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# High pressure synthesis of the infinite-layer compounds $Sr_{0.9}La_{0.1}CuO_2$

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**Abstract**

We report high pressure synthesis of electron doped infinite layer superconductor $Sr_{0.9}La_{0.1}CuO_2$. Structural and low-field magnetization study demonstrated that the superconducting quality of our samples were best among all kind of bulk samples reported until now. With these good samples, many new findings are under going by our collaborators and us.

*Keywords* : Electron doped, infinite layer, superconductor

## I. Introduction

The infinite layer compound $Sr_{1-x}Ln_xCuO_2$ (Ln=La, Gd, Nd, Pr, Sm etc) consists of infinite stacking of $CuO_2$ planes separated only by alkaline earth ions at A site.[1] This is the simplest structure which contains only key ingredients of all cuprate high temperature superconductor, i.e., dopable $CuO_2$ plane. Superconductivity with infinite layer structure was first observed in Nd doped sample by Smith.[2] The simplicity of the structure seems to provide a unique opportunity of exploring superconducting states of the high-$T_c$ cuprates. But the precise analysis of superconducting properties of this compound has been hindered mainly by insufficient quality of bulk sample. This is the reason of very weak understanding of this compound even with more than 10 year's experience after its birth.

High pressure synthesis method was found to be preferable for this system due to its ability of doping other various lanthanide ion into A site with larger superconducting volume fraction.[3]-[9] But temperatrue control under high pressure environment is not easy and the amount of one batch with uniform quality is somewhat smaller for several kind of analysis. Thus synthesis of high quality samples with larger quantity in one batch is extermely important for this compound.

Generally there are two methods to increase $T_c$ in high-$T_c$ cuprates. One is to dope optimally and the other is to enhance interlayer coupling between $CuO_2$ planes. Infinite layer compounds do not have charge reservoir block so we might guess the interlayer coupling is very strong in this system due to short distance between $CuO_2$ planes. But $T_c$ is only about 43 K for electron doped infinite layer compound.[3]-[9][There are still much debate on the superconductivity of hole doped system. [3], [4]] Moreover $T_c$ does not depend on the species and amount of Ln element.[5]

In this paper, we report the high pressure synthesis of high quality infinite layer superconductor $Sr_{0.9}La_{0.1}CuO_2$(La112). We checked the quality of sample in terms of structural purity and superconducting volume fraction and sharpness of superconducting transition. Rietveld structural analysis on powder x-ray diffraction XRD data using Cu Kα radiation and low field magnetization were studied for this purpose.

## II. Experimentals

High-pressure synthesis was performed with a

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cubic multi-anvil-type press.[5] The starting material of $La_2O_3$, $SrCO_3$ and CuO were mixed to the nominal composition of $Sr_{0.9}La_{0.1}CuO_2$ and was calcined at 950 °C for 36 hours with several intermittent grindings. We made pellet of precursor to dense packing inside of Au capsule. This was put into Au capsule inside high-pressure cell with Ti oxygen getter. Then slightly compressed using a small press to remove captured air between the precursor and inner wall of Au capsule. The pressure cell was compressed to 4 GPa for 10 hours and then heat-treated with a graphite-sleeve heater. First ramping was done for 1-2 hours and second(main) heating at 1000-1100 °C was maintained for 2 hours and finally it was postannealed at 500-600 °C for 4 hours. We found that there are two important conditions to get sample with nearly uniform and high quality. One thing is long-term stability in synthesizing temperature and the other is the uniformity of temperature inside sample cell. For this purpose we optimized all the dimension of the part of pressure cell. The weight of the obtained samples in one batch was as large as 230 mg, which is about 3 times larger than others.[6] Monitoring the heating power just across heating element rather than primary temperature controller was found to be critical for high temperature stability, which is not adopted by other groups.

Until now we made about 150 samples for this compound. Everyday we made a sample and at night we measured magnetization and XRD and tried new synthesis condition at the other day. Recently the condition was nearly fixed and now about 50 % percent samples were found to have best quality.

Low field magnetization was obtained using a SQUID magnetometer(Quantum Design, MPMS*XL*}) for both zero-field-cooled(ZFC) and field-cooled(FC) mode at typically 10 Oe. Remnant stray field was less than about 1 Oe, which was corrected for analysis.

## III. Results

Our successful synthesis of high quality sample could be most clearly seen at Fig. 1. We measured low-field magnetization curve at zero-field-cooled mode and calculated nominal superconducting volume fraction $f_{nom}$ without considering demagnetization factors[10] Important thing is not that superconducting volume fraction is just larger than those of others but that we obtained samples with homogeneous superconducting properties and the obtained sample is really phase pure. Phase purity in terms of structural data such as XRD or neutron powder diffraction does not mean real purity in superconducting phase. Many of the reports have shown that their sample is phase pure in structure but superconducting volume fraction is so widely distributed especially in this infinite layer compound.[5]-[9]

The zero-field-cooled low-field susceptibility $4\pi\chi$ ($T$) was measured for calculation of $f_{nom}$, as shown in the inset of Fig. 1. The superconducting transition onset appears at 43 K, which is typical value for La-doped infinite-layer superconductors.[5]-[9] However, our data shows some notable features as contrasted with the previous reports. The curves shows considerably sharp transition below $T$ = 43 K and well-developed saturated behavior at low temperatures, which reflects the formation of homogeneous La112 phase in our sample. Roughly our samples have about two times higher volume fraction and two times sharper transition width.[6]

For superconducting sphere with radius $R$, the $4\pi\chi$ is given by the Shoenberg formula $-3/2(1-(3/x)\coth x + 3/x^2$, where $x = R/\lambda_{avg}(T)$ and $\lambda_{avg}(T)$ is the average magnetic penetration depth, i.e., $\lambda_{avg}(T) = \lambda_{ab}^2\lambda_c)^{1/3}$.[11] In the limit of $x >> 1$, the absolute value of $4\pi\chi$ can be about 1.5, which is 50 % lager than ideal value due to the demagnetization effect.[10] If we employ the typical value of $\lambda \sim 2000$ • for high-Tc cuprates and the grain size R ~ 5 μm obtained from SEM picture on our sample, the value of $4\pi\chi$ is estimated to be about -1.3 from the Shoenberg formula, which is close to the above measured value. Thus we can infer that the superconducting volume fraction of our sample is nearly 100 %, really phase pure.

The Rietveld structural refinement on the powder XRD pattern showed that the structure corresponds to that of a infinite layer compound, with tetragonal space group *P4/mmm* and a lattice parameter $a = b =$ 3.950 and $c$ = 3.410 • . These obtained values of lattice constant agree quite well with those obtained by neutron powder diffraction within one thousandth • .[6] Structural analysis on our sample also could

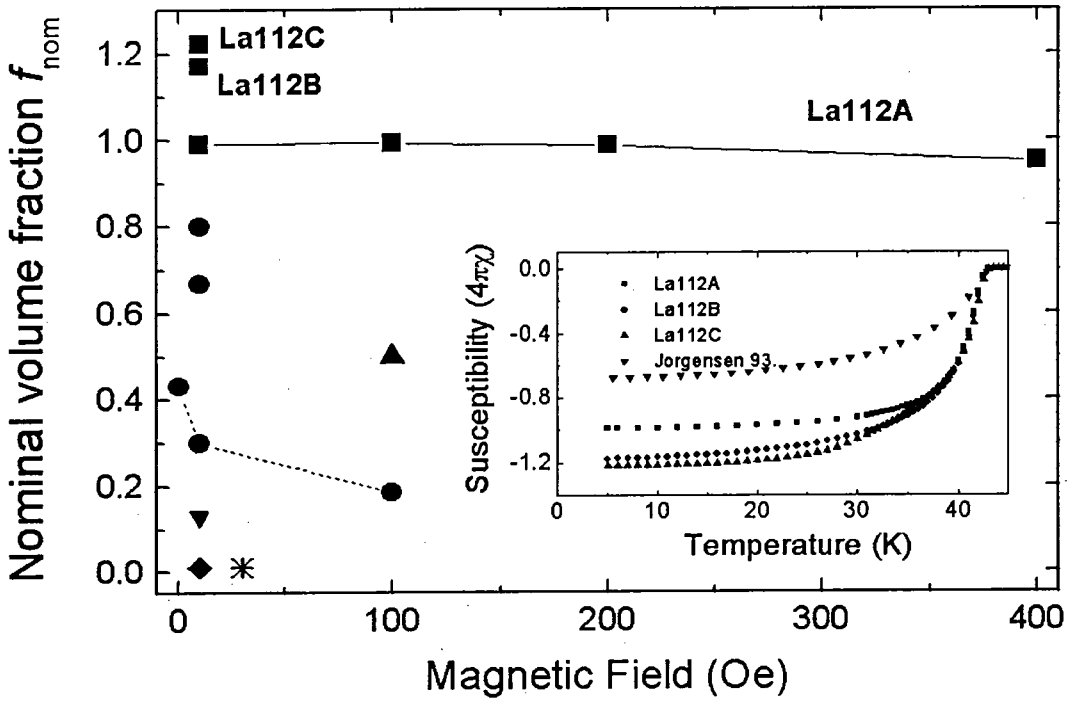


Fig. 1. Nominal superconducting volume fraction of infinite layer superconductor. Rectangular symbol for our samples, Other symbols from other groups. Inset shows low field susceptibility data of our samples. One of the best samples reported by other groups is included for comparison.[6]

confirm the stoichiometry because tetragonal lattice constant has been known to be sensitive to doping concentration in this system.[5] So our sample was found to be phase pure in structure and stoichiometry also.

## IV. Summary

We made high quality samples of electron doped infinite superconductor $Sr_{0.9}La_{0.1}CuO_2$. Structural and low-field magnetization study demonstrated that the superconducting quality of our samples were best among all kind of bulk samples reported until now.

Various unrevealed mysteries on this compound are under study. These includes the pinning properties, anisotropy, and symmetry of superconducting gap.[12]-[14]


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