

# Preparation of a superconducting ceramic of $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$

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## Abstract

The preparation of the superconducting ceramic  $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$  was carried out according to known procedures.

This process seeks to create a material that has an approximate critical temperature of 92 Kelvin and presents superconducting characteristics such as magnetic shielding and perfect electrical conduction.

# Introduction

The superconducting state<sup>1</sup> was discovered in 1911 and since then it has revolutionized the technological world with various inventions such as magnetic resonance machines, magnets that guide particles in an accelerator (LHC) and even in the railway industry.

In the decade of the 80s the issue of superconductivity took greater relevance with the discovery of superconducting ceramics, based on copper synthesized by Georg Bednorz and Alex Müller who were awarded in 1987 the Nobel Prize<sup>2</sup> due to their contributions to the physics of materials, thus starting the race for superconductors of high critical temperature<sup>3</sup>.

There are currently many types of superconductors with a wide variety of chemical compositions.

The  $T_c$  value ranges from 0.1 to 0.5

When the compounds cool below their critical temperature, the material enters a superconducting state, due to which the electrical resistance of the material decreases to a point close to zero and a reaction is generated in the presence of an external magnetic field and repels it, this phenomenon is known as the Meissner effect, which is also responsible for the well-known magnetic levitation in superconductors.

Superconductors have limits on the magnetic field they can repel and the current they can conduct which are known as critical magnetic field  $B_c$  and critical electric current  $I_c$ , when one of these two values is exceeded the superconducting state disappears.<sup>4</sup>

There are two types of superconducting materials:

Type I: Within this classification we usually find metals that are subjected to liquid helium temperatures:

-Hg (Mercury) with a  $T_c \approx 4.2$  Kelvin

-It is (Tin) with a  $T_c \approx 3.7$  Kelvin

-Pb (Lead) with a  $T_c \approx 7.2$  Kelvin

-Zn (Zinc) with a  $T_c \approx 0.9$  Kelvin

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<sup>1</sup> Superconducting state:

It is defined as the state in which a compound exhibits perfect conduction of electric current and shielding against magnetic fields. (REF. Introduction to superconductors: Yesenia Arredondo León, p. VII)

<sup>2</sup> The Nobel Prize in Physics 1987. NobelPrize.org. Nobel Prize Outreach AB 2023. FRI. 30 jun 2023. <<https://www.nobelprize.org/prizes/physics/1987/summary/>>

<sup>3</sup> It is the temperature below which the transition from normal to superconducting state takes place.

<sup>4</sup> <https://wp.icmm.csic.es/superconductividad/superconductividad/parametros-criticos/#:~:text=Corriente%20cr%C3%ADtica%3A%20La%20corriente%20en,material%2C%20la%20>

Type II: Among the materials that fall into this classification are:

-ceramics of Cuprates;

La<sub>2</sub>-XBaCuO with a  $T_c \approx 30$  Kelvin

YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-d</sub> with a  $T_c \approx 92$  Kelvin

-Other;

NbTi (Neobio-Titanium) with a  $T_c \approx 10$  Kelvin and  $B_c$  of 10 Teslas.

This discipline has searched for decades for a unified equation that explains the superconducting state.

For more than 40 years there was no theory that explained the superconducting state, but with the introduction of the concepts of quantum mechanics came a tool that helped scientists understand this strange state of matter.

Thus arose the theory of Soviet physicists Vitaly Ginzburg and Lev Landau of 1950 which explains the operation of the phase transition that a superconductor undergoes when cooling below its critical temperature.<sup>5</sup>

This theory is complemented by the BCS theory<sup>6</sup> named after its creators J. Bardeen, L. N. Cooper\* and J. R. Schrieffer which managed to explain the behavior of superconductors created until then with the theoretical model of a new quasiparticle called phonon, which corresponds to the collective vibrations of the atoms of a crystalline solid in a medium of Cooper pairs.

Together these two theories manage to explain type I superconductivity well, but it is flawed in trying to explain type II superconductivity.

Finally, we have the theory of Lev Landau's doctoral student; In 1957 Alexei Abrikosov theorized that knowing that in a type II superconductor there are areas that are superconducting and areas that are not, with this in mind Abrikosov used the concepts of topology<sup>7</sup> to explain the magnetic field vortices that penetrate the type II superconductor and how they move along the material under the influence of an external magnetic field.<sup>8</sup>

In YBCO ceramics there is a periodic crystal structure of perovskite-orthorhombic type with ordered CuO<sub>2</sub> planes which allow us to explain its superconducting state as follows:

YBCO is an anisotropic compound due to its laminar structure. It is formed by an arrangement of conductive planes of copper and oxygen (CuO<sub>2</sub>), through which the

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<sup>5</sup> Physics 127c: Statistical Mechanics Superconductivity: Ginzburg-Landau Theory

<sup>6</sup> <https://journals.aps.org/pr/abstract/10.1103/PhysRev.108.1175>

<sup>7</sup> "Study of geometric figures due to their respective properties and positions, without considering their shape or size"

<https://etimologias.dechile.net/?topologi.a#:~:text=La%20palabra%20%22topolog%C3%ADa%22%20est%C3%A1%20formada,considerar%20su%20forma%20o%20tama%C3%B1o%22.>

<sup>8</sup> TYPE II SUPERCONDUCTORS AND THE VORTEX LATTICE Nobel Lecture, December 8, 2003, by Alexei A. Abrikosov.

superconducting current flows, separated by charge reserve blocks that allow modifying the number of carriers in the CuO<sub>2</sub> planes. This structure gives rise to an anisotropic behavior, both in the normal state where we have a resistivity in the c direction greater than that we have in the ab plane ( $r_c / r_{ab} \sim 50$ ) and in the superconducting properties. The amount of current that can circulate parallel to the CuO<sub>2</sub> planes (planes ab) is greater than that which can pass through the c-axis.<sup>9</sup>

When performing heat treatments, an important part of the oxygen in the composition is lost, due to this we must perform oxygenations to adjust the stoichiometry.<sup>10</sup>

When performing the synthesis of the material, two different oxygen tanks were used, which may give us a reason for the differences in oxygenation between the two samples.

In sample 1 an old tank of approximately 3 years of use was used while in the second a new tank that was purchased two days before being used was used.

In addition to XRD<sup>11</sup>, it was determined if the material had deficiencies or excess oxygen in its composition by observing its coloration:<sup>12</sup>

Bluish: oxygen deficiency in its composition (presence of cations).

Greenish: Excess oxygen in the sample (presence of anions).

## Methodology

The following reagents should be weighed and incorporated into an Agatha mortar:

- ❖ CuO: 0.7157g  
99.0% purity  
Carbon Comp.: 0.01%  
Comp. Chloride: 0.005%  
Comp. Nitrogen: 0.002%  
Comp. Sulfur: 0.022%
- ❖ Y<sub>2</sub>O<sub>3</sub>: 0.339g  
99.99% purity
- ❖ BaCO<sub>3</sub>: 1.1840 g  
99.999% purity

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<sup>9</sup> PREPARATION OF SUPERCONDUCTING OXIDE YBa<sub>2</sub>Cu<sub>3</sub> OR 7-x BY THE SOL-GEL METHOD A. Bustamante, A. Osorio, J.C. González, M. Carhuacho, N. Salas, L. De Los Santos, N. De La Cruz and A. Díaz Faculty of Chemistry and Chemical Engineering. Universidad Nacional Mayor de San Marcos.

<sup>10</sup> <https://cdigital.uv.mx/bitstream/handle/123456789/5368/199726P47.pdf?sequence=2&isAllowed=y>  
Characterization of YBCO superconducting samples. - José Sergio Duran Niconoff.

<sup>11</sup> R-X diffractometry.

<sup>12</sup> Since ancient times, compounds of copper anions and cations have been used for the production of pigments: Eastaugh, N. et al: "Pigment Compendium: A Dictionary and Optical Microscopy of Historical Pigments". Butterworth-Heinemann, 2008.

*To achieve superconductivity, the material must be subjected to a heat treatment at its recrystallization temperature (950 degrees Celsius) thus seeking the nucleation and growth of new grains that contain few dislocations and therefore a reduction in the distance between the copper-oxygen perovskite conductive planes.<sup>13</sup>*

1. The materials are incorporated into an Agatha mortar.
2. They are compressed under high pressure to form a tablet.
3. One to two heat treatments are carried out at 950 degrees Celsius.
4. Two to three oxygenations are performed at 450 degrees Celsius.

## Experimental

Measurements were made by XRD confirming that the reagents were suitable for the synthesis of the material.

### Sample 1

The reagents were milled for 30 minutes and then pelletized at high pressure for a period of 20 minutes.

A first cycle of heat treatment was performed at 950o Celsius for 24 hours and then an attempt at oxygenation at 950 ° Celsius (aborted at 16 hours).

To make up for oxygen deficiencies they performed two cycles of oxygenation at 450o Celsius for 24 hours (Old oxygen).

By testing the ceramics and not presenting Meissner effect, XRD was made showing that the superconductor is in phase.

Subsequently, a heat treatment cycle was performed at 950o Celsius and finally an oxygenation cycle was performed at 450o Celsius for 24 hours (new oxygen).

### Sample 2

A second superconducting ceramic was prepared with the same stoichiometry.

The reagents were milled for 30 minutes and then pelletized at high pressure for 20 minutes.

A heat treatment cycle was performed at 950° Celsius for 24 hours and then an oxygenation cycle at 450° Celsius for 24 hours (new oxygen).

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<sup>13</sup> Idea based on the book Science and engineering of materials. -Ronald. D Askeland (pages 191-192).

# Results

Sample 1: At the end it was removed from the muffle and a test was performed at cryogenic temperatures to verify if there was a Meissner effect.

The sample showed a bluish coloration and absence of Meissner effect when subjected to cryogenic temperatures.

It is inferred from the bluish coloration that the ceramic has an oxygen deficiency.

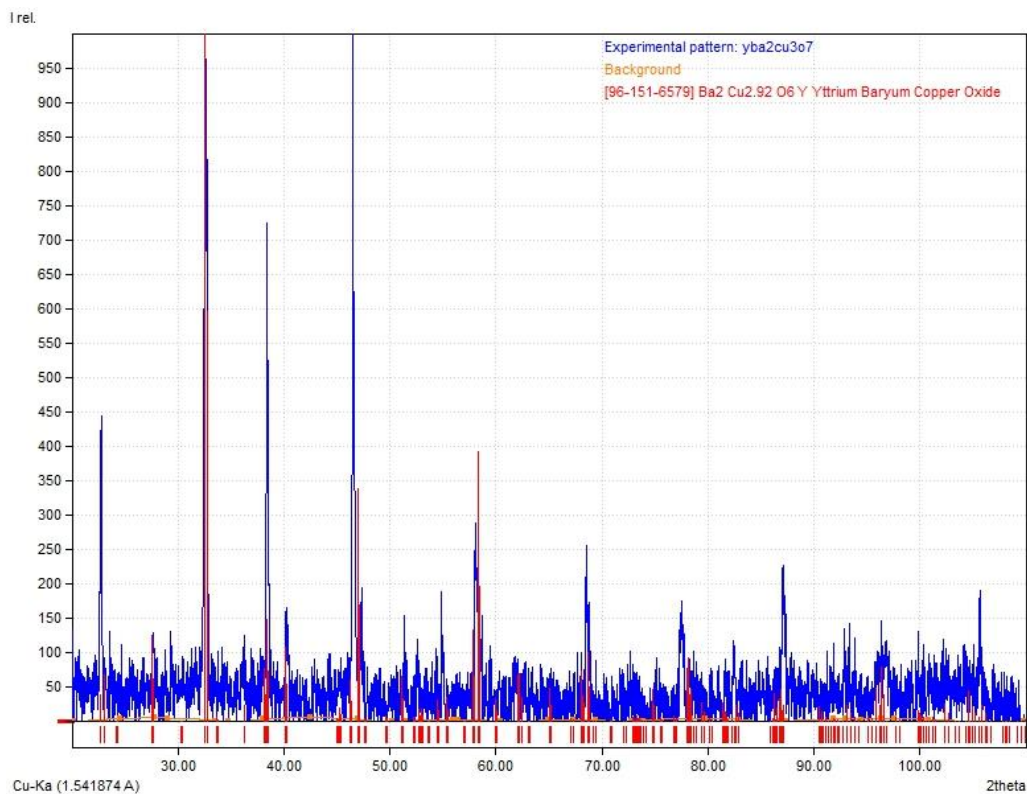
Sample 2:

This process gave us a ceramic with an irregular morphology.

A test was performed at cryogenic temperatures on a magnet rail presenting a magnetic levitation with a duration of 29 seconds.

D R-X:

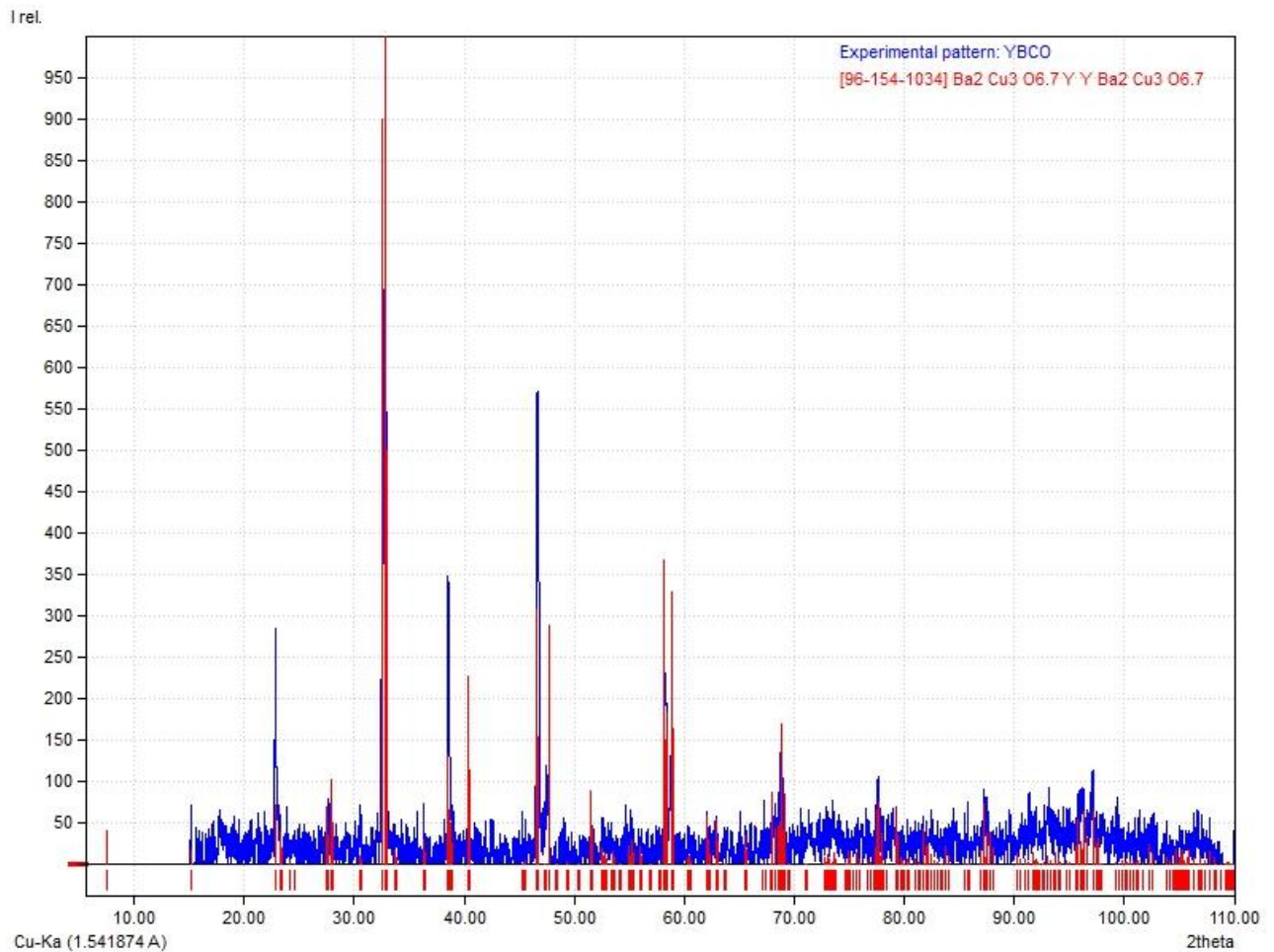
Sample 1:



(Fig.1 The diffraction pattern is in phase with that of YBa2Cu2.92O6 so it is classified as oxygen deficient).

When performing the XRD in sample 1 the analysis gives us patterns that coincide with a deficient YBCO oxygen which is confirmed due to the coloration presented at the end of its synthesized, also due to the absence of the Meissner effect we confirm the absence of superconducting state in the sample.

Sample 2:



(Fig.2 The diffraction pattern agrees with the phase of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub>, therefore its presence in the analyzed sample is confirmed).

In sample 2 I present superconducting state, visually confirmed by the presence of Meissner effect and when performing the XRD analysis we found YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> cells in the sample.

Analyses were performed with a Bruker D8 R-X diffractometer.

## Discussion/conclusions

According to the results obtained it is observed that the method used produces ceramics with superconducting capacity, when synthesizing sample 2 does not present the desired morphology something that can be explained because only one heat treatment was performed.

When performing the XRD in sample 1 the analysis gives us patterns that coincide with a deficient YBCO oxygen which is confirmed due to the coloration presented at the end of its synthesis, also due to the absence of the Meissner effect we confirm the absence of superconducting state in the sample.

In sample 2 I present superconducting state, visually confirmed by the presence of Meissner effect and when performing the analysis of XRD we found in the sample cells of  $\text{YBa}_2\text{Cu}_3\text{O}_7$ .

It also reaches an important conclusion about oxygen since in the oxygenated sample with the first tank no superconducting effect was witnessed, meanwhile in the oxygenated sample with the new tank the superconducting state was observed after only one oxygenation.

### **For the elaboration of samples it is recommended:**

Perform analysis of the reagents before performing the reaction to verify their purity.

Homogenize the reagents well during the first grinding.

Check the oxygen status before starting.

## Acknowledgments

Always grateful to my mother, unconditional company without whom this article would never have been possible.

To Teacher. Ana Bobadilla for the guidance she provided me when doing this work.

And finally to my lab colleagues:

Dr. Rodolfo Lopez

Lic. Francisco Dávila

Isaac Tellez

Lic. Sebastián Hernández Ruiz

For your comments and help in the lab that helped shape this article.

To Dr. Roberto Escudero for the opportunity to work in his laboratory and for his helpful laboratory tips.



# References

## -Annex I.

Magnetic susceptibility in superconducting materials; Journal of Physics Research. Vol. 12 N2 (2009) (J.C. González, A. Osorio, A. Bustamante)

The liquefaction of Helium (Sciences No.82 June 2008)

Tunnel effect and Josephson joints (Roberto Escudero)

Bulk superconductivity at 91 K in single-phase Oxygen-deficient perovskite Ba<sub>2</sub>YCu<sub>3</sub>O<sub>9-d</sub> (AT&T Bell laboratories: R. J Cava, R. B van Dover, D. W Murphy, S. Sunshine, T. Siegrist, J. P. Remeika, E.A Rietman, S. Zahurak and G.P. Espinosa 5/March/1987)

History of SQUID (B.D Josephson)

I was provided with notes on molecular orbital theory, basic concepts of conductivity and fermi bands, as well as content and notes that give indications on the sintering of YBaCuO samples (Synthesis of a superconductor YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-d</sub> and characterization of its magnetic properties: Prof. Esmeralda Lizet Martínez Piñero. IIM, Laboratory A-005), student notes

## -Summary

## -Introduction

### Superconducting state:

It is defined as the state in which a compound exhibits perfect conduction of electric current and shielding against magnetic fields. (REF. Introduction to superconductors: Yesenia Arredondo León,

### Critical temperature:

It is the temperature under which the transition from normal to superconducting state occurs.

### Ginzburg-Landau theory:

Physics 127c: Statistical Mechanics Superconductivity: Ginzburg-Landau Theory

### BCS Theory

<https://journals.aps.org/pr/abstract/10.1103/PhysRev.108.1175>

### Topology:

"Study of geometric figures due to their respective properties and positions, without considering their shape or size"

<https://etimologias.dechile.net/?topologi.a#:~:text=La%20palabra%20%22topolog%C3%ADa%22%20est%C3%A1%20formada,considerar%20su%20forma%20o%20tama%C3%B1o%22.>

### Abrikosov theory:

TYPE II SUPERCONDUCTORS AND THE VORTEX LATTICE Nobel Lecture, December 8, 2003, by Alexei A. Abrikosov.

### Superconductivity and perovskite-like structure:

PREPARATION OF SUPERCONDUCTING OXIDE YBa<sub>2</sub>Cu<sub>3</sub> OR 7-x BY THE SOL-GEL METHOD A. Bustamante, A. Osorio, J.C. González, M. Carhuacho, N. Salas, L. De Los Santos, N. De La Cruz and A. Díaz Faculty of Chemistry and Chemical Engineering. Universidad Nacional Mayor de San Marcos.

### XRD:

R-X diffractometry.

Staining by copper anions and cations:

Since ancient times, compounds of copper anions and cations have been used for the production of pigments: Eastaugh, N. et al: "Pigment Compendium: A Dictionary and Optical Microscopy of Historical Pigments". Butterworth-Heinemann, 2008

**-synthesis**

Muffle:

"A muffle is a furnace normally intended for firing ceramic materials and for smelting metals through thermal energy." ("Muffle: What They Are and What They Are For - Types and Applications") This oven is used when it is required to reach temperatures above 350 ° C. It is necessary to mention that only special materials can be used inside the muffle furnace, for example: A high quality porcelain, quartz or stainless steel crucible, due to the high temperatures that the oven can reach 1800 ° C. and the conditions of the atmosphere that it can generate in the process.

<https://www.cislab.mx/mufla-que-son-y-para-que-sirven/>

Liquid nitrogen has a boiling temperature of 77.33 Kelvin.

The critical temperature of the YBaCuO is 92 Kelvin.

Synthesis of a superconductor YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-d</sub> and characterization of its magnetic properties: Prof. Esmeralda Lizet Martínez Piñero. IIM, Laboratory A-005.

**-Methodology**

Idea based on the book Science and engineering of materials. -Ronald. D Askeland (pages 191-192).

**-Result of the synthesis**

**-Results**

**-Discussion and conclusions**

DOI: 10.5281/zenodo.8310177