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# Microstructure and Critical Current of Hot-Pressed YBCO Ceramics

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**Abstract**

The effect of hot-pressing (HPing) on the densification and the superconducting properties of YBCO were investigated. The phases and microstructure were analysed by XRD and SEM and transport properties were studied by I-V and AC susceptibility measurements. Relative density up to 93% and critical current density of 509 A/cm$^2$ at 77 K were achieved through uniaxial HPing at 800°C under 325 kg/cm$^2$ pressure for 4 hours. HPing raised the $J_c$ from 80 A/cm$^2$ to 509 A/cm$^2$. These results and AC susceptibility measurements reveal that the weak links or pinning forces were improved by increasing HPing pressure.

**Key Words:** A. High temperature superconductors; B. Hot pressing; C. YBCO; D. I-V; E. AC susceptibility.

## 1. Introduction

Oxide superconductors are highly attractive for technical applications since they can became superconductor at liquid nitrogen temperature [1-5]. For most technical application the important properties is the critical current density ($J_c$) and this depends on temperature, magnetic field and the microstructure. Common among all high temperature superconducting systems is the observation that the transport $J_c$ are rather low, the two main reasons for this being that the plate-like grains growing in random directions cause low density and highly porous structure. Usually, increased density and proper grain alignment is achieved by the techniques, which involves deformation under high pressure at elevated temperatures. Hot forming techniques; such as sinter-forging [6], hot-rolling [7] and hot pressing [8-10] are known to generate "textured" micro-structures in ceramics. The goal of the present work was to produce highly dense YBCO bulk samples by the hot pressing technique in order to improve critical current density.

## 2. Experimental Details

The experimental hot pressing set-up is shown in Figure 1. The experimental cell is made of two alumina plungers with diameter of 20 mm guided by a stainless steel holder. This system is located in a vertical homemade furnace. The pellets are pressed between two platinum foils. The temperature is controlled and monitored by two Chromel-Alumel thermocouples. Samples were prepared by mixing and grinding 99.99% pure powder of $Y_2O_3$, $BaCO_3$ and CuO and reacting them in a alumina crucible in air at 850°C for 24 hours. The reacted material was ground and pressed into pellets at room temperature. Then the pellets were heat treated in flowing $O_2$ (3 ml/minute) at 950°C for 48 hours and cooled to room temperature. Following this, two pellets were hot-pressed. One pellet (sample B) was uniaxially HPed in air at 800°C under 162 kg/cm$^2$ pressure for 4 hours and the other pellet (sample C) was HPed at the same temperature and same period of time under 325 kg/cm$^2$ pressure. The HPed samples were finally annealed under the same conditions as the sintering process.

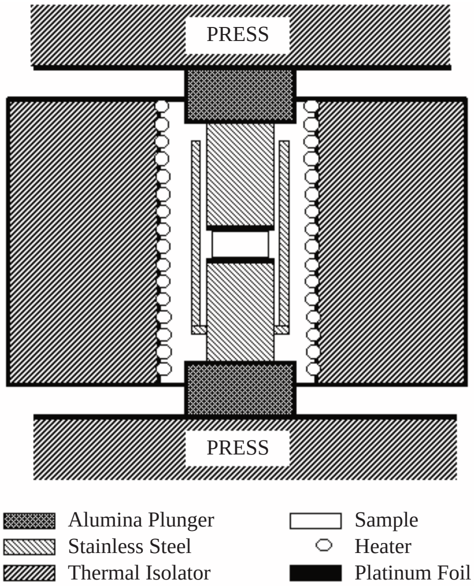



**Figure 1.** Schematic diagram of the hot-pressing equipment.

The bulk density of the samples was measured by the geometric method. Microstructural analysis was carried out on the samples using X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. The superconducting properties were investigated by transport critical current density and AC susceptibility measurements. The transport critical current density was determined from I-V measurements carried out at 77 K and in zero applied magnetic field with voltage criterion of 1 $\mu$V/cm. In order to eliminate size effect, tetragonal samples having dimensions of 0.3 x 0.4 x 6 mm$^3$ were used in the determination of $J_c$. The AC susceptibility was measured in a uniaxial pair of primary and secondary coils. A lock-in arrangement was used for the measurements. The primary coil allowed application of AC fields with the various amplitudes between 5.6 mOe-1867 mOe at 16 Hz constant frequency in the temperature range 77 K-300 K. The dimensions of the samples used for AC susceptibility were 3 x 1.5 x 1 mm$^3$.

# 3. Experimental Results and Discussion

X-ray powder diffraction patterns results for non HPed (A) and HPed (C) samples are shown in Figure 2. Analysis of the patterns indicated that all the samples are in the 92 K single superconducting phase of YBCO and no impurity phases were detected. The HPing effect on the $Y_1Ba_2Cu_3O_{7-x}$ samples were observed on their densities. The measured densities were 5.09 g/cm$^3$ for non HPed, 5.72 g/cm$^3$ for sample B and 5.92 g/cm$^3$ for sample C. These values are 80%, 89% and 93% of the theoretical density (6.365 g/cm$^3$ ), respectively. The change in the density of the HPed samples is a result of the increased pore elimination that caused to a remarkable grain growth. In order to correlate the effect of the HP on the microstructure SEM investigation has been carried out. Figure 3. shows SEM photographs of the non HPed and HPed samples. SEM photographs results reveal that with increasing HPing pressure, the average grain size increases, and samples become less porous. Generally, pressing at room temperature is not effective in reducing porosity of the high temperature superconductors (HTSC) because of the high deformation resistance of the pellet. Increasing the pressure and temperature, reduces the deformation resistance which allows the grain growth. The transport critical current densities at 77 K are given in Table 1. The values of $J_c$ were 90, 218 and 509 A/cm$^2$ at 77 K for sample A, B and C, respectively. This result strongly indicates that the weak links at grain boundaries and/or the flux pinning forces within the grains were improved with increasing HPing pressure. AC susceptibility of the HTSC is widely measured and analysed to understand microstructure of HTSC and the nature of the coupling between the grains [11-13]. Figure 4. presents temperature variation of the AC susceptibility of the samples at the 186.7 mOe AC magnetic field amplitude. The diamagnetic onset temperature $T_{con}$ of the superconducting transition is about 92 K and superconducting transition temperature temperature $T_c$ are 90.4 K, 89.6 K and 91.5 K for samples A, B and C, respectively. For all the samples, two distinct superconducting transitions are observed in the real part ($\chi$') of the AC susceptibilities. When the temperature of the samples are just below $T_c$, the superconducting grains first shields the magnetic field. This is observed as a negative $\chi$'. If the temperature is reduced enough inter-granular component of $\chi$' is observed. At extremely low temperatures $\chi$'-T curve saturates. In the imaginary part ($\chi$") of the AC susceptibility appeared with only one peak at the 186.7 mOe AC magnetic field amplitude which corresponds to inter-granular component of $\chi$'. This peak observed in the imaginary part represent the flux penetration into boundaries between the grains. Moreover, the maxima of the peaks at the imaginary part associated with inter-grain diamagnetic transition temp ($T_{cj}$), shifted to higher temperatures and getting sharper as the HPing pressure increased. The values of $T_{cj}$ were 87.6 K, 88.6 K and 89.6 K for samples A, B and C, respectively. The amount of the shift is directly proportional to the magnitude or strength of the pinning force. So we concluded that the HPed samples have a much better inter-grain coupling than a non HPed sample. This results shows that the weak links were improved by hot pressing process.

**Table 1.** HPing conditions, and some characteristic values of the samples.

| Sample code | HPing temperature (°C) | HPing pressure ($kg/cm^2$) | HPing time (h) | Density ($g/cm^3$) | Relative density (%) | $J_c$ ($A/cm^2$) | $T_{CON}$ (K) | $T_C$ (K) | $T_{CJ}$ (K) |
|---|---|---|---|---|---|---|---|---|---|
| A | - | - | - | 5.09 | 80 | 90 | 92.6 | 90.4 | 87.6 |
| B | 800 | 162.5 | 4 | 5.72 | 89 | 218 | 92.3 | 89.6 | 88.6 |
| C | 800 | 325.0 | 4 | 5.92 | 93 | 509 | 92.2 | 91.5 | 89.6 |

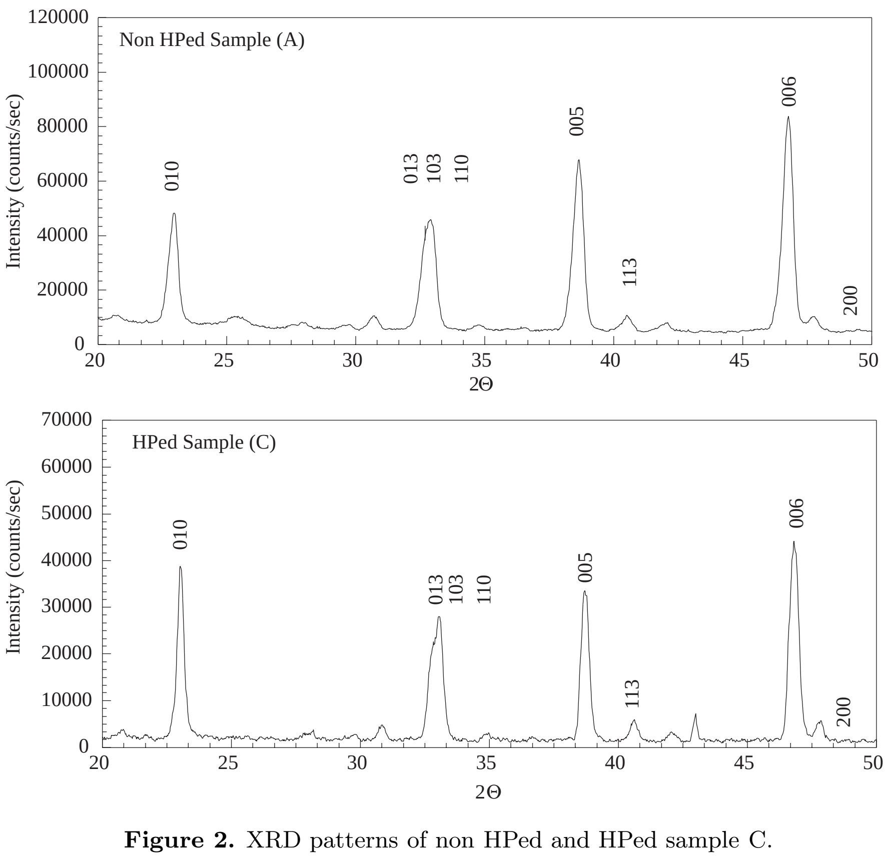
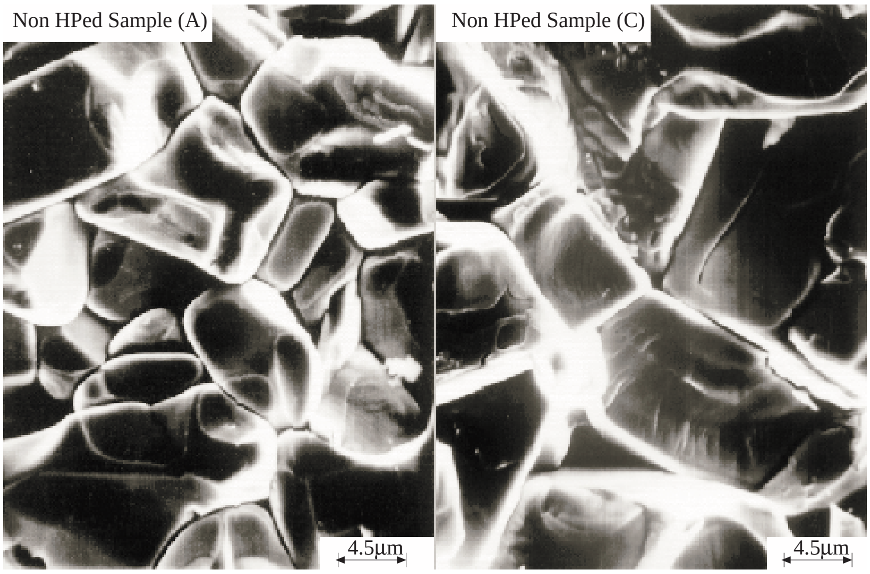



**Figure 2.** XRD patterns of non HPed and HPed sample C.




(a) (b)

**Figure 3.** SEM images for YBCO (a) non HPed and (b) HPed sample C.

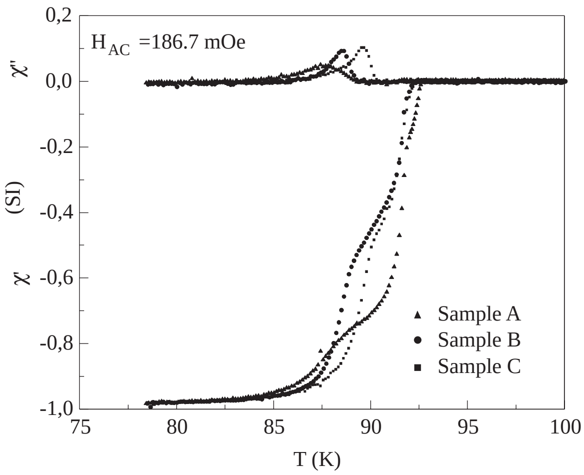



**Figure 4.** AC susceptibility versus temperature varion of the samples A,B and C at 186.7 mOe magnetic field.

In summary, the bulk density of a normal sintered sample of YBCO is 5.092 g/cm$^3$ and shrinkage of sample was barely observable. By HPing at pressure of 325 kg/cm$^2$ at 800°C for 4 hours, the bulk density of the HPed sample reached to 5.92 g/cm$^3$, which was over 93% of the theoretical density and critical current density at 77 K was increased to 509 A/cm$^2$. It is expected that $J_c$ will further increase by optimizing the HPing parameters.

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