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# 中国核科技报告

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OXIDE SUPERCONDUCTORS



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# 如何确定高 $T_c$ 氧化物超导体的 $H_{c1}$

## HOW DETERMINING $H_{c1}$ OF HIGH $T_c$ OXIDE SUPERCONDUCTORS

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## 摘 要

长度逐次减短的圆柱形 $\text{Bi}_{1-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ 块状超导体的直流磁化测量已经表明,只要样品长度大于直径, $H_{c1}$ 的测量值与样品长度无关,也就是与退磁因子无关。高 $T_c$ 氧化物的这个行为与磁场在 $H_{c1}$ 以下就穿透进超导体有关。

**关键词** 超导体 临界磁场

# HOW DETERMINING $H_{c1}$ OF HIGH $T_c$ OXIDE SUPERCONDUCTORS

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## ABSTRACT

The magnetizations of cylinder  $\text{Bi}_{1-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}$ , bulk superconductor whose length was shorten step by step had shown that  $H_{c1}$  were independent of the length of cylinder, i. e. independent of the demagnetic factor, as the length was larger than the diameter; that  $H_{c1}$  were dependent of demagnetic factor, as the length was smaller than the diameter. But the demagnetization factor was a constant about  $1/4$  for  $l < d$ .

The type II superconductor has two critical magnetic fields: lower critical field  $H_{c1}$  and upper critical field  $H_{c2}$ . They can be determined from magnetization curve of long cylinder whose demagnetization factor,  $n$ , is close to zero. For an ideal type II superconductor,  $H_{c1}$  is a field at which magnetization peak appears. For a non-ideal type II superconductor,  $H_{c1}$  is defined as a field at which the magnetization curve begins to deviate from linearity. Provided the demagnetization factor is not equal to zero, the above-mentioned field defined is no longer the real lower critical field and is written as  $H'_{c1}$ . The relation between  $H_{c1}$  and  $H'_{c1}$  is given by

$$H_{c1} = \frac{H'_{c1}}{1-n} \quad (1)$$

The expression (1) is undoubtedly correct for the traditional type II superconductors.

$H'_{c1}$  of the traditional ideal or closed to ideal type II superconductor, can be determined by the dependence of critical current  $I_c$  upon the magnetic field which is perpendicular to current.  $H'_{c1}$  is a field corresponding to the linear part in low field to extrapolate to  $I_c=0$ . And the expression (1) is applicable. The demagnetization factor,  $n$ , of cylinder in the parallel field is smaller than  $n_c$  of one in the perpendicular field, i. e.  $n < n_c$ . It can be known from expression (1),  $H'_{c1}(\parallel) > H'_{c1}(\perp)$ . However, we had observed experimentally that both  $H'_{c1}$  determined respectively from magnetization curve and from dependence of  $I_c$  upon magnetic field H was similar, i. e.  $H'_{c1}(\parallel) \approx H'_{c1}(\perp)$ .<sup>[1,2]</sup> The reason to produce this contradictory is not yet ambiguous.

In addition, the volume fraction of superconducting phase in oxide superconductor is smaller, about 30~50% for  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ , about 10~30% for both Bi-Sr-Ca-Cu-O and Tl-Ba-Ca-Cu-O. They are much smaller than traditional superconductor. This fact implies that the distortion of magnetic field led by the diamagnetism of high  $T_c$  oxide superconductors should be smaller than one by traditional superconductor. However, the demagnetization factor,  $n$ , represents the degree of the distortion led by superconductor. In that way, it is an interesting question what is the relation between  $H_{c1}$  and  $n$ .

This letter reports experimental result about the dependence of  $H_{c1}$  upon demagnetic factors in high  $T_c$  oxide Bi-Sr-Ca-Cu-O.

The specimen of nominal composition  $\text{Bi}_{1-x}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}$ , was prepared by solid state reaction. The raw materials are pure  $\text{Bi}_2\text{O}_3$ ,  $\text{PbO}$ ,  $\text{SrCO}_3$ ,  $\text{CaCO}_3$  and  $\text{CuO}$ . The mixture was heated at 830°C for 15 hours. After grinding and pressing, it was sintered at 360°C for 60 hours, then quenched

to room temperature. The X-ray diffraction shows that there is little 85K superconducting phase in the sample. Meissner effect in the field of 15 Oe gives that the volume fraction of 85K phase in superconducting phases is smaller than 8%.

The specimen is a cylinder with the diameter 4mm and the length 16.8mm. The magnetization were measured at 77.3 K by a ballistic galvanometer connected to two opposite series pick-up coils.

The demagnetization factor of cylinder sample with special diameter depends on length,  $l$ . For studying the dependence of  $H_{c1}$  upon demagnetization factor, low field magnetizations of the specimen which was shortened step by step were measured. The magnetization of the specimen for  $l=l_0$ ,  $l=\frac{4}{5}l_0$ ,  $l=\frac{3}{5}l_0$ ,  $l=\frac{2}{5}l_0$ ,  $l=\frac{1}{5}l_0$  and  $l=\frac{1}{8}l_0$  were shown in Fig.1, here  $l_0$  is the initial length of specimen.  $H'_{c1}$  determined from magnetizations of specimen with various length were given in Fig.2. It can be seen that  $H'_{c1}$  do not depend on the demagnetization factor, although the slopes of the magnetization curves below  $H_{c1}$  decrease with the decrease of the length of sample. In fact, there were different  $H'_{c1}$  in two ranges of length, i. e.  $H'_{c1}=28\text{Oe}$  for  $l \geq 2/5l_0$  and  $H'_{c1}=21\text{Oe}$  for  $l \leq 1/5l_0$ . What  $H_{c1}$  do not depend on  $n$  means that  $H_{c1}$  is equal to  $H'_{c1}$ . If the diameter of sample,  $d$ , is employed as a measurement unit of the length, we can conclude that  $H_{c1}$  is independent of demagnetization factor for  $l > d$ , and  $H_{c1}(77\text{K})=28\text{Oe}$  for  $\text{Bi}_{1-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{O}$ , bulk superconductor. As  $l < d$ ,  $H_{c1} = \frac{H'_{c1}}{1-\frac{1}{4}} = 21\text{Oe}$ , i.

e. the demagnetization factor is always equal to about 1/4.

What the slope of magnetization curve below  $H_{c1}$  decreases with the shortening of the length of sample indicates that the magnetization is a volume effect. The area surrounded by the magnetization curve decreased with shortening of the length of sample, as shown in Fig.3. This result indicated also that the magnetization was a volume effect.

The above-mentioned experimental result has shown that  $H_{c1}$  of the oxide Bi-Sr-Ca-Cu-O bulk superconductor is independent of the demagnetization factor. It is related to the flux penetration into sample in the field below  $H_{c1}$ . In the oxide superconductors, there are giant weak links and a portion of them has been penetrated by flux in the field below  $H_{c1}$ . On the other hand, the magnetic shielding signal of zero-field-cooled specimen is considerably smaller than the complete magnetic shielding signal in the field below  $H_{c1}$ , i. e. there only is small superconducting volume fraction in the oxide superconductors. In our specimen, it is about 32%. This fact implies

that the flux density,  $B$ , in the specimen is not equal to zero, i. e. the flux has penetrated into the oxide superconductors in the field lower than  $H_{c1}$ .

It can be also seen from above experimental results that,  $H_{c1}$  changes from 28 Oe to 21 Oe as the length of the specimen decreases from  $2/5l_0$  to  $1/5l_0$ , i. e.  $l_0 \sim d$ . This is a unknown question. It wants yet to be further studied.

### References

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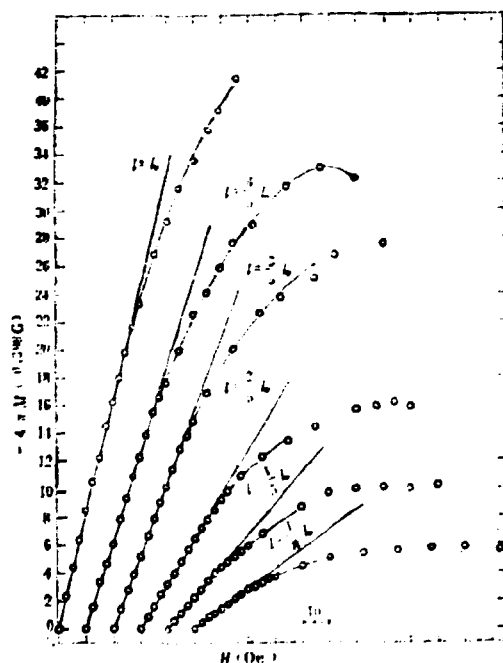


Fig. 1 The magnetizations of cylinder  $\text{Bi}_{1-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$  bulk superconductor with shortening of length in region of low magnetic field,

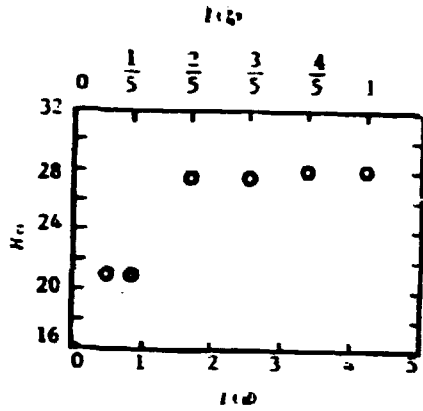


Fig.2 The relation between  $H_{c1}$  and the length of sample

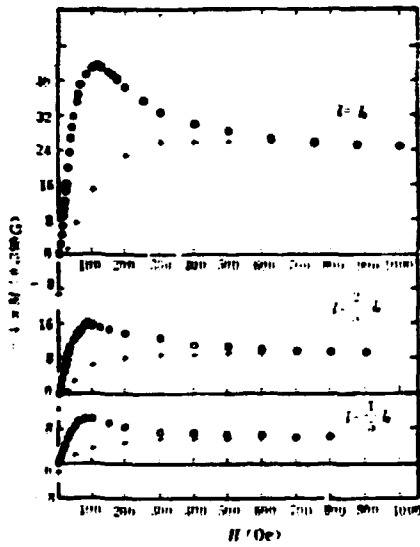


Fig.3 The magnetization curves of  $\text{Bi}_{1-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_7$  bulk cylinder with various length at 77.3 K.



Ⅱ类超导体有两个临界磁场：下临界场 $H_{c1}$ 和上临界场 $H_{c2}$ 。它们可以由退磁因子近似为零的长圆柱体的磁化曲线来确定。对于理想Ⅱ类超导体， $H_{c1}$ 对应于磁化曲线峰值场。换句话说，在 $H_{c1}$ 以下的磁场范围内，磁化和磁场成正比；对于非理想Ⅱ类超导体，磁化达到峰值之前，磁化就已偏离了线性，人们通常把开始偏离线性对应的磁场定义为 $H_{c1}$ 。如果样品的退磁因子 $n$ 不等于零，那么由上述方法所确定的下临界场就不是样品的真正的下临界场，我们把它表示为 $H'_{c1}$ ，它与 $H_{c1}$ 的关系为

$$H_{c1} = \frac{H'_{c1}}{1-n} \quad (1)$$

对于传统超导体，式(1)无疑是正确的。

对于理想或接近理想的Ⅱ类超导体，其 $H_{c1}$ 还可以这样来确定：处于垂直场中的长圆柱体的临界电流 $I_c$ 与磁场 $H$ 的关系，在低场部分是线性的，把它线性部分外推到 $I_c=0$ ，即 $I_c(H)$ 线性部分外推到与 $H$ 轴的交点对应的磁场定义为 $H_{c1}$ ，并且仍可以由式(1)求出 $H_{c1}$ 。长圆柱形超导体在平行场中（磁化测量）的退磁因子小于在垂直场中（ $I_c(H)$ 测量）的退磁因子，即 $n < n_{\perp}$ ，所以 $H'_{c1}(\parallel) > H'_{c1}(\perp)$ 。

此外，在氧化物超导体中，超导相的体积比是较低的，对于 $YBa_2Cu_3O_{7-x}$ 约为30~50%， $Bi-Sr-Ca-Cu-O$ 和 $Tl-Ba-Ca-Cu-O$ 约10~30%。可以看出，远小于传统超导体。这个事实意味着，高 $T_c$ 氧化物超导体的抗磁性所引起的外磁场的畸变也小于传统超导体。但是，所谓退磁因子实际上代表超导体所引起的外场畸变的程度。因此，氧化物超导体的 $H_{c1}$ 与退磁因子之间是什么关系，换言之，它们的 $H_{c1}$ 是否也受退磁因子的影响呢？这显然是一个值得研究的问题。

另外，我们在实验中观察到，对于一个长圆柱体超导体，由磁化曲线 $M(H)$ 和 $I_c(H)$ 所获得的 $H'_{c1}(\parallel) \approx H_{c1}(\perp)$ ，其原因也是不清楚的。

本文报导高 $T_c$ 氧化物超导体 $Bi-Sr-Ca-Cu-O$ 的 $H_{c1}$ 与退磁因子之间的关系的实验结果。

样品用固相反应法制备，名义组分为 $Bi_{1-x}Pb_xSr_2Ca_2Cu_2O_y$ 。纯 $Bi_2O_3$ ， $PbO$ ， $SrCO_3$ ， $CaCO_3$ 和 $CuO$ 粉末充分混合均匀，在830℃预烧15小时。研碎，压成长条状，在860℃烧结60小时，然后淬火到室温。X射线衍射分析表明，以110K高温相为主，但也有少量的85K超导相，Meissner效应测量给出，在15Oe磁场下，85K相所占超导相的体积比小于8%。

样品是一个直径4mm，长度16.8mm的圆柱体，样品的磁化用冲击电计测量。测量在77.3K进行。

一定直径的圆柱形超导体，其退磁因子决定于长度 $l$ ，无限长圆柱体的 $n=0$ 。当长度逐渐减小时，其退磁因子逐渐增大。因此，为了研究退磁因子与 $H_{c1}$ 的关系，我们分别测量了同一样品在不同长度状态的低场磁化。其中包括 $l=l_0$ ， $4/5l_0$ ， $3/5l_0$ ， $2/5l_0$ ， $1/5l_0$ 和 $1/8l_0$ 的低场磁化曲线给在图1（见4页），这里 $l_0$ 是样品的原始长度。由图1可以确定出不同长度状态下的 $H_{c1}$ 值。它与长度 $l$ 的关系表示在图2（见4页）。可以看出，在 $l \geq 2/5l_0$ 的长度范围内，虽然在 $H_{c1}$ 以下的磁化曲线斜率随样品长度的减小而减小，但是 $H_{c1}$ 却与样品长度无关。实际上，在两个长度范围内相应地存在着两个不同的 $H'_{c1}$ 。即 $l \geq 2/5l_0$ 时， $H'_{c1} = 28\text{Oe}$ ； $l \leq 1/5l_0$ 时， $H'_{c1} = 21\text{Oe}$ 。 $H'_{c1}$ 与长度无关意味着 $H_{c1} = H'_{c1}$ 。如果用样品的直径 $d$ 作为量度长度的单位，我们能够作出结论，在 $l > d$ 时， $H_{c1}$ 与退磁因子无关，并且 $Bi_{1-x}Pb_xSr_2Ca_2Cu_2O_y$ 超导体在77K的 $H_{c1} \approx 28\text{Oe}$ 。当 $l < d$ 时， $H_{c1}(77\text{K}) = 21\text{Oe}$ ，相当于它的退磁因子 $n$ 总是等于十。

磁化曲线在 $H_{c1}$ 以下的斜率随样品长度 $L$ 缩短而减小的事实，表明磁化是一个体积效应。磁化曲线下面所包围的面积也是随样品长度的减小而减小，如图3（见5页）所示。这个结果更进一步说明了磁化是一个体积效应。

上述实验结果已经表明，氧化物Bi-Sr-Ca-Cu-O块状超导体的 $H_{c1}$ 与退磁因子无关。这与在 $H_{c1}$ 以下的磁场就已穿透进了氧化物超导体有关。在氧化物超导体中，存在着大量的弱连结，而且它们中的一部分在 $H < H_{c1}$ 的磁场中就被穿透；另一方面，零场冷却样品在 $H_{c1}$ 以下的磁场中的磁屏蔽信号明显地小于完全磁屏蔽信号，也就是氧化物超导体仅具有较小的超导体积比。在我们的样品中大约是32%。这个事实意味着，样品中的磁通密度 $B$ 不等于零，也就是，在 $H_{c1}$ 以下，磁场就已经穿透了氧化物超导体。

上述结果还表明，样品从 $2/5L_0$ 减小到 $1/5L_0$ 时， $H_{c1}$ 从28 Oe减小到21 Oe。产生这一突变的原因是什么，仍然是一个有待解决的问题。

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