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Preparation of Pure $DyBa_2Cu_3O_{7-X}$ Nanocluster Superconductors using Biopolymer Chitosan

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We used sol-gel method for synthesizing nanoclusters of $DyBa_2Cu_3O_{7-X}$ high T_C type II superconductor in presence of biopolymer chitosan. In the first stage, the precursor and biopolymer aggregated into amorphous matrix and hydrogels are then formed by thermogelling. The fibrous nature of the biopolymer chitosan is retained at high temperatures up to 500 °C. After heating to 900 °C, complete decomposition of $BaCO_3$ and formation of the superconductor nanoparticles (with a diameter of 10-20 nm) occurred subsequently. Characterization of specimens was performed using scanning electron microscopy and transmission electron microscopy, supported by other techniques including XRD diffraction, energy dispersive X-ray, FT-IR spectrum and magnetic susceptibility measurements.

Keywords: Superconductor, Chitosan Biopolymer, Sol gel Method, Nanostructures, $DyBa_2Cu_3O_{7-X}$.

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1. INTRODUCTION

It is well known that, in the high-T_C superconductor $YBa_2Cu_3O_{7-X}$, the Y^{3+} ion can be replaced by most of the rare-earth ions, including the dysprosium ion Dy^{3+} without any appreciable effects on its superconducting properties [1]. $DyBa_2Cu_3O_{7-X}$ bulk superconductors have attracted due to high critical current density (Jc) and critical temperature (T_C) [2]. Dy–Ba–Cu–O bulk is one of the promising candidates for high field engineering applications such as flywheel energy storage and magnetic bearings [3] due to excellent field trapping ability.

Control over crystal morphology is of the utmost importance in superconductor fabrication [4]. It can be possible to control the morphology of superconducting crystals by catalytic outgrowth from discrete sites in the sol-gel matrix. One possible method for achieving this could involve the preorganization of barium ions within an appropriate polymer matrix. In this regard, we used the biopolymer chitosan in the sol gel processing of $DyBa_2Cu_3O_{7-X}$. Chitosan is a linear polysaccharide comprising glucosamine and N-acetylglucosamine that is characterized by high biocompatibility and biodegradability. Chitosan has the ability to preferentially sequester transition and post-transition metal ions from aqueous solutions. In addition, it is a remarkably stable biomaterial, able to withstand temperatures of up to 160 °C without undergoing any significant change in molecular structure [5]. So, we can limit the size of barium carbonate particles and restrict their spatial distribution by using this polymer. It has found applications in the medicine area as polymeric carriers for drug delivery [6] because of its ease and environmentally benign nature. In this work, synthesis of $DyBa_2Cu_3O_{7-X}$ (Dy123) nanoparticles was carried out in the presence of chitosan.

2. METHODS OF SAMPLE MANUFACTURING

Dysprosium, barium and copper nitrate dissolved in distilled water to achieve an overall cation stoichiometry of Dy:Ba:Cu = 1:2:3. Then, 10 mL of Dy123 solution was added to chitosan solution and was stirred continuously throughout the process using a magnetic stirrer until a blue viscous gel is obtained. The homogenous gel was then cast into Petri dishes and allowed to form a thin and flexible film. This film was heated to 500 °C then was calcined to 900 °C. Finally, the obtained powders annealed to 600 °C (for 1 h) under O_2 atmosphere. In order to investigation the role of chitosan, the other sample of Dy123 was prepared in the absence of chitosan.

3. RESULTS AND DISCUSSION

3.1 Structural Properties

FT-IR spectra of product shows that the chitosan retains to 500 °C and after 820 °C decomposition of $BaCO_3$ occurred gradually as the superconductor Dy123 phase formed. The XRD pattern of the sample calcined at 900 °C shows the single phase of Dy123 without any trace of the carbonate salts or impurity that has an orthorhombic structure, space group Pmmm [7]. The crystal size of the Dy123 nanoclusters was estimated by Debye–Scherrer equation ($d = 0.9\lambda/\beta\cos\theta$) and it was about 15.7 nm. Further analysis of the structural details from the EDAX spectra confirmed the presence of Dy (29.73 Wt %), Ba (36.78 Wt %) and Cu (33.49 Wt %) metals, as shown in Fig. 1.

It was reported that besides its good biocompatibility, chitosan has an excellent cell affinity [8] and provides discrete sites for the nucleation of barium carbonate nanoparticles throughout the fiber network of the chitosan matrix during the initial stage of calcination. Firstly, the chitosan diffuses through the precursor solution randomly and hydrogels are then formed by thermogelling.

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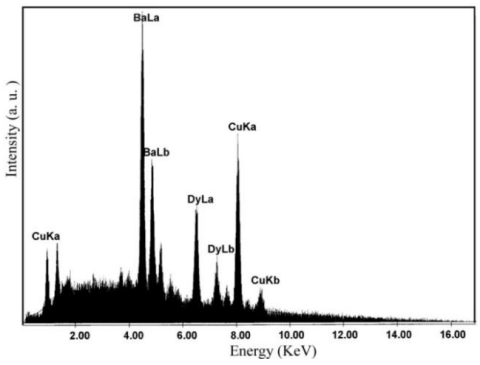

Fig. 1 – EDX spectrum of the synthesized sample at 900 °C

After heating to 500 °C, the chitosan polymer decomposes completely and sintering of the barium carbonate particles is minimized due to the stability and retention of fiber structure in the chitosan matrix on calcination.

3.2 Morphological Properties

Fig. 2 shows the SEM images of the uniform chitosan–Dy123 composite film. By heating to 500 °C, pores and holes are detected in the SEM image owing to partial decomposition of the polymer (Fig. 3) as the synthesized sample in the absence of chitosan at 500 °C presents a considerable porosity (Fig. 4).

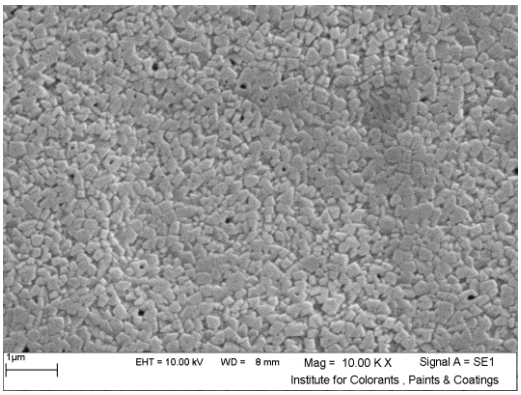

Fig. 2 – SEM image of uniform chitosan–Dy123 composite film at 35 °C

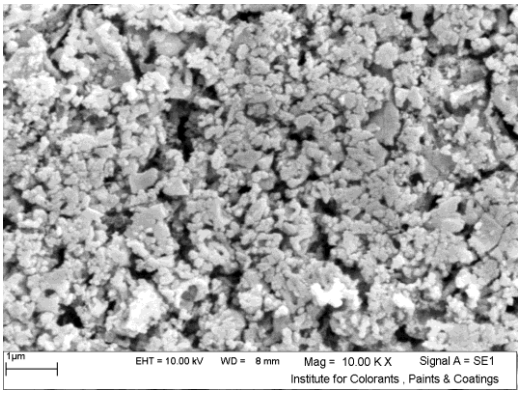

Fig. 3 – SEM image chitosan–Dy123 composite film at 500 °C

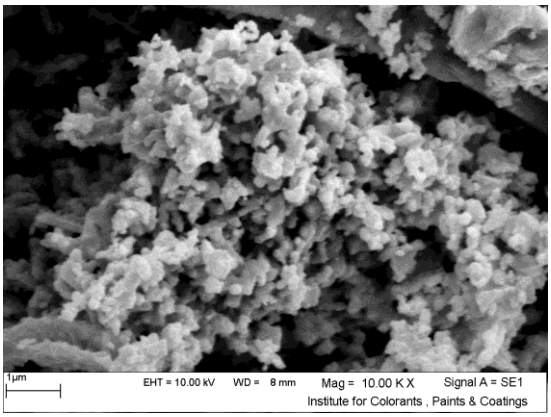

Fig. 4 – SEM image of the prepared sample Dy123 in the absence of chitosan at 500 °C

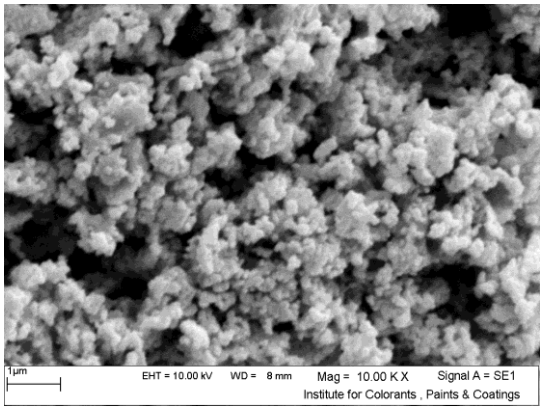

Fig. 5 – SEM image chitosan–Dy123 composite film at 900 °C

Fig. 5 gives an overall view of the surface morphology of the prepared samples at 900 °C and reveals that the products consist of tiny nanoparticles. TEM photograph of the products shows the assemblies of tiny particles with a diameter of 10 – 20 nm in the form of the nanoclusters of less than 100 nm of Dy123 (Fig. 6), which is in good agreement with the XRD analysis result. It is seen that the size of nanoparticles can be controlled by adjusting the amount of Dy123 precursor added to the chitosan solution. Magnetic susceptibility measurements showed that Tc of Dy123 nanoparticles is above 84 K [7].

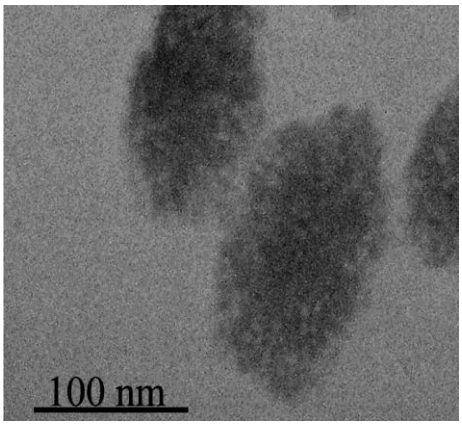

Fig. 6 – TEM image chitosan–Dy123 composite film at 900 °C.

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