AFFIDAVIT OF RULING MENG

THE STATE OF TEXAS)
COUNTY OF HARRIS)

On this the 6th, day of March, 2006, before me, a Notary Public in and for the State of Texas, personally appeared Ruling Meng, who, being duly sworn, upon oath deposes and says:

That I am of full legal age, have never been convicted of a felony or a crime involving moral turpitude and am in all respects competent to make this affidavit; that I reside in Houston, Harris County, Texas and that my business address: Superconductivity Center, Houston Science Center, Houston, Texas 77204.

- I am a material scientist. Before I came to the U.S. I was a research Scientist at The Chinese Academy science, Institute of Physics in Beijing, China.
- I started my research on superconducting materials since 1976.
- In 1979 I was invited by Dr.C.W. Paul Chu to join to his group as a visiting scholar in the Department of Physics at the University of Houston until 1981.
- Before I returned to China, in 1982, I was invited by Prof. Ernst Bucher at University of Konstanz, Germany and spent half year in their lab. My research projects were superconductor's single crystal growth. During this period I continued the collaboration with Dr. Chu. By sending him superconducting Single crystal for his studies.
- In mid 1982 I return to the Institute of physics in Bejing China and continued the collaboration on superconducting materials with Dr.C.W. Paul Chu
- In July of 1984 I was invited again by Dr. Chu and came back to the UH. and joined his group as a visiting scientist. My responsibility in this group was to research and study superconducting and related materials. I synthesized and characterized various compounds myself independently and supervise students on their materials studies.
- In 1986, Dr.C.W.Chu was on leave and served at the National Science Foundation in Washington D.C. He only came back Houston on

weekend to discuss research activity in the lab. At mid-November of 1986 I learned from my friend in China, Professor Z.X. Zhao about the paper "Possible High T_c Superconductivity in the Ba – La – Cu – O System," by J.G. Bednorz and K.A. Muller. I asked a graduate student Li Gao to go to the library to copy the paper on a Friday afternoon. Then I put a copy of the paper on Dr. Chu's desk. [Reference 1, on the paper with my hand writing note] On Saturday morning I came in and discussed the paper with him. The paper reported that they used the wet-chemical method and it also indicated that the solid-state reaction processing might be difficult to obtain the superconducting phase. I told Dr. Chu that I believed the solid-state reaction method could be used to synthesize this material. My expertise was not in wet-chemistry and I felt more confident with using the solid-state method.

- After this discussion, on the 14th of November I began to synthesize the first LaBaCuO compound with composition La_{4.25}Ba_{0.75}Cu₅O₃. And successfully repeated Dr.Bednorz and Muller's results. [Reference 2]
- In late November we began to apply pressure to the La-Ba-Cu-O compounds. The transition temperature increased from 35 to 57 K and leveled off. We realized that simulating the pressure effect we would have to substitute a small size element for Ba. Therefore, we began to substitute Sr for Ba in early December. However Dr. Chu asked me to stop the Sr substitution experiment in our lab because he had asked M.K. Wu (Alabama) to do this. He said that M.K.Wu couldn't compete with us. I therefore to replace Ba with Ca which has a smaller atoms size than Sr. at the same time, we learned that Bell lab has reported that LaCaCuO display a transition temperature which was lower than LaSrCuO. During this time Dr. Chu called back from Washington, D.C. every day to ask about the new results and set up the contact with outside corporations.
- On November 25th We observed a transition temperature around 70 K in a LaBaCuO sample made by Pei Hor. Unfortunately, the sample was not stable and we would not repeat the results. The results indicated that there were higher temperature superconductors existed.
- On December 30th Dr. M.K. Wu and his student brought his sample La-Sr-Cu-O to Houston for magnetic measurements. During his visit we had a discussion among Pei Hor, M.K. Wu, Li Gao and myself in Pei's office on Jan 1st (or 2 rd) 1987. Since the LaSrCuO indeed displayed a higher transition temperature at 40-45 K. while the Ca

substituted lower the transition temperature, we realized that we should substitute La by other elements. During the discussion Pei Hor suggested that we should replace La with Y (Yttrium). I suggested that we also replace La By Lu (Lutetium) because it has a smaller atomic radius.

- Two days later (around 3 or 4 of January) M.K. Wu returned to Alabama. Before his leaving our lab, I suggested him to go back to Alabama NASA 's lab to get Y₂O₃ and made the Y substitution compound. This could speed up the process, because we do not have Y₂O₃ and it if I placed the order the next day, it would take two weeks for us to get our order.
- From December 1986 to January 1987, I continued studying the La BaCuO compounds to optimize the composition and preparation condition. On January 11th we obverses a transition temperature up to 90K at the LaBaCuO sample that I made with formulas (La_{0.4}Ba_{0.6})₂CuO₄ (J2). [Reference 3] and X-ray data shown almost pure 123 structures. Unfortunately, I do not know the 123 phase yet and misidentify as 214 phase with impurity phase. Again the sample was not stable. [Reference 3,4].
- On January 13 we received our order of Y₂O₃ and Lu₂O₃ and then we began to work on substituting La with Y. But most of my effort still on LaBaCuO in order to repeat the 90 K result and I was under great pressure of providing outside groups with L:aBaCuO samples.
- In 29th of January 1987 Dr.Chu received a phone call from M.K.Wu said that he got a sample with a transition temperature up to 90 K and Dr. Chu asked him to bring the sample to our lab. For magnetic measurement to confirm the results.
- The sample with 90K is YBaCuO was mix phase and was in green color.
- For future application and academic study, it is important to separate the mix phase and identify the phase which contribute to the high temperature superconducting. By 27th of Feb. I successfully separate black and green crystals from the mixed phase by studied a set of YBCO sample with difference composition [reference 5].Pei Hor and students did the magnetic measurement and identified the green phase was non-superconducting with rich Y content. And the phase contributed to superconducting is Black color YBa_{1.8}Cu₃O. This sample was sent to Dr. H.k.Mao at Geophysical Laboratory in

- Washington. D.C for the crystal structure characterization and identifying the correct formula.
- In March 8th 1987, based on our green and black samples and the information we provided Dr.Robert M Hazen and H.K Mao at Geophysical Laboratory at Washington identify the high temperature superconductor YBCO 's formula is :YBa2Cu3O6.7 with tetragonal structure.(So call 123 phase).
- Within half months from Feb.22th to March 15th. I had successful synthesized the entire substitute La by rare earth elements RBaCuO (R= rare earth) except Preseodymium and Cerium.
- In 1987, I attended a meeting with Dr.Chu, Pei Hor and attorney Charles M. Cox. During the meting Charles Cox asked who actually suggested to replace La by Y? Dr. Chu immediately Point to me and said Ruling, remember I call you and told you to replace La by Y. I was shocked and did not know what to told. Because I clearly remember it was Hor suggested to substitute La by Y. during the discussion and Dr. Chu was not in the discussion. I am his employee and do not want to make him mad and instead stated "I do not remember."
- May be a couple of months later (I could not remember the exact date). Charles Cox called me and asked me again. that whether I remember Dr.Chu had called and directed me to replace La by Y. I did not answer and he said if you would not identify Dr.Chu told to you about his idea about the substitution of La by Y. Then the University of Houston would lose the patent to University of Alabama. I immediately said no this was our (UH) idea and they learned from us. Then Charles said then we need to identify Dr. Chu, he represents UH. I knew, if I say yes! I was lying. I asked Cox. whether I had to go to court? He said, "No, no, you would not go to the court" I knew this patent should be ours (UH). I would do every thing to help UH to earn the patent. Therefore, I said, "maybe Dr. Chu had called me and talk about the substitution."
- One day, unexpectedly, Charles brought me the declaration writing by him or Chu and asked me to sign. All the detail and date was written and by Cox or Chu all I did was signing my signature. After that Dr. Chu thanked me for helping him in the critical time.
- In 1993, I was asking to make a deposition for U.H against the Uni. Of Alabama. I do not have any choice but continued to lie by

testifying Dr. Chu had called and told me about the substitution of La with Y. I feel very uncomfortable about this. But I think I did it for UH. Beside this all of my other statements in the deposition are truth. Indeed, in the pasts between 1986 to 1990 I made numerous high temperature Superconducting materials, helped to collect data and thou provided evidence for the patent,. I always thought the patent on high temperature superconductors was the join effort of our group.

- I did make a lie about the discovery of YBCO. I was a law illiterate. In 1987 I came to U.S from China where we could only obeying to the head of group, and the leader of the party. Whatever they said was the law. I do not understand any thing about declaration or deposition. I thought I did for the University. In addition I was Dr. Chu's employee and I trust him. and do not want to make him mad of me if not lying.
- However, no matter what was the reason, I should not have lied. The guilty feeling haunted me for about 20 years. Due to my health situation. I am planning to retire soon, I do not want to carry this guilty feeling to my retirement life. I want to have a peaceful mind. After a long mind struggling. Finally on Jan. 2006, I went to tell Pei Hor about how Cox asked me to testified Dr, Chu had called me and asked me to substitute La with Y. I apologized to him for what I have been wrong doing. I also told him that recently Dr.Chu mention our patent will be issued soon. Hor asked me, do you know we are also inventors? I said I think we should but I do not know, I never asked. Hor suggested me to find out. I went to asked John P. Warren the Associate Vice Chancellor for intellectual property management about the inventorship for the YBCO patent, and found out that Dr. Chu is a solo inventor for U.H. YBCO patent. We were shocked, disappointed and angry. We decided to talk to Mr. Warren and let him know that we are fully justified as this patent's inventors. We required and deserved fair treatment.

Dr.Chu came back from Hong Kong the next week, He want to talk to me about this matters. I was sick and do not want to talk to him. On Feb. 9th Dr. Jacobson (the director for Texas Center for Superconductivity at the University of Houston) talked to me about this issue and he said that Dr. Chu said Pei and I are not the some case and should be treated differently. Dr. Chu is willing and prepared to compensate me as recognition for my contribution to the high temperature superconductor invention.

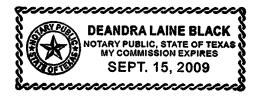
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SUBSCRIBED AND SWORN TO BEFORE ME this 6^{th} day of March

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Possible High T_c Superconductivity in the Ba-La-Cu-O System

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Metallic, oxygen-deficient compounds in the Ba-La-Cu-O system, with the composition $Ba_xLa_{5-x}Cu_5O_{5(3-y)}$ have been prepared in polycrystalline form. Samples with x=1 and 0.75, y>0, annealed below 900 °C under reducing conditions, consist of three phases, one of them a perovskite-like mixed-valent copper compound. Upon cooling, the samples show a linear decrease in resistivity, then an approximately logarithmic increase, interpreted as a beginning of localization. Finally an abrupt decrease by up to three orders of magnitude occurs, reminiscent of the onset of percolative superconductivity. The highest onset temperature is observed in the 30 K range. It is markedly reduced by high current densities. Thus, it results partially from the percolative nature, bute possibly also from 2D superconducting fluctuations of double perovskite layers of one of the phases present.

I. Introduction

"At the extreme forefront of research in superconductivity is the empirical search for new materials" [1]. Transition-metal alloy compounds of A 15 (Nb₃Sn) and B 1 (NbN) structure have so far shown the highest superconducting transition temperatures. Among many A 15 compounds, careful optimization of Nb—Ge thin films near the stoichiometric composition of Nb₃Ge by Gavalev et al. and Testardi et al. a decade ago allowed them to reach the highest T_c = 23.3 K reported until now [2, 3]. The heavy Fermion systems with low Fermi energy, newly discovered, are not expected to reach very high T_c 's [4].

Only a small number of oxides is known to exhibit superconductivity. High-temperature superconductivity in the Li-Ti-O system with onsets as high as 13.7 K was reported by Johnston et al. [5]. Their x-ray analysis revealed the presence of three different crystallographic phases, one of them, with a spinel structure, showing the high T_c [5]. Other oxides like perovskites exhibit superconductivity despite their small carrier concentrations, n. In Nb-doped SrTiO₃, with $n = 2 \times 10^{20}$ cm⁻³, the plasma edge is below the highest optical phonon, which is therefore unshielded

[6]. This large electron-phonon coupling allows a T_c of 0.7 K [7] with Cooper pairing. The occurrence of high electron-phonon coupling in another metallic oxide, also a perovskite, became evident with the discovery of superconductivity in the mixed-valent compound BaPb_{1-x}Bi_xO₃ by Sleight et al., also a decade ago [8]. The highest T_c in homogeneous oxygen-deficient mixed crystals is 13 K with a comparatively low concentration of carries $n = 2-4 \times 10^{21}$ cm⁻³ [9]. Flat electronic bands and a strong breathing mode with a phonon feature near 100 cm⁻¹, whose intensity is proportional to T_c , exist [10]. This last example indicates that within the BCS mechanism, one may find still higher T_c 's in perovskite-type or related metallic oxides, if the electron-phonon interactions and the carrier densities at the Fermi level can be enhanced further.

Strong electron-phonon interactions in oxides can occur owing to polaron formation as well as in mixed-valent systems. A superconductivity (metallic) to bipolaronic (insulator) transition phase diagram was proposed theoretically by Chakraverty [11]. A mechanism for polaron formation is the Jahn-Teller effect, as studied by Höck et al. [12]. Isolated Fe⁴⁺, Ni³⁺ and Cu²⁺ in octahedral oxygen environment

show strong Jahn-Teller (J.T.) effects [13]. While SrFe(VI)O₃ is distorted perovskite insulator, LaNi(III)O₃ is a J.T. undistorted metal in which the transfer energy b_{π} of the J.T. c_{π} electrons is sufficiently large [14] to quench the J.T. distortion. In analogy to Chakraverty's phase diagram, a J.T.-type polaron formation may therefore be expected at the borderline of the metal-insulator transition in mixed perovskites, a subject on which we have recently carried out a series of investigations [15]. Here, we report on the synthesis and electrical measurements of compounds within the Ba - La - Cu - O system. This system exhibits a number of oxygen-deficient phases with mixed-valent copper constituents [16], i.e., with Enerant electronic states between the non-J.T. Cu³⁺ and the J.T. Cu2+ ions, and thus was expected to have considerable electron-phonon coupling and metallic conductivity.

II. Experimental

1. Sample Preparation and Characterization

Samples were prepared by a coprecipitation method from aqueous solutions [17] of Ba-, La- and Cu-nitrate (SPECPURE JMC) in their appropriate ratios. When added to an aqueous solution of oxalic acid as the precipitant, an intimate mixture of the corresponding oxalates was formed. The decomposition of the precipitate and the solid-state reaction were performed by heating at 900 °C for 5 h. The product was pressed into pellets at 4 kbar, and reheated to 900 °C for sintering.

2. X-Ray Analysis

X-ray powder diffractograms (System D 500 SIE-MENS) revealed three individual crystallographic phases. Within a range of 10° to 80° (20), 17 lines could be identified to correspond to a layer-type perovskite-like phase, related to the K2NiF4 structure (a=3.79 Å and c=13.21 Å) [16]. The second phase is most probably a cubic one, whose presence depends on the Ba concentration, as the line intensity decreases for smaller x(Ba). The amount of the third phase (volume fraction > 30% from the x-ray intensities) seems to be independent of the starting composition, and shows thermal stability up to 1,000 °C. For higher temperatures, this phase disappears progressively, giving rise to the formation of an oxygen-deficient perovskite (La₃Ba₃Cu₆O₁₄) as described by Michel and Raveau [16].

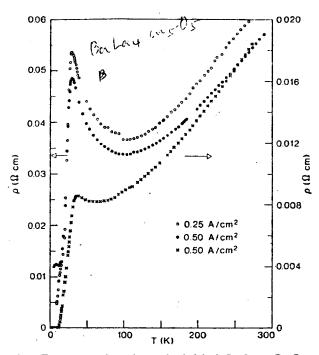


Fig. 1. Temperature dependence of resistivity in $Ba_x La_{5-x} Cu_5 O_{5(3-y)}$ for samples with x(Ba) = 1 (upper curves, left scale) and x(Ba) = 0.75 (lower curve, right scale). The first two cases also show the influence of current density

3. Conductivity Measurements

The dc conductivity was measured by the four-point method. Rectangular-shaped samples, cut from the sintered pellets, were provided with gold electrodes and contacted by In wires. Our measurements between 300 and 4.2 K were performed in a continuous-flow cryostat (Leybold-Hereaus) incorporated in a computer-controlled (IBM-PC) fully-automatic system for temperature variation, data acquisition and processing.

For samples with $x(Ba) \le 1.0$, the conductivity measurements, involving typical current densities of 0.5 A/cm², generally exhibit a high-temperature metallic behaviour with an increase in resistivity at low temperatures (Fig. 1). At still lower temperatures, a sharp drop in resistivity (>90%) occurs, which for higher currents becomes partially suppressed (Fig. 1: upper curves, left scale). This characteristic drop has been studied as a function of annealing conditions, i.e., temperature and O₂ partial pressure (Fig. 2). For samples annealed in air, the transition from itinerant to localized behaviour, as indicated by the minimum in resistivity in the 80 K range, is not very pronounced. Annealing in a slightly reducing atmosphere, however, leads to an increase in resistivity and a more pronounced localization effect. At the same time, the onset of the resistivity drop is shifted

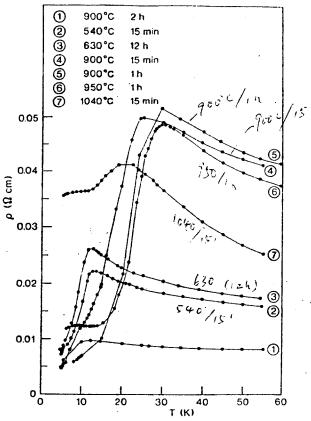


Fig. 2. Low-temperature resistivity of samples with x(Ba) = 1.0, annealed at O_2 partial pressure of 0.2 bar (curve ①) and 0.2×10^{-4} bar (curves ② to ⑦)

towards the 30 K region. Curves (4) and (5), recorded for samples treated at 900 °C, show the occurrence of a shoulder at still lower temperature, more pronounced in curve 6. At annealing temperatures of 1,040 °C, the highly conducting phase has almost vanished. As mentioned in the Introduction, the mixed-valent state of copper is of importance for electron-phonon coupling. Therefore, the concentration of electrons was varied by the Ba/La ratio. A typical curve for a sample with a lower Ba concentration of 0.75 is shown in Fig. 1 (right scale). Its resistivity decreases by at least three orders of magnitude, giving evidence for the bulk being superconducting below 13 K with an onset around 35 K, as shown in Fig. 3, on an expanded temperature scale. The latter figure also shows the influence of the current density, typical for granular compounds.

III. Discussion

The resistivity behaviour of our samples, Fig. 1, is qualitatively very similar to the one reported in the Li-Ti-O system, and in superconducting

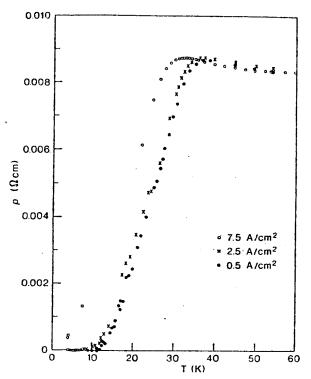


Fig. 3. Low-temperature resistivity of a sample with x(Ba) = 0.75, recorded for different current densities

BaPb_{1-x}Bi_xO₃ polycrystalline thin films [5, 18]. Upon cooling from room temperature, the latter exhibit a nearly linear metallic decrease of $\rho(T)$, then a logarithmic type of increase, before undergoing the transition to superconductivity. One could, of course, speculate that in our samples a metal-to-metal structural phase transition occurs in one of the phases. The shift in the drop in $\rho(T)$ with increasing current density (Fig. 3), however, would be hard to explain with such an assumption, while it supports our interpretation that we observe the onset of superconductivity of percolative nature, as discussed below. In BaPb_{1-x}Bi_xO₃, the onset of superconductivity has been taken at the resistivity peak [18]. This assumption appears to be valid in percolative systems, i.e., in the thin films [18] consisting of polycrystals with grain boundaries, or when different crystalline phases with interpenetrating grains are present, as found in the Li-Ti-O [5] or in our Ba-La-Cu-O system. The onset can also be due to fluctuations in the superconducting wave functions. We assume one of the Ba-La-Cu-O phases exhibits this behaviour. Therefore, under the above premises, the peak in $\rho(T)$ at 35 K, observed for an x(Ba) = 0.75 (Fig. 1), has

to be identified as the start to superconductive cooperative phenomena in the isolated grains. It should be noted that in granular Al. Cooper pairs in coupled grains have been shown to exist already at a point where $\rho(T)$ upon cooling has decreased by only 20% of its highest value. This has been proven qualitatively [19] and more recently also quantitatively [20] by the negative frequency shift occurring in a microwave cavity. In 100 Å films, a shoulder in the frequency shift owing to 2D fluctuations was observed above the T_c of the grains. In our Ba-La-Cu-O system, a series of layer-like phases with considerable variety in compositions are known to exist [16, 21], and therefore 2D correlations can be present.

The granularity of our system can be justified from the structural information, and more quantitatively from the normal conductivity behaviour. From the former, we know that more than one phase is present and the question arises how large are the grains. This can be inferred from the logarithmic fingerprint in resistivity. Such logarithmic increases are usually associated with beginning of localization. A most recent example is the Anderson transition in granular Sn films [22]. Common for the granular Sn and our samples is also the resistivity at 300 K, lying in the range of 0.06 to 0.02 Ω cm, which is near the microscopic critical resistivity of $\rho_c = 10 L_0 \hbar/e^2$ for localization. From the latter formula, an interatomic distance L_0 in the range of 100 Å is computed, thus a size of superconducting grains of this order of magnitude must be present. Upon cooling below T_c , Josephson junctions between the grains phaselock progressively [23] and the bulk resistivity gradually drops to zero by three orders of magnitude, for sample 2 (Fig. 1). At larger current densities, the weaker Josephson junctions switch to normal resistivity, resulting in a temperature shift of the drop, as shown in Fig. 3. The plateau in resistivity occurring below the 80% drop (Fig. 1) for the higher current density of 0.5 A/cm², and Fig. 2 curve 6) may be ascribed to switching of junctions to the normal state.

The way the samples have been prepared seems to be of crucial importance: Michel et al. [21] obtained a single-phase perovskite by mixing the oxides of La and Cu and BaCO₃ in an appropriate ratio and subsequent annealing at 1,000 °C in air. We also applied this annealing condition to one of our samples, obtained by the decomposition of the corresponding oxalates, and found no superconductivity. Thus, the preparation from the oxalates and annealing below 950 °C are necessary to obtain a non-perovskite-type phase with a limited temperature range of stability exhibiting this new behaviour. The formation of this phase at comparatively low temperatures is favoured by the intimate mixture of the compo-

nents and the high reactivity of the oxalates owing to the evolution of large amounts of H₂O and CO₂ during decomposition.

IV. Conclusion

In the concentration range investigated, compounds of the Ba-La-Cu-O system are metallic at high temperatures, and exhibit a tendency towards localization upon cooling. Samples annealed near 900 °C under reducing conditions show features associated with an onset of granular superconductivity near 30 K. The system consists of three phases, one of them having a metallic perovskite-type layer-like structure. The characterization of the new, apparently superconducting, phase is in progress. An identification of that phase may allow growing of single crystals for studying the Meissner effect, and collecting specific-heat data to prove the presence of high T_c bulk superconductivity.

The authors would like to thank H.E. Weibel for his help in getting familiar with the conductivity measurement system, E. Courtens and H. Thomas for discussions and a critical reading of the manuscript.

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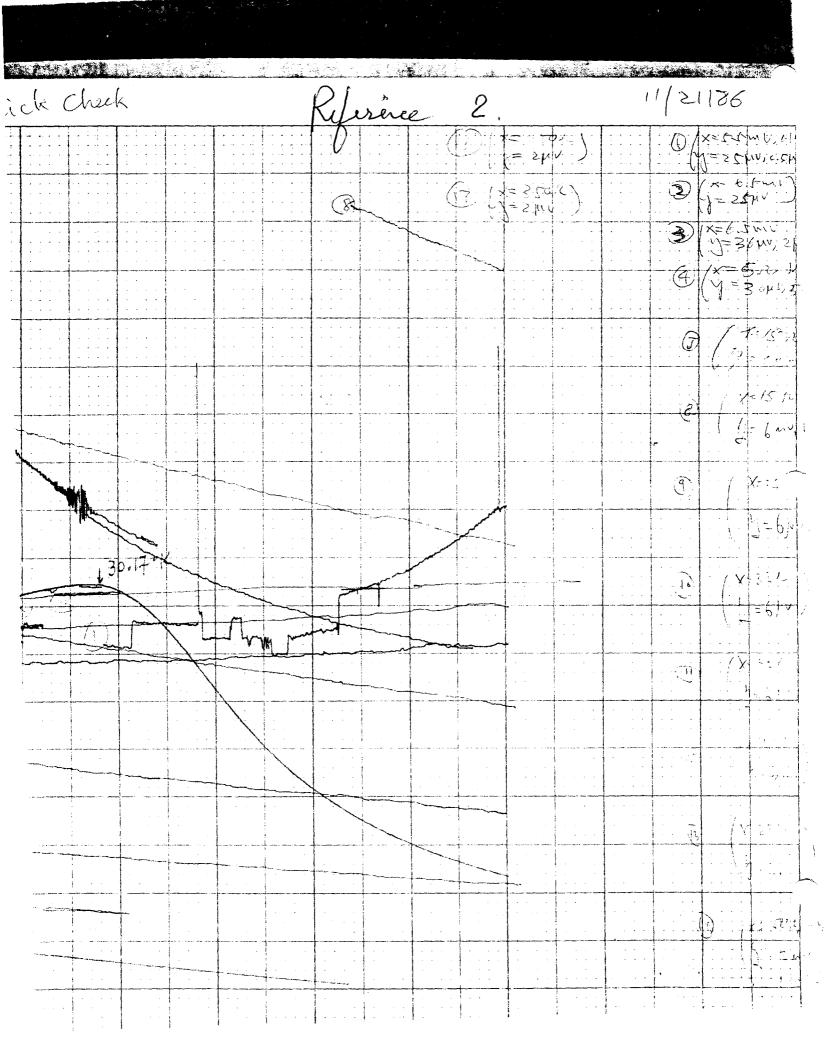
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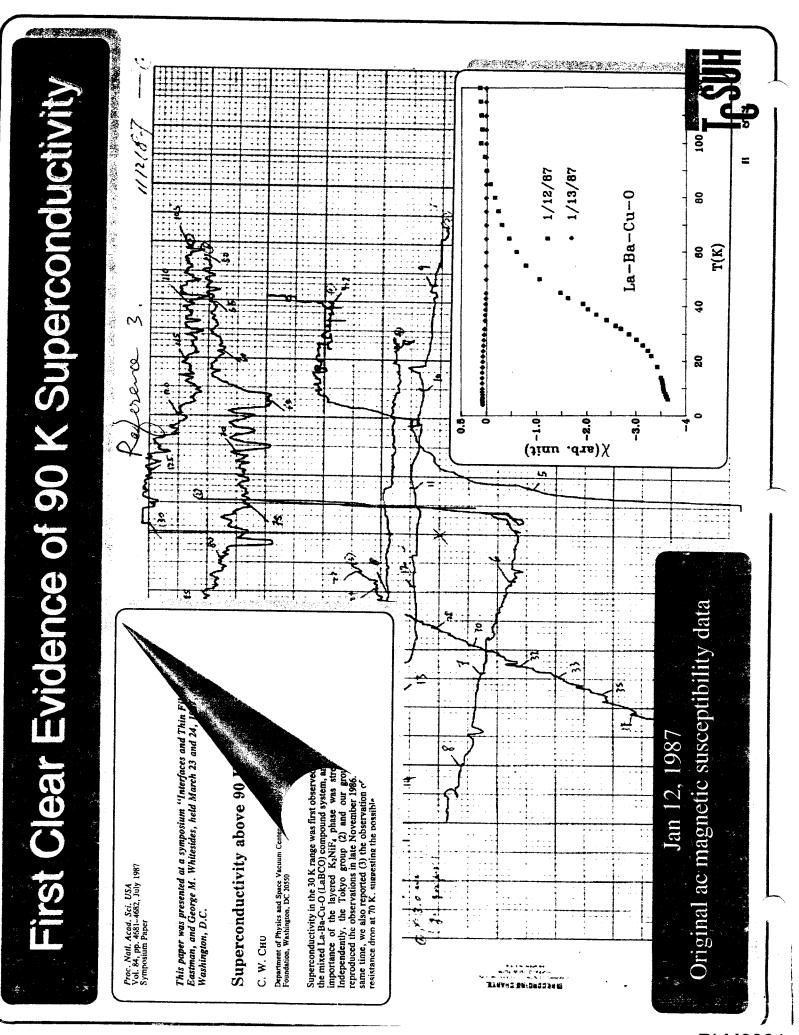
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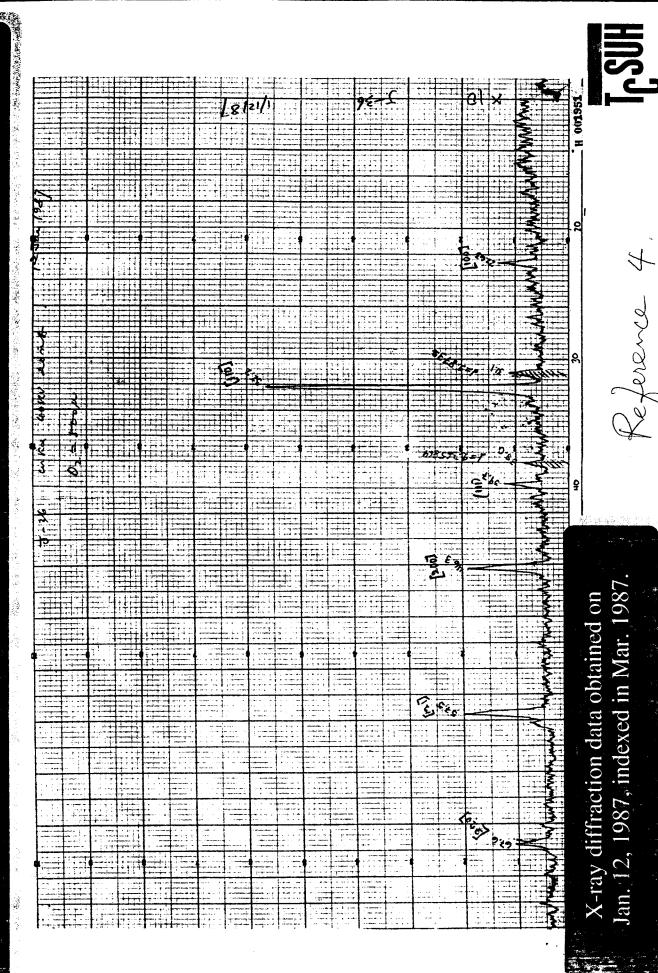
Note Added in Proof

Chemical analysis of the bulk composition of our samples revealed a deviation from the ideal La/Ba ratios of 4 and 5.66. The actual ratios are 16 and 18, respectively. This is in agreement with an identification of the third phase as CuO.





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Reference

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 (Ba \times cm_{1-x}), O_{2}
 $X = 0.0$, 0.2 , 0.4 , 0.5 , 0.6
 0.8 , 1.0 .

Black:

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