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FINAL REPORT

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Study of Some Superconducting and Magnetic Materials

On High T Oxide Superconductors

by

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Submitted by

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#### INTRODUCTION

Superconductivity was accidentally discovered amidst efforts to reach absolute zero nearly 75 years ago. Despite concerted efforts since then to develop high temperature superconductors, it wasn't until 1986 that the superconducting transition temperature  $(T_c)$  was raised above 23.2K, the  $T_c$  of the Nb<sub>3</sub>Ge superconductor discovered<sup>1</sup> in 1973. It was in 1986 that the scientific community started taking nonconventional approaches<sup>2</sup> to the problem, taking advantage of possible nonconventional superconducting mechanisms.<sup>3</sup> This fresh start led to the September 1986 report by Bednorz and Muller<sup>4</sup> that they had found the possible existence of percolative superconductivity in  $(La_{1-x}Ba_x)Cu_{3-6}$  with x=0.2 and 0.15 in the 30K range. Subsequent magnetic studies<sup>5-7</sup> confirmed that high-temperature superconductivity indeed exists in this system.

Takagi and his team<sup>8</sup> attributed the observed superconductivity in the La-Ba-Cu-O (LaBCO) system to the  $K_2NiF_4$  phase.

Further research revealed that  $pressure^{7,9}$  enhances the  $T_c$  of the LaBCO system at a rate greater than  $10^{-3}$ K bar<sup>-1</sup>, raises the onset  $T_c$  to 57K, and results in a zero-resistance<sup>10</sup> state at  $T_c$ =40K.

To mimic the pressure effects, we replaced<sup>7</sup> the Ba atoms in the  $K_2NiF_4$  structural LaBCO with the smaller Sr and Ca atoms. The  $T_c$  of La-Sr-Cu-O (LaSCO) was enhanced to 48.6K and the transition width narrowed<sup>11,12,13</sup> to 2k at ambient pressure. The  $T_c$  of La-Ca-Cu-O (LaCCO), on the other hand, was suppressed to approximately 28K. This relationship suggested the existence of an optimal interatomic distance for high  $T_c$  in the oxides.

These observations led us to the proposition<sup>14</sup> that a  $T_c$  above 77K may exist in compound systems represented generically by the formula  $(L_{1-x}M_x)_aA_bD_y$ , and not necessarily of the pure  $K_2NiF_4$  phase. Working with this theory, we determined that optimal interatomic distances could be achieved when L=Y, M=Ba, A=Cu, and D=O with nominal compositions a=2, b=1, x=0.4, y\leq=4. This led to our discovery of the new superconducting compound system, Y-Ba-Cu-O (YBCO), with  $T_{co}$ =93K and  $T_{c1}$ =80K. By improving the quality of the compounds we have achieved  $T_{co}$ =98K and  $T_{c1}$ =94K in the YBCO multiphased compound system.

Improvements in the quality of the compounds allowed us to achieve  $T_{CO}$ =98K and  $T_{C1}$ =94K in the YBCO multiphased compound system.

Our attempts to understand the new High  $T_c$  superconductivity have enabled us to resistively determine the hydrostatic effects on  $T_c$  in the YBCO compound system up to 19 kbar. The  $T_c$  of YBCO was affected only slightly by pressure, strongly contrasting with observed effects in the  $K_2NiF_4$  phase in LaBCO and LaSCO. This, together with our preliminary x-ray powder diffraction data, suggests that the superconducting YBCO compound system might be very different from the superconducting LaBCO and LaSCO systems with maximum onsets<sup>15</sup> of 48.6K at ambient pressure.

Information on magnetic properties is important in the understanding of recent high  $T_c$  superconductivity breakthroughs. We therefore carried out measurements on the dc magnetic moment of YBCO. We have shown that this material, cooled in zero field or in a high field ( $H_{cool}$ >90G), is diamagnetic below  $T_{cm}$ =90K. This is consistent with previous measurements.<sup>16,17</sup> However, when the sample is cooled in a small field

(<85G), the magnetization first becomes negative (diamagnetic) below T<sub>cm</sub>, but further cooling results in a jump of M to a positive value (paramagnetic) at low temperature. We have also observed the switching by the application of an additional small field when the sample was cooled in a small field.

The work of various research groups has identified the superconducting phase of YBCO as an oxygen defect perovskite corresponding to the composition  $YBa_2Cu_3O_y$ .<sup>18</sup> Extensive studies have found that in this oxide system the yttrium element can be completely substituted by almost all the rare earth elements<sup>19</sup> with very little effect on the T<sub>c</sub>. Complete substitution of the Ba ion by other alkaline earth elements, however, was found to destroy the superconductivity and make the compound a semiconductor. Therefore, it is speculated that there may exist a metal-to-semiconductor phase boundary in the  $R(A_{1-x}B_x)_2-Cu_3-O_y$  system, where R can be any of the trivalent ions and A and B are alkaline earth elements. We have studied R=Y, A=Ba, and B=Sr (YBSRCO).

## EXPERIMENTAL METHOD

The compounds we investigated during the initial 93K T<sub>c</sub> discovery were prepared with nominal compositions of  $(Y_{1-x}Ba_x)_2CuO_{4-\delta}$ , with x=0.4, through solid-state reaction of appropriate amounts of  $Y_2O_3$ , BaCO<sub>3</sub>, and CuO.<sup>7</sup> Our 98K T<sub>c</sub> investigations involved compositions of

 $(Y_{0.6}Ba_{0.4})_2CuO_{4-y}$  and  $(Y_{0.6}Ba_{0.4})_2CuO_{3-y}$ . Bar samples 1mm x 0.5mm x 4mm were cut from the sintered cylinders and a four-lead technique employed for resistance measurements.

A Linear Research ac inductance bridge was used for magnetic susceptibility determinations. Temperature was measured with Au+0.07% Fe-Chromel and Chromel-Alumel thermocouples in the absence of a magnetic field, and with a carbon-glass thermometer in the presence of a field. The latter was calibrated against the former without a field. Magnetic fields of up to 6T were generated by a superconducting magnet.

A pressure environment was provided by a Be-Cu clamp with a fluid medium. Pressure levels were determined with a pressure gauge at room temperature and corrected by subtraction of the loss due to the freezing of the pressure medium, estimated to be about 2 kbar, at temperatures below 180K.

Powder x-ray measurements were carried out with a GE diffractometer having a resolution of 5%.

The YBSRCO samples were prepared from the appropriate mixture of  $BaCO_3$ ,  $SrCO_3$ ,  $Y_2O_3$ , and CuO. The mixture was calcined in a platinum crucible and heated at 930C for 13 hours in a flowing oxygen atmosphere. Bar samples of 3mm x 1mm x 1mm were used for resistivity and magnetic susceptibility measurements. For mid-infrared transmittance measurements a 0.5mm thick disk was made of the mixture of the oxide compound (~5% in weight) and dry KBr was used. IR measurements were made with a Mattson Fourier Transform Infrared Spectrometer. Resistivity and magnetic susceptibility measurements were made in the same manner as previously discussed.

## RESULTS

The temperature dependence of resistivity R, determined in a simple liquid-nitrogen dewar, is shown in figure 1. R initially drops almost linearly with temperature T. A deviation of R from this T dependence is evident at 93K and a sharp drop starts at 92K. A "zero-R" state is achieved at 80K.

The variation of magnetic susceptibility M with T is shown in figure 2. It is evident that a diamagnetic shift starts at 91K and the size of the shift increases rapidly with further cooling. At 4.2K the diamagnetic signal corresponds to 24% of the superconducting signal of a Pb sample with similar dimensions. Resistivity in a magnetic field is shifted toward lower T. At our maximum field of 5.7T, the "zero-R" state remains at a T as high as 40K.

Preliminary x-ray powder diffraction patterns show the existence of multiple phases uncharacteristic of the  $K_2NiF_4$  structure in the samples.

Subsequent refinements in material fabrication have resulted in a T<sub>C</sub> of 98K in both  $(Y_{0.6}Ba_{0.4})_2CuO_{4-y}$  and  $(Y_{0.6}Ba_{0.4})CuO_{3-y}$  with the former having a sharper transition. The temperature dependence of R is shown in figure 3 for two different samples of  $(Y_{0.6}Ba_{0.4})_2CuO_{4-y}$ . It is evident that transitions with T<sub>CO</sub>=98K, T<sub>C1</sub>=94K, and T<sub>CO</sub>=93K, T<sub>C1</sub>=90K have been achieved, respectively, for samples I and II.







The variation of M with T is displayed in figure 4 for sample II. M starts to turn negative at  $T_{CO}$  93K. At 4.2K the diamagnetic ac M reaches a value of approximately 25% of the signal of a superconducting Pb sample of a similar size. However, a dc M measured by a Quantum Design SQUID Magnetometer gave a value of '38% when the sample was zero-field cooled. Both measurements are supposed to determine the upper limit of the Meissner effect since they include the shielding effect. The cause for the difference remains unknown. In the presence of a magnetic field the superconducting transition is suppressed and broadened, as shown in figure 5. At 5.7T,  $T_{C1}$  is shifted down to 70K from 90K, showing that the R=0 state can still be maintained at 70K.

The results of R, M, and magnetic field (H) effects on R demonstrate unambiguously that a sharp superconducting transition with  $T_{CO}$  up to 98K in YBCO, is obtained at ambient pressure. By neglecting the positive curvature at low fields one gets a value of  $dH_{C2}/dT$  near  $T_C$  to be 3T/K or 1.5T/K, depending on whether  $H_{C2}(T_C)$  is taken at the 10% drop of the normal state R or at the 50% drop. In the weak-coupling limit  $H_{C2}(0)$  is thus estimated to be between 94 and 190T for Sample II. This is consistent with more recent high field results.<sup>20</sup> The sample examined consists of more than three phases whose X-ray diffraction patterns do not seem to fit the  $K_2NiF_4$  and ABO<sub>3</sub> structures.

Figure 6 shows typical temperature dependencies of R, normalized to that at 100K for simplicity, at a few pressures. At ambient pressure, R initially decreases with temperature linearly. It starts to deviate from this linear dependence at  $^{93K}$  and a "zero-R" state is reached at 80K.



Figure 4: Temperature Dependence of Magnetic Susceptibility.



7 T



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One may define the superconducting onset temperature  $T_{co}$  and the complete temperature  $T_{cf}$  as the intersection temperatures of the main part of the transition with the linear extrapolation of the R(T) curve and the "zero-R" line, above and below the transition, respectively. Under pressure,  $T_{co}$  is shifted up slightly.  $T_{cf}$ , under pressure, is first shifted up, then down, as shown in figure 7. The observation is reversible during pressure cycling when proper precautions are taken. The midpoint of the transition, i.e. where R(T)/R(100 K) = 0.5, changes only slightly. This is in strong contrast to that observed<sup>7,9,10</sup> previously in the  $K_2NiF_4$  phase of the LaBCO and LaSCO systems with amaximum  $T_{co}$  48.6K at ambient pressure. The pressure effects on these systems are represented by a dashed line in figure 7 for comparison.

As discussed earlier, the YBCO samples have multiple phases devoid of  $K_2 NiF_4$  structure, a narrow transition beginning at  $T_{CO}$ =95K, and, when the samples are cooled in zero field, M is diamagnetic below  $T_{CM}$  and the susceptibility below ~25K reaches 35% of perfect diamagnetism.

We have also measured M when the sample is cooled in a field,  $H_{cool}$ . figure 8 shows the magnetization obtained at various  $H_{cool}$  values. Clearly, M is diamagnetic right below  $T_{cm}$  at all  $H_{cool}$  values employed. The 30-G data (denoted by solid circles) shows a jump from a negative value at 87K to postive at 85K and an increase in M with lowering temperature. For  $H_{cool}$ =40G the switching takes place at 83K. For high  $H_{cool}$  values, as shown in figure 9, the switching temperature,  $T_s$ , moves lower. The dependence of the switching temperature on  $H_{cool}$  is shown in the inset. For  $H_{cool}$ =87G the switching disappears. Aside from the



Tc(K)

SI



Figure 8: Magnetization vs. H<sub>cool</sub>.



Figure 9: Magnetization vs. H<sub>cool</sub>.

switching, the data for warming is identical to those for cooling within our experimental errors. As shown in figure 9, M at low temperatures is much greater than the paramagnetic magnetization above  $T_{cm}$ .

The temperature dependence of resistivity in the  $Y(Ba_{2-x}Sr_x)_2^{-Cu_3^{-O_y}}$ compounds is displayed for different x values in figure 10. The results for the x=0 sample are similar to those reported for a single phase  $YBa_2Cu_3O_y$ , but the room temperature resistivity of the compound is found to increase with Sr content. A sudden rise in resistivity occurs at  $x^1.0$ . However, the typical characteristic of the temperature dependence of resistance in a semiconductor appears for  $x^1.4$ , as shown in figure 10. The results indicate that a metal-to-semiconductor transition appears in the  $YBa_{2-x}Sr_xCu_3O_y$  compound system with the critical concentration near x=1.4.

Anomalous resistance behavior is observed near critical concentration. The resistance first increases, then decreases, on cooling with the peak resistance temperature at 20 for x=1.4, the temperature at which resistance exhibits a peak decrease as x increases. The superconducting transition temperature decreases monotonically with x. For x>1.5 the compound is no longer superconducting.



6 T

## DISCUSSION

The above results demonstrate unambiguously that superconductivity occurs in the YBCO system with a transition between 80K and 93K. We have determined the upper critical field  $H_{c2}(T)$  resistively. If the positive curvature at very low fields is neglected, one gets a value of  $dH_{c2}/dT$ near  $T_c$  of 3T/K or 1.3T/K, depending on whether  $H_{c2}(T_c)$  is taken at the 10% or the 50% drop from the normal-state R. In the weak-coupling limit,  $H_{c2}(0)$  is estimated to be between 80T and 180T in the YBCO system investigated. We believe that the value of  $H_{c2}(0)$  can be further enhanced as the materials are improved. The paramagnetic limiting field at 0K for a sample with a  $T_c$  90K is 165T. Because of the porous and multiphase characteristics of the samples it is difficult to extract any reliable information about the density of states from the slope of  $H_{c2}(T)$ 

The size of the diamagnetic signal (M) provides useful information about the nature of the superconducting state as well as the quality of the samples examined. It should be noted that M gives different meanings when measured by different modes. For instance, ac M, which includes the ac shielding, gives only the upper limit of the Meissner effect. Dc M, which is determined on zero-field cooling and includes the dc shielding (or persistent current shielding), also represents only the upper limit of the Meissner effect. Only the dc M determined on field-cooling provides a real Meissner effect measurement. Possible field trappings in ceramic samples like the high  $T_c$  oxides require the real Meissner effect

measurements performed with extreme caution. Unfortunately no detailed information has been given concerning the M-measurements on recent high  $T_c$  oxide experiments, except one.<sup>5</sup>

Careful examination of data on LaBCO and LaSCO available from various laboratories indicate that no experiment to date has detected 100% Meissner effect. Only 100% shielding has been achieved, despite the general claim of bulk superconductivity. The less than 100% Meissner effect can be attributed to 1) the highly anisotropic nature of the  $K_2NiF_4$  phase of the LaBCO and LaSCO, 2) unusual excitations giving rise to high  $T_c$  superconductivity associated with an anisotropic geometry of the active components in the samples and/or 3) poor quality of the samples. More careful studies of this kind are clearly in order.

Many models have been proposed to account for the high  $T_c$  in the layer-like  $K_2NiF_4$  phase of LaBCO and LaSCO. Band structure calculations<sup>21,22</sup> show that conduction bands form only in the Cu-O octahedron layers due to the strong hybridization of the Cu 3d and O 2p orbitals and the Fermi energy Ef lies near a van Hove singularity for incipient Peierls instabilities. The high  $T_c$  was then attributed to the high electron density of state N(Ef) and soft-modes associated with the van Hove. The high vibrational frequency of the oxygen bond-stretching modes were also considered<sup>22</sup> to be important. Since N(Ef) for LaSCO is larger than that for the superconducting Ba-Pb-Bi-O, it was then argued<sup>11</sup> that a  $T_c$  30K is explained by electron-phonon interaction by scaling. However, it should be pointed out that the cause for a  $T_c$  13K in Ba-Pb-Bi-O remains an open question.<sup>23</sup> In addition, no theory to date based on electron-phonon interaction alone has predicted a  $T_c$ >40K.

Recent gap studies<sup>24</sup> also point to possible deficiencies in the model. Enhanced superconducting interaction giving rise to a high  $T_c$  near metal-semiconductor interfaces has been previously proposed.<sup>3</sup> The added electron-pairing interactions can arise from the virtual exchange of excitons, for instance. Negative U-center interaction has also been proposed<sup>25</sup> in this type of materials system.

It was recently  $proposed^{26}$  that, in a two-dimensional lattice, if the antiferromagnetic interactions between the next-nearest neighbors is strong enough to frustrate the Neel state, and, if the virtual phonon interaction is not strong enough for a spin-Peierls instability, a resonating-valence-bond (RVB) state can exist with nearest magnetic singlet pair formation. In a metallic system these pairs become charged superconducting pairs. Such a model appears to be consistent with our observation of a Curie-Weiss behavior of the YBCO above  $T_{CO}$  with a clear indication of the existence of an antiferromagnetic interaction, if one assumes that the interaction is not due to the non-superconducting phase.

Pairing between the two nearest neighbors in polarons has been suggested,<sup>27</sup> leading to possible transitions among insulating, metallic, and superconducting phases. Recently, it was further proposed<sup>28</sup> that a Bose-Einstein condensation of bipolarons may give rise to infinity conductivity and large diamagnetism at high temperature. The oxygen deficient superconducting LaBCO, LaSCO and YBCO have been considered to be potential candidates for such a mechanism.

In order to investigate the swtiching, we have cooled the sample in  $H_{cool}$  and then increased the field. As shown in figure 11, when the sample is cooled to 5K in  $H_{cool}$ =30G, M is positive (data denoted by open squares). However, as H is increased to 40G, M switches to a negative value and becomes even more negative as H increases. Similar results have been found for  $H_{cool}$ =30G, T=20K, (denoted by open circles),  $H_{cool}$ =40G, T=5K, and 35K, and  $H_{cool}$ =50G, T=5K, and 35K. However, for  $H_{cool}$ =100G, T=35K, M is always negative (denoted by solid triangles). For comparison, the zero-field cooled data (denoted by shaded squares) are also displayed. They are more negative than those when the sample is field-cooled. In the inset, we demonstrate the details of the swtiching as the sample is cooled to 20K at  $H_{cool}$ =40G, and then an increment field between 4.0G and 4.5G. Similar switching phenomena have been observed at different  $H_{cool}$  and temperatures.

We would like to point out that the switchings observed are unique to  $Y_{1.2}Ba_{0.8}CuO_{4-\delta}$  because we have not observed any switching phenomena for other ceramic superconductors,  $(La_{1-x}Ba_x)_2CuO_{4-\delta}$  and  $(La_{1-x}Sr_x)_2CuO_{4-\delta}$ , using the same magnetometer.

X-ray diffraction patterns show that the  $YBa_2Cu_3O_y$ -type phase is the major phase in the samples studied except for x>1.5. Detailed analysis of the x-ray data for high Sr concentrations is currently underway. Preliminary IR transmittance results indicate an absorption at ~1380cm<sup>-1</sup> (0.17ev) for the  $YBa_2Cu_3O_y$  sample, as shown in figure 12. As the Sr concentration increases an absorption peak for x=0.6 is observed at ~1250 cm<sup>-1</sup>. The rate of change in the absorption energy is about the







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same as the decrease in  $T_c$ . For samples with x>1.5, the absorption peak is undetectable. These results suggest that the excitation observed may be related to the high temperature superconductivity in this compound system. More detailed measurements on IR spectra for all the compositions and at low temperature are ongoing. It should be noted that a similar absorption at  $450 \text{ cm}^{-1}$  has been reported in the  $\text{La}_{1.85}\text{Sr}_{.15}\text{CuO}_4$ compound.

## CONCLUSIONS AND FUTURE PLANS

On the basis of existing data it appears that high-temperature superconductivity above 77K reported here occurs only in compound systems consisting of a phase other than the  $K_2NiF_4$  phase.

We have obtained a narrow superconducting transition with  $T_{CO}^{=98K}$ and  $T_{C1}^{=94K}$  in YBCO. Preliminary results indicate that YBCO is rather different from the layered LaBCO, LaSCO, and LaCCO. While electron-phonon interaction cannot be absent from this compound system, non-conventional enhanced superconducting interactions due to interfaces, RVB states, etc., or even a superconducting state beyond the BCS framework, may be required to account for the high  $T_C$  in YBCO. We believe that study of the possible subtle correlation between magnetism and superconductivity will definitely provide important insight into the superconducting mechanism in YBCO and other oxides.

In a recent theoretical paper, Ebner and Stroud<sup>29</sup> investigated the diamagnetic response of weakly linked superconducting clusters. They have shown that the magnetization could be positive or negative depending on H. Our sintered powder sample might consist of weakly linked superconducting clusters; hence, the magnetization could be positive. Additionally, as pointed out by these authors, a large frustrated cluster may, in principal, choose among numerous competing, low, nearly-degenerate states, as in the case of a spin glass,<sup>30</sup> and the switching might be the result of the cluster hopping from one configuration to another. Understanding this unexpected switching phenomena will undoubtedly require more experimental and theoretical work.

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