

THERMAL DECOMPOSITION ROUTE FOR SYNTHESIS YBa₂Cu₃O_{7-x} SUPERCONDUCTIN NANORODS IN PRESENCE OF A NOVEL PRECURSOR

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ABSTRACT

In order to prevent agglomeration of high temperature YBa₂Cu₃O_{7-x} (YBCO) superconducting materials, the solid state processing method based on new and appropriate yttrium complex precursor was used without applying any surfactant. For the preparation of YBCO materials, instead of metal (Ba, Cu, Y) salts or metal alkoxides, the use of chelate type metal compounds as highly effective and stable precursors has been suggested. These have been prepared by the reaction of yttrium nitrate with 2-hydroxyacetophenone coordination compound for forming [tris(2-hydroxyacetophenato)yttrium(III)], [Y(HAP)3(H2O)3] as a new precursor for the synthesis of Y-123 nanostructures. These techniques provide proper control on nanoparticles size distribution. The generated steric hindrance due to the structure of the novel precursor, acts like a protecting agent and prevents from agglomeration. We synthesized nanorods of YBCO with the length about 320-350 nm and diameter about 60-90 nm with homogeneous morphology at calcination temperature 870 °C. The maximum values of transition temperature into the superconducting state have been found to be T_c(0) ~ 88 K with a temperature interval of ΔT = 3 K for YBCO nano-superconductor. The synthesized products were characterized by powder X-ray diffraction, fourier transform infrared spectroscopy (FT-IR), transmission electron micrograph and scanning electronic microscopy.

Key words: superconductor, coordination compositions; solid state process; nanostructures; YBa₂Cu₃O_{7-x}.

INTRODUCTION

Currently, YBa₂Cu₃O_{7-x} is still the best suited high-T_C superconductor for most applications [1]. A great effort has been made by scientists in both academia and industry to study this material. One of the most important and conventional synthesis techniques of YBCO is solid state reaction which despite

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some limitations and problems [2] in this method, due to its relative ease, is widely used for the synthesis of ceramics superconductors.

It has been found that several factors, such as the selection of the starting reactants, techniques employed and the calcination procedures play important roles in the phase formation and the characteristics of fabricated samples [3]. Most of the early fabricated compounds were synthesized by reacting Y_2O_3 and CuO with $BaCO_3$ [4–6] or BaO_2 [3, 7, 8] and a long time calcination was required because the carbonates decompose with a slow rate. So, the particles were agglomerated without a high quality structure. Another problem is carbon retention in sintered compacts that affected the critical current densities.

In recent years, considerable endeavors have been done for the development of nanometer-sized superconductors [9, 10] Control over crystal morphology is of the uttermost importance in superconductor fabrication. The ideal morphology for superconducting nanodevices and for applications in computer circuitry is the nanowires and nanorods. Herein, we report on the facile synthesis of YBCO nanorods via thermolysis of $[Y(HAP)_3(H_2O)_3]$ complex as a new yttrium source by conventional solid state method. To the best of our knowledge, this is the first report on the synthesis of Y-123 nanostructures via thermal decomposition of this new precursor. It was demonstrated that by using the coordination compositions, its possible to achieve the superconducting phase at lower calcination temperature due to compatibility of the coordination compositions in solution. So, chemical reactions and decomposition of $BaCO_3$ happens sooner and the calcination temperature was reduced to 870 °C.

METHODS OF SAMPLE MANUFACTURING AND ANALYSIS

At the first stage, we synthesized $[Y(HAP)_3(H_2O)_3]$ as an active precursor. This was prepared by the reaction of high-purity nitrate salt of Y with 2-hydroxyacetophenone ($C_8H_8O_2$) solution. The resulting solid residue, was filtered, washed and dried. FT-IR spectra of product confirmed the formation of Y complex because the stretching C=O vibration is shifted to lower frequency (about 1600 cm^{-1}) indicating the coordination of carbonyl group to the metal. Then, an appropriate amounts of BaO, CuO, $[Y(HAP)_3(H_2O)_3]$, were adjusted (Y:Ba:Cu = 1:2:3) and mixed to obtain pure YBCO. The powder was grinded and heated at temperatures 820 °C, 870 °C and 920 °C for 12 h with intermediate grindings. Finally, the powder annealed to 500 °C in oxygen flow, then cooled down to room temperature.

RESULTS AND DISCUSSION

The EDX spectra of the calcined sample confirmed the presence of Ba (~31 %.wt), Y (~ 14 %.wt), Cu (~ 42 %.wt) and oxygen (~ 10%.wt). XRD results of calcined sample at different temperatures shows that at 870 °C YBCO phase is formed with an orthorhombic structure along with Y_2CuBaO_5 phase as a minor phase with no trace of $BaCO_3$ as shown in *Fig. 1*. So, by comparing

with other work [4-6], temperature of calcination is reduced in this sample due to pliability and compatibility of coordination compositions in solution. By the use of Debye-Scherrer equation ($d = 0.9\lambda/\beta\cos\theta$), crystallite sizes of products were estimated and results indicated: 17.2 nm at 820 °C, 23.4 nm at 870 °C and 28.1 nm at 920 °C so the extra heating makes the larger size of grains.

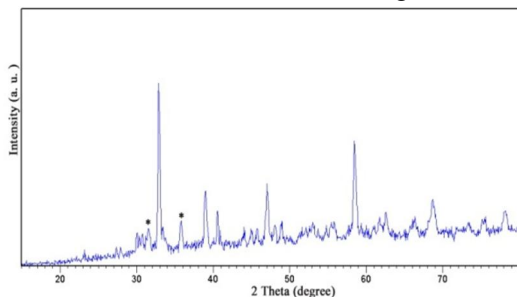


Fig. 1 – XRD patterns of the sample calcined at 870 °C. The peaks are indexed with (*) denote Y_2CuBaO_5 phase

precursors of yttrium were used as an appropriate precursor that have steric hindrance and therefore there was no need to use a surfactant for size control. The length of nanorods were estimated about 320-350 nm with diameters about 60-90 nm by TEM image as shown in *Fig. 2b*.

Measurements of temperature dependence of resistivity of the sample at 870 °C produced by using heat-treated new precursors method were done and indicated a superconducting state with an onset at $T_C = 88$ K and $\Delta T \approx 3$ K. As the magnetic field applied on the sample increases, visible decreases in T_C temperature value were observed.

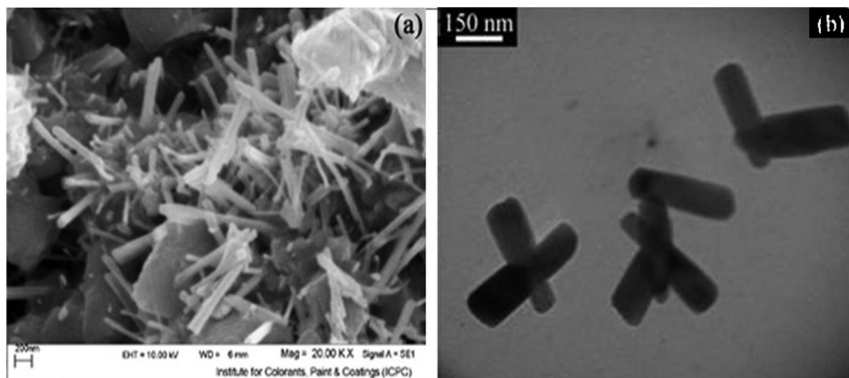


Fig. 2 – (a) SEM and (b) micrographs of product calcined at 780 °C

The SEM image shows that the sample calcined at 870 °C (*Fig. 2a*) is composed of nanorods due to the nucleation and growth of discrete nanoparticles in the big precursor matrix. According to the SEM images, starting materials selection plays a key role in the size control of YBCO. For this propose, the new

precursors of yttrium were used as an appropriate precursor that have steric hindrance and therefore there was no need to use a surfactant for size control. The length of nanorods were estimated about 320-350 nm with diameters about 60-90 nm by TEM image as shown in *Fig. 2b*.

The obtained critical temperatures were slightly reduced compared with typically quoted values of bulk YBCO material (91 K) [4] but were enhanced compared with of nanosize YBCO reported elsewhere (80 K) [11].

CONCLUSIONS

In summary, we have demonstrated the synthesis of YBCO nanostructures from the ([tris(2-hydroxyacetophenato)yttrium(III)], $[Y(\text{HAP})_3(\text{H}_2\text{O})_3]$) for the first time via a solid state process at different temperature for 12 h. It was found that by using of the coordination compositions as new raw materials, it's possible to achieve the superconducting phase at lower calcination temperature due to pliability and compatibility of coordination compositions in solution that causes the chemical reactions and decomposition of BaCO_3 happens sooner. This precursor has steric hindrance and therefore there was no need to use a surfactants for size control. At 870 °C, nearly single phase of YBCO is formed. From the results of SEM and TEM images, the as-prepared samples show relatively good morphologies corresponding to rod-like nanostructures with length of about 320-350 nm and diameters about 60-90 nm. The transition temperature to superconductivity of the YBCO was obtained about 88 K.

Acknowledgements

Authors are grateful to council of University of Kashan and Islamic Azadfor university (branch of Tehran Science and Research) providing financial support to undertake this work.

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