the  $\text{Bi}_{1.8}\text{Pb}_{0.3}\text{Sr}_{1.9}\text{Ca}_2\text{Cu}_3\text{O}_m + 0.3\text{CaCuO}$  ceramic rings with the outer diameter 4mm, the inner diameter 2.5mm and different heights.

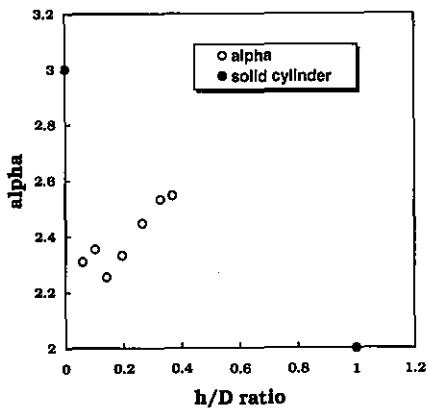


Fig. 2. The exponent  $\alpha$  dependence on the height-to-diagonal ratio of the HTSC-rings.