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Magnetic response and critical current properties of mesoscopic-size YBCO superconducting samples

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Abstract In this contribution superconducting specimens of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ were synthesized by a modified polymeric precursor method, yielding a ceramic powder with particles of mesoscopic-size. Samples of this powder were then pressed into pellets and sintered under different conditions. The critical current density was analyzed by isothermal AC-susceptibility measurements as a function of the excitation field, as well as with isothermal DC-magnetization runs at different values of the applied field. Relevant features of the magnetic response could be associated to the microstructure of the specimens and, in particular, to the superconducting intra- and intergranular critical current properties.

1. Introduction

Superconducting compounds constitute one of the most studied groups of strongly correlated electronic materials whose structural, electronic and magnetic properties are highly dependent on the synthesis routes and occasional subsequent processing.

Furthermore, improvements in critical current density (J_c) in the presence of high applied magnetic fields are crucial to high temperature superconducting (HTS) applications. A standard routine to improve J_c is associated to the controlled introduction of pinning centers and the enhancement of their capacity to trap magnetic flux. Due to the small coherence length associated with HTS materials, microstructural and stoichiometric defects can be used as good pinning centers.

The critical current of a superconducting polycrystalline sample is commonly analyzed as a composition of two independent contributions: one related to the response of the grain itself (intragranular material) and the other related to the grain boundaries, controlled by weak-links, WLs (intergranular). Thus, magnetization isothermals used to determine the critical current density are directly associated with the sum of the two contributions (total critical current density $J_c^{\text{tot}} = J_c^{\text{inter}} + J_c^{\text{intra}}$). Moreover, there is a temperature condition, the critical temperature of the WLs (T_c^{wl}), below which the WLs magnetic response is strongly dependent with the applied magnetic field. Above this temperature, the intergranular critical current density vanishes [1,2]. T_c^{wl} is obtained from two or more AC-susceptibility curves as a function of temperature, $\chi'(T)$, measured using different excitation field amplitudes, which are different at low temperatures but collapse at T_c^{wl} and above.

In this contribution, $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ ceramic samples (YBCO) were prepared using a modified polymeric precursor method [3]. This method allows obtaining samples which present very homogeneous and small-size particles, weakly aggregated, which usually present a narrow intergranular critical current distribution. Superconducting intragranular and intergranular critical current properties were analyzed

in samples in powder and pressed-pellet form. Using the imaginary part of the AC-susceptibility, measured as a function of the excitation field, $\chi''(h)$, we have determined the temperature dependence of J_c^{inter} [4] for two different cylindrical specimens; one pelletized using 2,830 kgf/cm² and the other with 5,660 kgf/cm². Both samples were sintered in the same conditions. Our data show that the increase on the pressure used to pelletize the samples causes a decrease on J_c^{inter} . This result was developed as a continuation of a previous study which describes the superconducting behavior on samples that were simply pelletized, without sintering or any further treatment [5].

2. Experimental Details

The samples studied in this work were prepared by a modified polymeric precursor method, details of which were described elsewhere [3]. In this method, citric acid (CA) was used as a chelating agent, in the ratio of 3/1 for metallic mol (Y, Ba and Cu). Ethyleneglycol (EG) was the polyesterification agent, in the ratio of 40/60 to CA mass. Ethylenediamine was added until the pH was stabilized at 7. The resulting blue solution was turned into a gel by heating, $T \sim 70^\circ\text{C}$. Following, successive calcinations steps were performed in 200, 400, 600, 890 and 850 °C respectively, resulting in a homogeneous black powder. To improve O₂ stoichiometry, samples still in powder form, were annealed for 48 h at 550 °C in O₂ flux. After that, two cylindrical pellets were prepared; one with a pressure of 2,830 kgf/cm² and the other using 5,660 kgf/cm²; both were then sintered at 950 °C during 4h also in O₂ flux.

Microstructural conformances of the obtained samples were studied by scanning electron microscopy (SEM) measurements. Magnetization and AC-susceptibility measurements were performed using a Quantum Design SQUID magnetometer.

3. Results and Discussion

Effects of different pressures and sintering processing on the microstructure of the specimens are presented on Fig. 1a and 1b. It can be seen that the sample prepared with 2,830 kgf/cm² presents a considerable porosity and also well-defined necks between the grains. On the other hand, the sample prepared with 5,660 kgf/cm² has a much denser microstructural conformance.

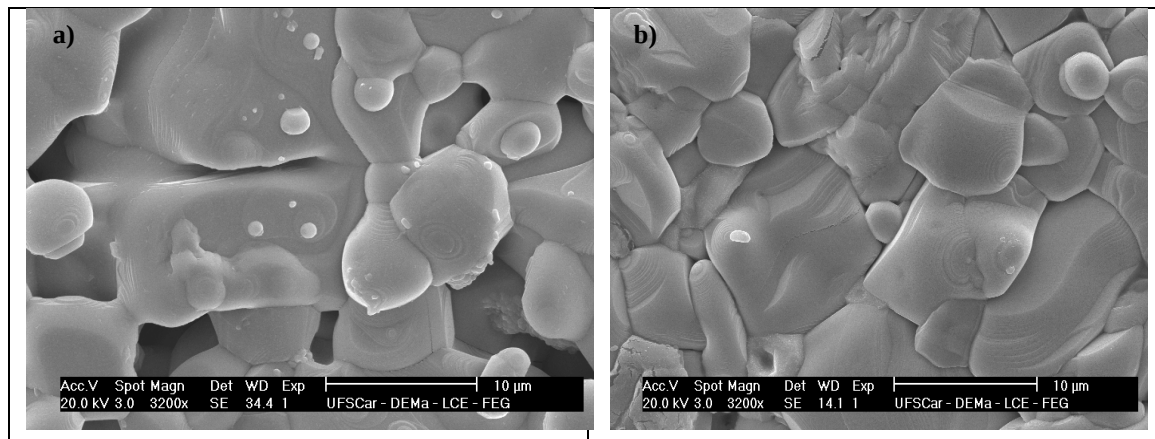


Figure 1: Microstructure of the YBCO samples: a) 2,830 kgf/cm² and b) 5,660 kgf/cm².

Magnetization vs. temperature measurements performed at low fields presented a small ZFC/FC magnetic irreversibility and similar diamagnetic responses, suggesting that the two pellets present same superconducting fraction. Also, the analysis of the AC magnetic susceptibility as a function of temperature measured at low fields, shows that the sample prepared using 5,660 kgf/cm² presents the largest diamagnetic response of the three studied specimens. It is also possible to observe that the magnetic response of the powder does not depend on the excitation field. This result is related with the strongly bounded particles which form the aggregates. Moreover, no important difference on the superconducting critical temperature for the three different samples was observed. It has been extensively discussed in the literature [6,7] that the dependence of $\chi'(T)$ with the excitation field below a certain temperature T_c^{wl} , can be related with the existence of weak links in the sample, and that the intergranular supercurrent density, J_c^{inter} , vanishes at T_c^{wl} and above. Evidently, T_c^{wl} is the critical temperature of the WLs. According to the obtained results shown in the inset of Fig. 2, the increase of the pressure used to perform the pellet reduces T_c^{wl} and, consequently, J_c^{inter} .

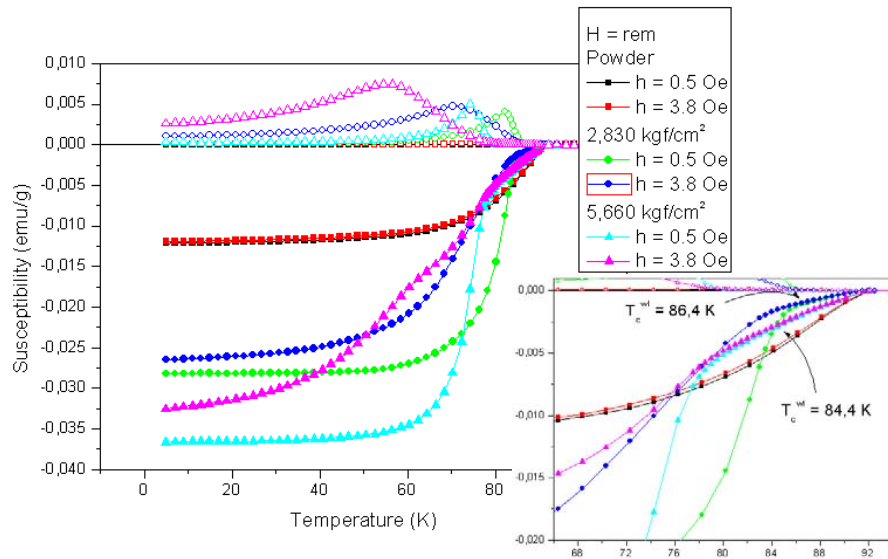


Figure 2: Ac-susceptibility as a function of temperature, measured in the remnant field of the superconducting magnet. Inset shows T_c^{wl} for the two pellets.

Using a critical-state model one can relate the excitation field corresponding to the peak on the imaginary part of the AC-susceptibility, h_p , with J_c^{inter} . For a cylindrical pellet with radius a , the relation between h_p and J_c^{inter} is $h_p = a < J_c^{inter} >$. Fig. 3 shows typical measurements of $\chi''(h)$, used to determine the intergranular critical current density of each of the studied pellets. Noticeably, the peak disappears for temperatures above T_c^{wl} .

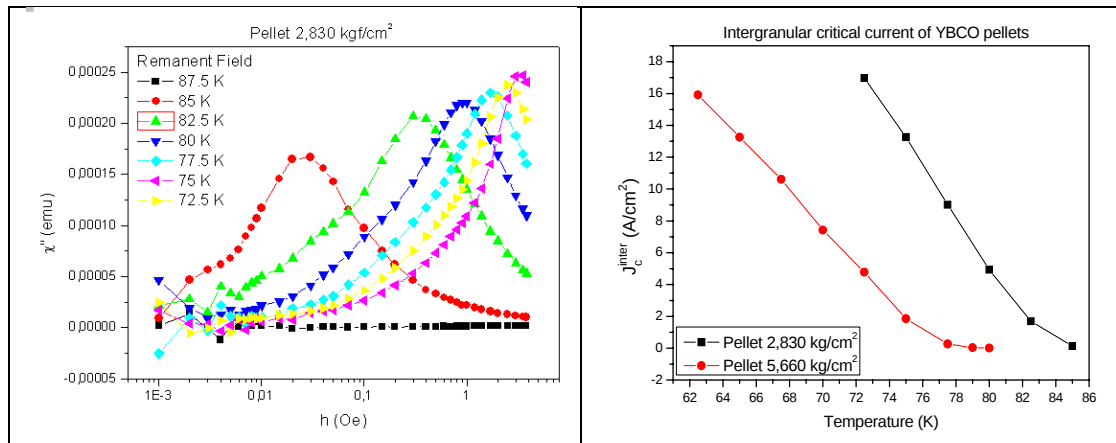


Figure 3: (a) Measurements of Ac- susceptibility as a function of the excitation field, in temperatures below T_c^{wl} . and (b) Intergranular critical current density as a function of the temperature.

The values of $h_p(T)$, obtained within the experimental range of 10^{-3} to 3.8 Oe, can be used to determine the behavior of the intergranular critical current density, for the two studied pellets, as shown in Fig. 3b. One can see that J_c^{inter} is larger for the sample prepared with 2,830 kgf/cm², a result that is consistent with the SEM results, which show a better neck formation between the grains, for the sample prepared with the smaller value of the pressure.

Measurements of the magnetization as a function of the applied magnetic field were used to derive the critical current density of the samples [8] and it was found that the 2,830 kgf/cm² pellet present a higher value of the critical current density in a specific range of fields, as shown in Fig. 4. This behavior is related to the weak links present in the two specimens.

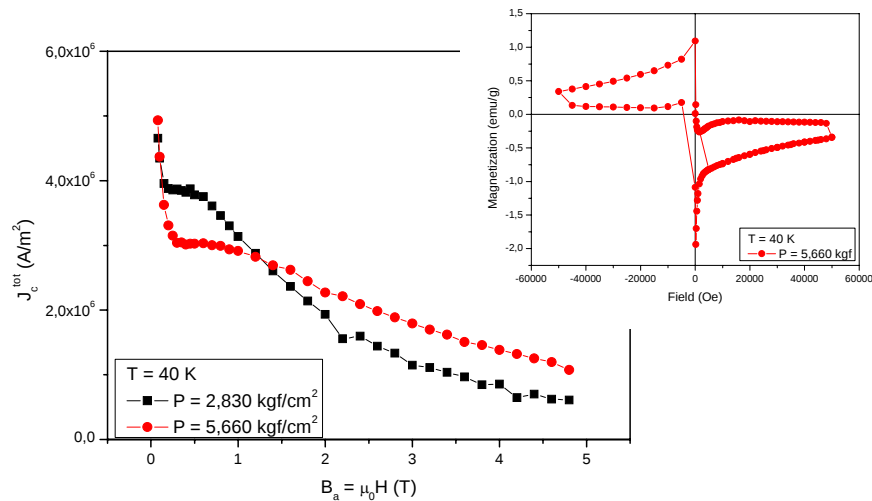


Figure 4: J_c^{tot} as a function of the applied field. Inset shows an isothermal of magnetization used to derive J_c^{tot} .

The crossover between J_c^{tot} curves for the two samples is associated with the volumetric density presented by the 5,660 kgf/cm² pellet, which is higher than the other pellet. It is also expected decrease of J_c^{inter} contributions in high magnetic field conditions.

4. Concluding Remarks

In this work we have studied the correlation between different microstructure conditions and the superconducting critical current density. To improve the quality of the ceramic YBCO samples, chemical synthesis was used to prepare the precursor powder. Low field $\chi'(T)$ measurements indicate that T_c^{wl} is smaller for the pellet pressed with 5,660 kgf/cm². Further analysis of $\chi''(h)$ measurements show that the sample prepared with 2,830 kgf/cm² presents a higher intergranular critical current density, which is related with the quality of the WLs. These results are consistent with SEM studies, which show significant differences on the necks between grains, through which intergranular superconductivity takes place. On the other hand, magnetization loops at high magnetic fields indicate a larger total critical current density for the 5,660 kgf/cm² sample, which is related to its larger volumetric density in comparison with the other specimen.

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