MICROWAVE ASSISTED, LIQUID PHASE SYNTHESIS OF SUPERCONDUCTING MATERIAL

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In this work we present the synthesis of high temperature superconducting material $YBa_2Cu_3O_7$ (YBCO-123) using microwave synthesis in liquid phase. For this we used as starting materials aqueous solutions of yttrium, barium and copper nitrates with molar ratios of 1:2:3. Nitrates solution is mixed with a NaOH solution and para amino benzoic acid (PABA) as shell formation agent for precursor oxidic nanopaticles. The mixture thus obtained is heated under the microwaves until the reaction is completed and a black powder is obtained in aqueous suspension. The resulting material was calcined at 900°C for one hour, after preliminary drying and analyzed using XRD, FT-IR and SEM.

Keywords: superconductor, microwave synthesis, YBCO

1. Introduction

The wide range of applications for high temperature superconductors stimulated the research in this area with the aim to obtain them using simple methods with a high degree of purity.

Superconductors are used to generate strong magnetic fields used in medicine for MRI [1] and in transports for magnetic levitation (MAGLEV) vehicles [2]. Based on their diamagnetic properties, high temperature superconductors are used in this domain. Since high temperature superconductors HTCS are type II superconductors, a magnetic field can be generated inside Abrikosov vortexes which leads to a better stability of this type of levitation [3].

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Other domains where yttrium based high temperature superconductors have applications are automatics and telecommunications [4, 5], security systems, etc.

There are several methods to synthesize YBCO high temperature superconductors, out of which we remind: co precipitation [6], non-fluorine metal organic deposition MOD [7], MOD with fluorine [8, 9], auto combustion [10], sol-gel [11, 12]. Each of these methods has advantages and disadvantages. The trifluoroacetate-metal organic deposition (TFA-MOD) method's main disadvantage is the release of hydrofluoric acid which is very corrosive. The great advantage of this method is obtaining YBCO 123 in nanostructured form which enhances its properties. Through the use of non-fluorine synthesis, HF is not released, which proves to be an advantage, but if the primary materials contain carbon, $BaCO_3$ can be formed during synthesis, so starting materials that do not contain carbon are preferred.

Microwave synthesis of YBCO was first used by Bagurst et all in 1988 [13]; they chose this method in order to reduce the synthesis time and to obtain YBCO with optimum physical and morphological properties.

To avoid many of the disadvantages of the other synthesis methods, in this work we used microwave synthesis in aqueous solution in order to obtain YBCO in nanostructured form. Among the method's advantages is the reduced synthesis time and the obtaining of the nanostructured product. We used the microwave synthesis in liquid phase starting from a mixture of yttrium, barium and copper nitrates.

2. Experimental

All the used materials were of analytical grade and were purchased from Merck and Chimreactiv. In this experiment we used as starting materials yttrium nitrate 0.25M, barium nitrate 0.5M and copper nitrate 0.25M solutions. The mixture thus obtained was added dropwise in a solution consisting of sodium hydroxide 10% (to precipitate oxides and hydroxides) and para amino benzoic acid as a shell forming agent for precursor oxide nanoparticles. We assumed that during nanoaparticles formation, the stoichiometry from the solution is preserved and this leads to the formation of the desired compounds.

The obtained mixture is heated in an ordinary microwave oven that was modified to allow the evacuation of nitrous oxides that are released during the reaction. From the reaction results a black powder in aqueous suspension. The powder is dried in an oven for 24h at 105°C, calcined at 900°C for one hour and left in the oven to cool to room temperature. The calcination product as powder is analyzed using XRD, FT-IR and SEM.

A 8 mm diameter compact black pellet was obtained and used for further characterization. X-ray diffraction was performed on a PANalytical Empyrean

equipment which uses CuK_α radiation (1.541874), equipped with programmable divergence slit on the incidence side and a programmable anti-scatter slit mounted on PIXcel3D detector on the diffracted side. The scan was done by using Bragg Brentanno geometry with a step size of 0.02° and a counting time per step of 100 s in the range of 2θ=20-70°.

Scanning electron microscope (SEM) QUANTA INSPECT F field emission gun resolution 1.2nm was used to analyze sample surface morphology, and using energy dispersive X-ray (EDX) with the resolution to MnK_α 133 eV, elemental distribution in the powder was determined.

Raman and FT-IR analysis were carried out on Thermo Scientific Nicolet™ iS™50 FT-IR Spectrometer with The Polaris™ long-life IR source, Tungsten-Halogen white light source, built-in mid- and far-IR capable diamond ATR, NIR module with Integrating Sphere and Fiber Optic connections. The FT-IR spectrum was recorded between 100 - 1950 cm^{-1}. Raman spectrum was recorded between 130 and 570 cm^{-1}, laser frequency was 9391 cm^{-1}, detector InGaAs, optical velocity 0,4747 and aperture 1.

3. Results and disscusion

The microwave synthesis in liquid phase of yttrium based superconducting material proved to be suitable to obtain this composite and the resulted product was characterized by the techniques mentioned above.

During microwave synthesis the solution changes its color from blue to black, which indicates the formation of the desired material.

In XRD patterns (fig.1) are observed both YBCO and oxide compounds ($BaCuO_2$, CuO, YBCO-211) that represent impurities in the material; even in the calcined material, even if there are differences compared to the uncalcined material, impurities are still present. This could be a result of unfavorable stoichiometry.

The patterns obtained confirm the presence of both YBCO and other intermediate compounds ($BaCuO_2$, YBCO-211) which demonstrates that limiting the temperature to 100°C in aqueous solution, can’t lead to a completion of the YBCO synthesis reaction.

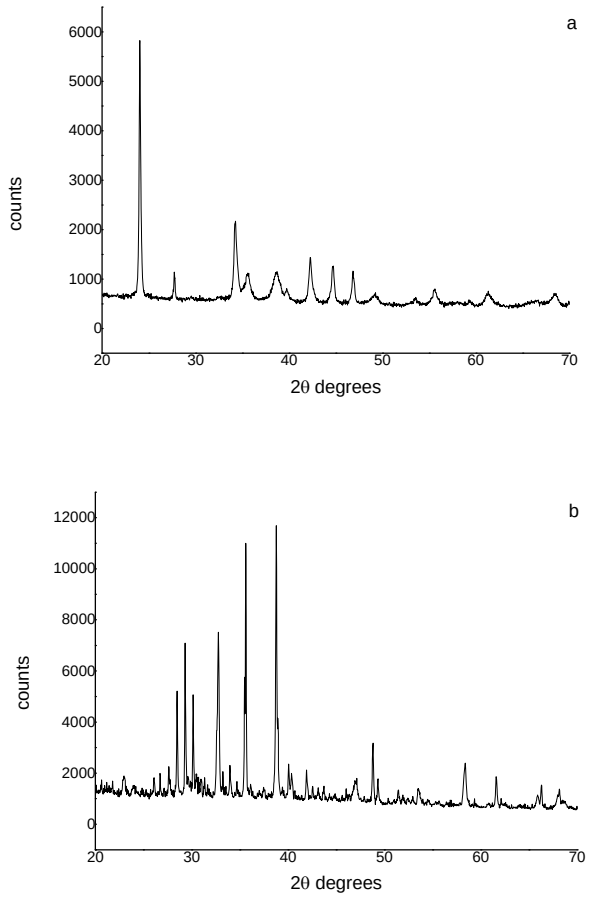

Fig. 1: XRD patterns for uncalcined (a) and calcined (b)YBCO 123

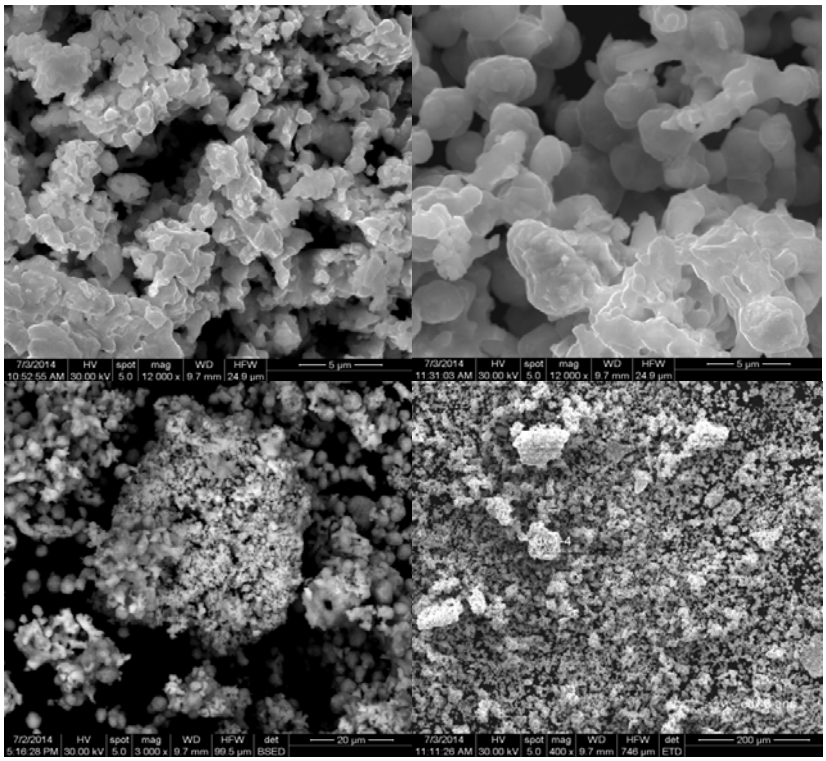

Fig. 2: SEM images for YBCO 123 obtained thorough microwave synthesis

Fig. 2 shows SEM images for YBCO 123 obtained using microwave synthesis. From the images there can be observed the rough surface and the micro structured grained morphology. This morphology is the result of the temperature at which the reaction takes place.

From the elemental analysis (Fig. 3) conducted after the calcination can be observed that the nitrogen and the carbon are not present in the sample, hence the sample is not contaminated with organic residues.

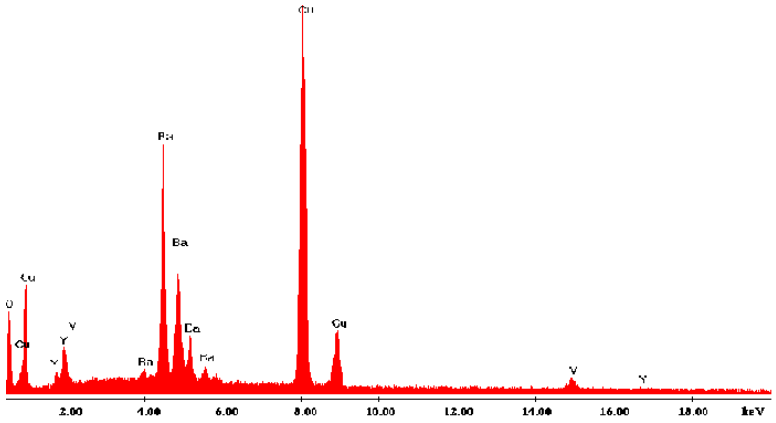

Fig. 3 EDS spectrum for YBCO 123 obtained using microwave synthesis

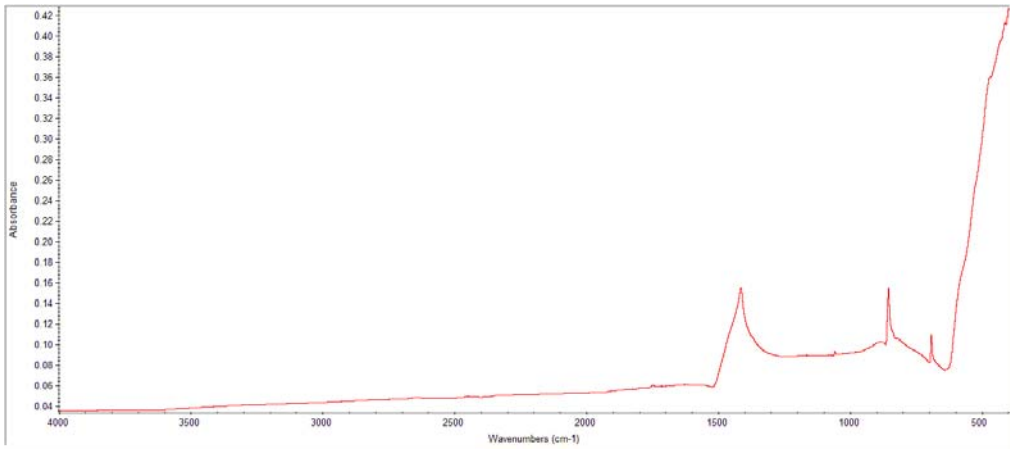

Fig. 4. FT-IR spectrum for YBCO 123 obtained using microwave synthesis

In Fig.4 is shown the FT-IR spectrum of the sample after calcinations process. It can be observed the specific peaks of barium oxide (1415.5, 860,690 cm^{-1}) and copper oxide (415 cm^{-1}) which was found also by XRD analysis.

4. Conclusions

YBCO 123 ceramic material was obtained by microwave in aqueous phase using a molar ratio of 1:2:3 between metal ions. The material resulted from the synthesis was dried in an oven for 24 hours at 105°C and then calcined for 1h at 900°C. The XRD pattern confirms the formation of YBCO in orthorhombic phase. Due to the temperature at which the reaction took place, besides the 123 superconducting phase, other substances that reduce superconducting properties of the ceramic material were formed.

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