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CHARACTERIZATION OF THE 105 K SUPERCONDUCTOR $\text{Tl}_2\text{Ba}_2\text{CaCu}_2\text{O}_8$ ("2212")

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Homogeneous $\text{Tl}_{1.8}\text{Ba}_2\text{CaCu}_2\text{O}_8$ ceramics were synthesized by a novel route, starting from $\text{Tl}_2\text{Ba}_2\text{O}_5$ precursors and using high gas pressures. This method allows tight control of thallium losses, resulting in dense, large-grained samples with sharp superconducting transitions above 105 K. Results of a characterization by X-ray diffraction, optical micrographs and micro-probe analysis are presented, together with selected physical properties.

1. INTRODUCTION

The thallium superconducting phases $(\text{TlO})_m\text{Ba}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2+2n}$ with $m=1$; $n=1, 2 \dots$ and $m=2$; $n=1, 2 \dots$ are particularly interesting materials for their high critical temperature and promising critical currents. However, the high thallium vapor pressure and its toxicity render this element difficult to use. Under high pressure (100 bar), in spite of the increase of the saturation vapor pressure, it is possible to effectively suppress the thallium loss. Consequently, we can synthesize the thallium compounds at high temperature in a short time without furnace contamination and health risk. The procedure results in dense and large-grain size samples. In this paper, we discuss the synthesis of $\text{Tl}_2\text{Ba}_2\text{CaCu}_2\text{O}_8$ (Tl-2212, $m=2$, $n=2$).

2. SAMPLE PREPARATION

Starting with $\text{Tl}_2\text{Ba}_2\text{O}_5$ precursors, we added BaO, CaO and CuO in order to obtain the $\text{Tl}_{1.8}\text{Ba}_2\text{CaCu}_2$ stoichiometry. The powders were then pressed into pellets under 4 kbar and fired at 850°C under 100 bar of a mixture of He-O₂ gas (99.5 bar of He and 0.5 bar of O₂) during 30 min. The reaction was completed by a second firing after regrinding. A final treatment was given at 900°C (100 bar), followed by rapid cooling at an initial rate of 500°C/min. A

typical microstructure is shown in Fig. 1. The mass loss during the procedure, attributed to all metallic elements and to a change in oxygen concentration, is about 2.4 %. In the following, we characterize a sample (T2-20) which was further annealed under low pressure, 1 mbar of O₂ at 500 °C for 3 days, in order to change the oxygen concentration.

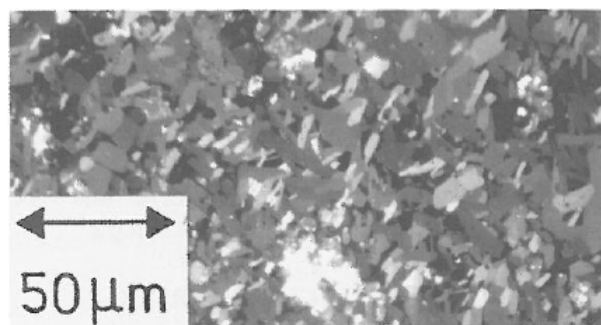


Figure 1: Optical micrograph of the T2-20 sample.

3. X-RAY DIFFRACTION AND MICROPROBE ANALYSIS

The crystal structure was studied at ambient temperature by X-ray diffraction using a Guinier camera with Cu K α radiation. The pure X-ray diffraction pattern of the T2-20 sample is presented in Fig. 2. Note that it contains the peaks of the silicon standard and two camera lines at $4\theta=88.84$ and 98.83° . The lattice parameters of the tetragonal ($I4/mmm$ [1]) Tl-2212 phase obtained by least squares fits are

$a=3.8548(5)$ and $c=29.328(4)$ Å.

The samples are X-ray pure, only optical micrographs and microprobe analysis shows the presence of a small amount of unreacted starting material.

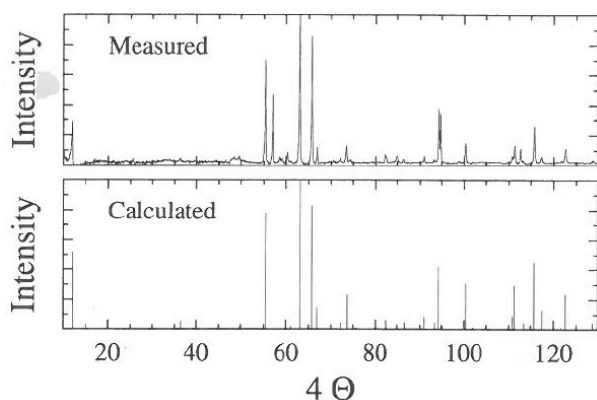


Figure 2: Measured and calculated X-ray diffraction patterns of the T2-20 sample.

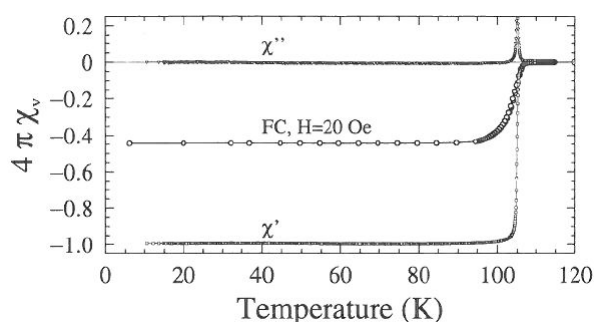


Figure 3: Field cooling measurement and AC susceptibility (arb. units) of the T2-13 sample.

4. SUPERCONDUCTING PROPERTIES

The Meissner field cooling susceptibility was measured using a r.f. SQUID magnetometer with a calibrated external magnetic field of 20 Oe. A geometric demagnetization factor, depending on the sample shape, was taken into account. The resistivity was measured with the four-point technique. The current density was 0.8 mA/cm² and the frequency was set at 75.5 Hz.

Fig. 3 shows the field cooling measurement and the AC susceptibility ($H_{rms}=0.10e$) of sample T2-13 (as synthesized, without low pressure annealing). The sharpness of the transition characterized by a peak of χ'' about 0.5 K wide coincides with the superconducting transition and suggests good oxygen homogeneity and well coupled Tl-2212 grains.

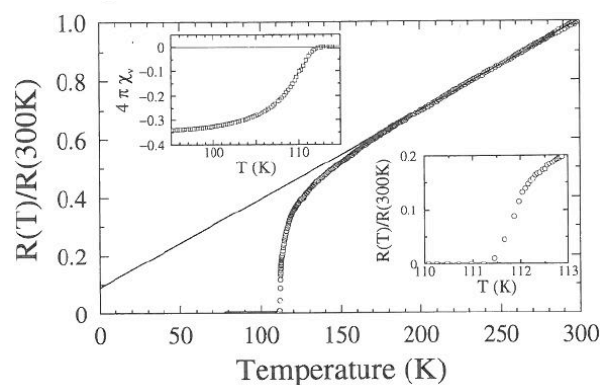


Figure 4: Normalised resistance of the T2-20 sample.

Fig. 4 shows the behavior of the resistivity as a function of temperature for the T2-20 sample. Zero resistivity is reached at 111.3K. The ratio $R(300K)/R(200K)=1.44$ is in good agreement with Raveau et al. [2]. A fit of the resistivity indicates superconducting fluctuations extending up to 60 K above T_c .

5. CONCLUSIONS

The present work has concentrated on the use of high pressure during the synthesis of Tl-2212. The results extend analogous conclusions in the case of Tl-2201 (Tl₂Ba₂Cu₂O₆) [3,4]. Pressures in the 100 bar range allow to choose the highest possible reaction temperatures leading to superior superconducting properties.

6. ACKNOWLEDGEMENTS

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