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# OPTIMIZATION IN THE PREPARATION OF THE $YBa_2Cu_3O_{7-\delta}$ COMPOUND USING $BaCO_3$ AND $BaO_2$ AS REAGENTS

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**Abstract**

The traditional superconductor $YBa_2Cu_3O_{7-\delta}$ (Y123) has an orthorhombic crystalline structure with spatial group Pmmm and its critical temperature is 90 K. The literature reports the use of reagent $BaCO_3$ for the preparation by the solid state reaction which requires one calcination and two thermal treatments in oxygen flow at 950 °C. In this work we report the comparison in the preparation of Y123 using the $BaO_2$ reagent as another possibility for preparation; in this case we have used only two thermal treatments in oxygen flow at 950 °C; therefore reducing the costs in preparation. The powder XRD pattern of samples prepared with $BaO_2$ confirm a well crystallized phase an the diffraction lines of the main phase can be indexed with an orthorhombic unit cell. The ac susceptibility measurement in the range 5-100 K using a Quantum Design (SQUID) magnetometer for two samples confirms the superconductor behavior with critical temperature $Tc \approx 91$ K. The critical temperatures for these systems were found taken account the derivatives of the susceptibility curves. Scanning Electron Microscopy (SEM) confirms the good behavior of the grains corresponding to both methods.

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***Keywords:*** Superconductivity, Y123, RDX, SEM

**Resumen**

El superconductor tradicional $YBa_2Cu_3O_{7-\delta}$ (Y123) tiene una estructura cristalina ortorrómbica con grupo especial Pmmm, su temperatura crítica es 90 K. La literatura reporta el uso del reactante $BaCO_3$ para su preparación mediante el método de reacción del estado sólido, el cual requiere una calcinación y dos tratamientos térmicos en flujo de oxígeno a 950 °C. En este trabajo nosotros reportamos la comparación en la preparación del Y123 usando el reactante $BaO_2$ como otra posibilidad de preparación, en este caso nosotros requerimos solamente dos tratamientos térmicos en flujo de oxígeno a 950 °C; por lo tanto se reduce el costo en preparación. Los patrones XRD en polvo de la muestra preparada con $BaO_2$ confirman una Buena fase cristalizada y las líneas de difracción pueden ser indexadas con una celda unitaria ortorrómbica. Las medidas de susceptibilidad ac en el rango 5-100 K usando un magnetómetro Quantum Design (SQUID) para las dos muestras confirman el comportamiento superconductor con temperatura crítica $T_c \approx 90$ K. La temperaturas críticas para estos sistemas fueron calculadas teniendo en cuenta las derivadas de las curvas de susceptibilidad. La microscopia Electrónica de Barrido (SEM) confirma el buen comportamiento granular correspondiente a los dos métodos.

***Palabras claves:*** Superconductividad, Y123, RDX, SEM

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## 1. Introduction

Power applications of high temperature superconducting materials is a very promising field growing day and day, specially in the field of large currents applications such as: motors, generators, transformers, current leads, fault current limiters, transmission cables, energy storage (flywheel, SMES) magnetic bearings etc. Most of these various devices are based on $YBa_2Cu_3O_{7-\delta}$, $Bi_2Sr_2CaCu_2O_x$ and $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ superconductors. A number of techniques for processing $YBa_2Cu_3O_{7-\delta}$ powders have been reported, including conventional solid-state reaction, precipitation, spry drying, combustion synthesis, sol gel method, acetate methods and flame synthesis[1-4]. In this work we report the comparison in the preparation of Y:123 using $BaCO_3$ and $BaO_2$ as reagents therefore reducing costs.

## 2. Experimental

Two samples of $YBa_2Cu_3O_{7-\delta}$, were prepared by a solid state reaction of stoichiometric mixtures of $Y_2O_3$ (99,99 %), CuO (99,99 %), $BaCO_3$ (99,99 %) and $BaO_2$ (99,99 %). Reagents were mixed and ground under methanol in agate mortars. The calcinations and synthesis were carried out in $Al_2O_3$ crucibles; when we use the $BaCO_3$ reagent (sample A) one calcination at a 950 °C for 24 hours and two thermal treatments in flowing oxygen atmosphere at a 950 °C for 24 hours were required. In the second case (sample B), was used $BaO_2$ and only two synthesis at 950 °C for 24 hours were required. In the final step of synthesis both the samples were cooled at a rate of 50 °C/h down to room temperature. Pellets of 1000 mg weight and 1 cm diameter were obtained by pressing the powders. The phase analysis and structural parameters were determined by X-ray diffraction, using Cu Kα radiation in steps of 0,02°. AC susceptibility measurements were made on a commercial (Quantum Design) SQUID magnetometer at a low field ($H_{ac}$ =1 Oe) to ensure the superconducting transition temperature $T_c$ of all samples.

An analytical Scanning Electron Microscopy (SEM) has been taken in a Phillips XL30 microscope equipped with energy dispersive X-ray spectroscopy (EDX) processor.

## 3. Results and discussions

Fig. 1 shows XRD patterns for both samples, this reveals the presence of the orthorhombic (Pmmm) $YBa_2Cu_3O_{7-\delta}$, as main phase. However in the case of sample A, CuO (represented in bold circle) appears as secondary phase, this is not appreciable in sample B, therefore we can optimize the purity in the preparation using $BaO_2$ reagent. The cell parameters were refined using Rietveld method and the best fit for both samples were: a=3,8149 Å, b=3,8821 Å and c=11,6652 Å.

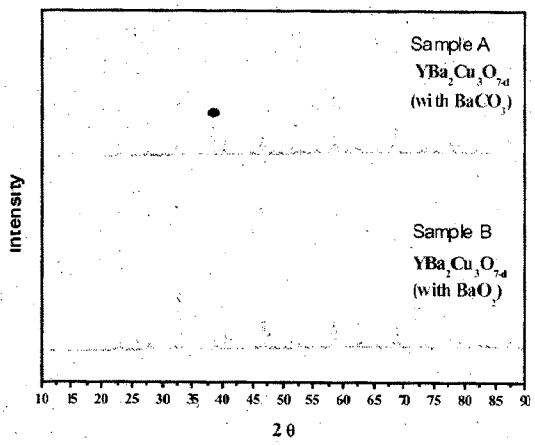


Fig 1. XRD patterns for $YBa_2Cu_3O_{7-\delta}$, sample A (using $BaCO_3$) and sample B (using $BaO_2$)

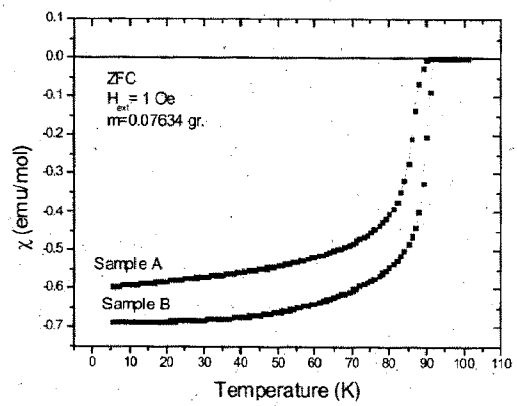


Fig 2. Magnetic susceptibilities versus temperature of the $YBa_2Cu_3O_{7-\delta}$ prepared with $BaCO_3$ (sample A) and $BaO_2$ (sample B).

The magnetic susceptibilities (emu/mol) versus temperature in the range 5-100K under an external applied field of 1 Oe in ZFC (Zero Field Cooling) is shown in Fig. 2; noticeably a saturated diamagnetic transition in both of the samples. Since the sharpness of the drop in the curves is a measure of the goodness or purity of the sample[5], because the magnetic susceptibility plot is a measure of all the existing phases in each sample. In the figure we can realise more impurity in sample A (prepared with $BaCO_3$)

than in sample B (prepared with $BaO_2$); this impurity phase in the first one was identified above as CuO by EDX analysis.

The transition temperatures $T_C$ were estimated from the point at which the first derivatives of the magnetic susceptibility curves reach their maximum value, as shown in Fig. 3, because these are the inflection points[5]. According with these, $T_C$ of $YBa_2Cu_3O_{7-\delta}$ prepared with $BaCO_3$ (sample A) is about 87 K and $T_C$ of $YBa_2Cu_3O_{7-\delta}$ prepared with $BaO_2$ (sample B) is 90 K. The little difference in the estimation of $T_C$ in the case of Y123 prepared with $BaCO_3$ is because the presence of the amount of secondary phase (CuO) mentioned above.

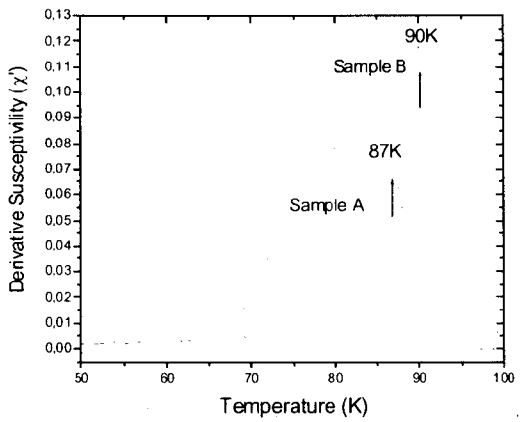


Fig 3. First derivative of the magnetic susceptibilities versus temperature of the $YBa_2Cu_3O_{7-\delta}$ prepared with $BaCO_3$ (sample A) and $BaO_2$ (sample B).

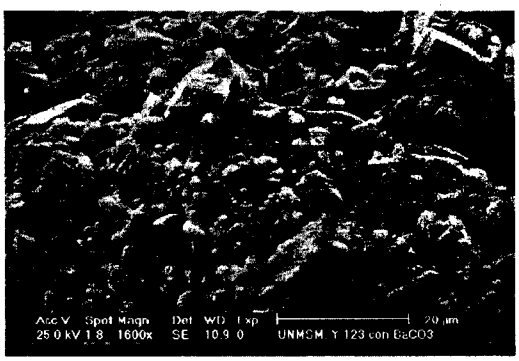


Fig.4 SEM micrograph of $YBa_2Cu_3O_{7-\delta}$ prepared with $BaCO_3$ (Sample A)

Surface morphology and microstructure characteristics of the $YBa_2Cu_3O_{7-\delta}$ samples prepared with $BaCO_3$ and $BaO_2$ were examined by SEM. Fig 4 shows SEM micrograph at 1600X amplification of sample A with a good granularity in its morphology. In sample B case, Fig. 5 shows the morphology at 3000X amplification; in this, the good granularity and very little needle shape grains confirm that two treatments were sufficient to prepare $YBa_2Cu_3O_{7-\delta}$ superconductor and to optimize the process.

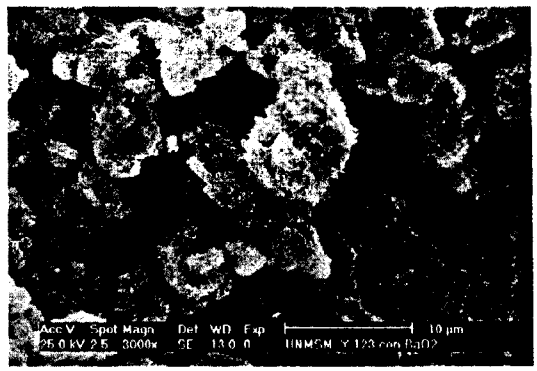


Fig.5 SEM micrograph of $YBa_2Cu_3O_{7-\delta}$ prepared with $BaO_2$ (Sample B).

The Energy Dispersive X-ray analyses (EDX analysis) for both samples are shown in Fig 6 and Fig 7; these were taken in the whole zones shown in the SEM micrographs above. The chemical composition of each one is the same: Y, Ba, Cu and O; the presence of Au peak in the EDX analysis is because samples were previously sprayed with Gold. The molecular weight percentage of the elements for sample A are: 12,5 % (Y), 54,3 % (Ba), 19,8 % (Cu) and 13,4 % (O), and for sample B are: 8,6 % (Y), 43,10 % (Ba), 26,9 % (Cu) and 21,4 % (O).

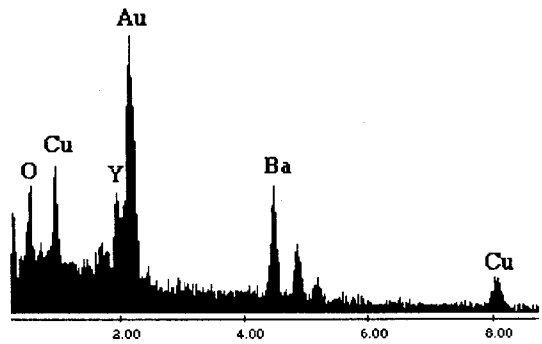


Fig.6 EDX of $YBa_2Cu_3O_{7-\delta}$ prepared with $BaCO_3$ (Sample A).

## 4. Conclusions.

The high $T_C$ superconductor $YBa_2Cu_3O_{7-\delta}$ prepared by the solid state reaction method using $BaO_2$ as reagent, responds higher qualification than prepared

with $BaCO_3$ because it has got almost pure phase and its $T_C$ is better identified. However the possibility that $YBa_2Cu_3O_{7-\delta}$ prepared with $BaO_2$ would respond better to the flow of electrical current must not be assured here since it would be necessary electrical resistivity behavior of the samples.

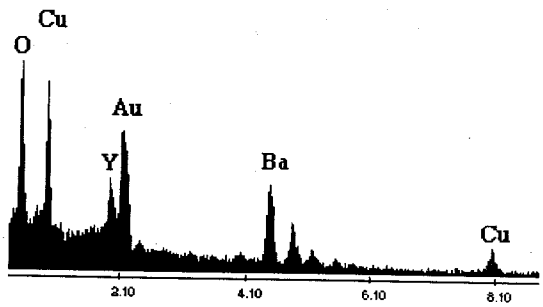


Fig.7 EDX of $YBa_2Cu_3O_{7-\delta}$ prepared with $BaO_2$ (Sample B).

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