

Microstructure and superconducting properties of Ca substituted $Y(\text{Ba}_{1-x}\text{Ca}_x)_2\text{Cu}_3\text{O}_{7-\delta}$ ceramics prepared by thermal treatment method



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ABSTRACT

The effect of Ca substitution in Ba site of $Y(\text{Ba}_{1-x}\text{Ca}_x)_2\text{Cu}_3\text{O}_{7-\delta}$, ($x = 0.00, 0.04, 0.08, 0.1$ and 0.125), ceramics prepared by thermal treatment method was investigated. Surface morphology, structural and superconducting were studied using field emission electron microscope (FESEM), X-ray Diffraction (XRD) and four-probe method. FESEM analysis showed an increasing of samples' grain size, homogeneity and compactness with increasing of Ca substitution. From XRD, the samples had orthorhombic crystal structure of space group $Pmmm$ besides small amount of unknown peaks. The critical temperature ($T_{c, R=zero}$) decreased from 87 K for the pure sample to 80 K for sample with $x = 0.08$, and it remained the same for samples with $x \geq 0.08$. Sample with $x = 0.04$ showed the sharpest superconducting transition (ΔT_c), which could be due to good microstructure morphology and better crystallinity.

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Introduction

$\text{YBa}_2\text{Cu}_3\text{O}_y$ (Y123) ceramic is the most promising materials for superconductor technology [1]. It has been proposed for usage in many applications such as energy storage systems, magnetic bearings and current limiters [2]. However, the applications of bulk polycrystalline Y123 is restricted due to its poor grain boundary conductivity that reduces the critical current density, J_c considerably [3]. Hence, synthesis of good quality Y123 specimen with improved grain boundary is a key for superconductors technology [4]. This may be achieved through chemical substitution [5,6] or suitable material preparation techniques that yield dense microstructure [7].

Chemical substitution is one of the strategies to improve microstructural and superconducting properties of Y123 [5,6]. Furthermore, the superconductivity in Y123 system is controlled by the oxygen content [8–11]. Both ionic substitution and application of high pressure on Y123 can control the level of electric charge of oxygen within the planes as a result of the manipulation of oxygen stoichiometry in the Cu–O chains [12–16]. Much work related to substitution into Y123 has been reported. Among these, substitu-

tion of Ca^{+2} into the Y^{+3} site in Y123 has received great attention as both ions have almost the same ionic size and different valence state [17]. This may result in the increment of carrier concentrations of Y123 system. Furthermore, substitution with Ca was found to increase the critical temperature, T_c , of oxygen deficient $\text{YBa}_2\text{Cu}_3\text{O}_y$ ($y \leq 6.5$) while it decreased the T_c in fully-oxygenated $\text{YBa}_2\text{Cu}_3\text{O}_y$ ($y \sim 7.0$) [18,19]. It has been reported that the J_c across grain boundary in Ca substituted Y123 is seven times higher than that in unsubstituted sample [20,21]. Earlier work showed that the Ca substitution on the Y site of Y123 has solubility up to 30% with reduction of T_c to 66 K [22,23], while it has higher solubility, up to 50%, with T_c reduction to 78 K when it substituted on Ba site [24]. Hence it is worthy to consider the Ca substitution in Ba site of Y123 for improving its microstructure properties.

Different synthesis techniques have been employed to prepare Y123 with the aim of improving the superconducting properties. Frequently, this material is produced by the conventional solid state method for which, metal oxides and carbonates are mixed and heated at a certain temperature [25]. Other synthetic ways, such as sol gel [26], co-precipitation [27] and spray drying [28] have continued to improve the composition and structures of Y123 leading to better superconducting properties. Recently, preparation of oxides by aqueous solution method using polymer as capping agent has received much attention [29,30]. This method

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is advantageous because it is simple, low cost, and can be used to obtain fine powders [31,32]. In this paper, we employed thermal treatment method to synthesize $Y(Ba_{1-x}Ca_x)_2Cu_3O_{7-\delta}$ ($x = 0.00, 0.04, 0.08, 0.1, 0.125$). The synthesis was based on aqueous solution of metals nitrates and a polymer such as polyvinyl pyrrolidone (PVP) that act as a capping agent without any other chemicals addition. By using XRD analysis, resistivity behaviour (ρ -T) and FESEM analysis, the effects of Ca substitution on Ba site of Y123 were investigated.

Experimental details

The $Y(Ba_{1-x}Ca_x)_2Cu_3O_{7-\delta}$ ($x = 0.00, 0.04, 0.08, 0.1$ and 0.125) ceramics were prepared by using the thermal treatment method [33,34,32]. In this method, stoichiometric amounts of metal nitrates, i.e. $Y(NO_3)_3 \cdot 6.6H_2O$, $Ba(NO_3)_2$, $Cu(NO_3)_2 \cdot 0.25H_2O$ and $Ca(NO_3)_2 \cdot 4H_2O$ were mixed and dissolved in 2% aqueous solution of 300 ml of polyvinyl pyrrolidone (PVP) that acted as capping agent at 80 °C. The weight ratio of total amount of metal nitrates to PVP in gram was 10:6. The solution was stirred for 2 h and then dried in the oven at 110 °C for 24 h to evaporate water and finally become solid like green gel. The gel was then crushed into fine powder using a mortar and pestle and then pre-calcined at 600 °C for 4 h. The powder was ground and calcined at 910 °C for 24 h and then cooled in air at the rate of 1 °C per min. The powder were ground and pressed into pellets and sintered in flowing of O_2 at 980 °C for 24 h and slowly cooled to room temperature at 1 °C per min. The phase formation and crystal structure of the samples were characterized using the X-ray Diffraction (XRD) (Xpert Pro Panalytical Philips DY 1861) in the scanning range of $2\theta = 20^\circ$ – 80° . Morphological and structural studies were conducted by using Field Emission Scanning Electron Microscope (FESEM) attached with Energy Dispersive X-ray (EDX), JEOL-JSM7500, while the critical temperature was measured by standard four point probe method.

Results and discussion

Formation mechanism

Fig. 1 depicts the proposed interaction between the Y, Ba, Ca, and Cu ions and PVP prior to the calcination procedure. $Y(NO_3)_3 \cdot 6.6H_2O$, $Ba(NO_3)_2$, $Cu(NO_3)_2 \cdot 0.25H_2O$ and $Ca(NO_3)_2 \cdot 4H_2O$ were combined with PVP in the presence H_2O acting as the solvent. As the solution is mixed, metallic ions attract amine groups of polymer chains via ionic-dipole interaction. Following this, a “mobility congealment” of metallic cations within the polymer depression was implemented, resulting from the absence of H_2O in the dry solution. The gradual disappearance of PVP throughout the calcination procedure and increased temperatures facilitate the formation and development of $Y(Ba_{1-x}Ca_x)_2Cu_3O_{7-\delta}$ nanoparticles, which then causes proximal particles to bind together. Once the calcination process was complete, PVP values, and unnecessary anions were eliminated from the sample, as $Y(Ba_{1-x}Ca_x)_2Cu_3O_{7-\delta}$ nanoparticles form, combine, and exceed interfacial surface energy, they amass into larger particles, the greater the temperature, the larger the resultant particles. It is proposed that PVP is contributing to the nucleation of $Y(Ba_{1-x}Ca_x)_2Cu_3O_{7-\delta}$ particles and hence acts as a manipulative tool to yield desired particle sizes [35,36].

Microstructure properties

Fig. 2 shows FESEM images of the pure Y123 powder sample calcined at 910 °C. The sample composed of particulates with the average grain size of $\approx 0.32 \mu m$. The grains have irregular shape,

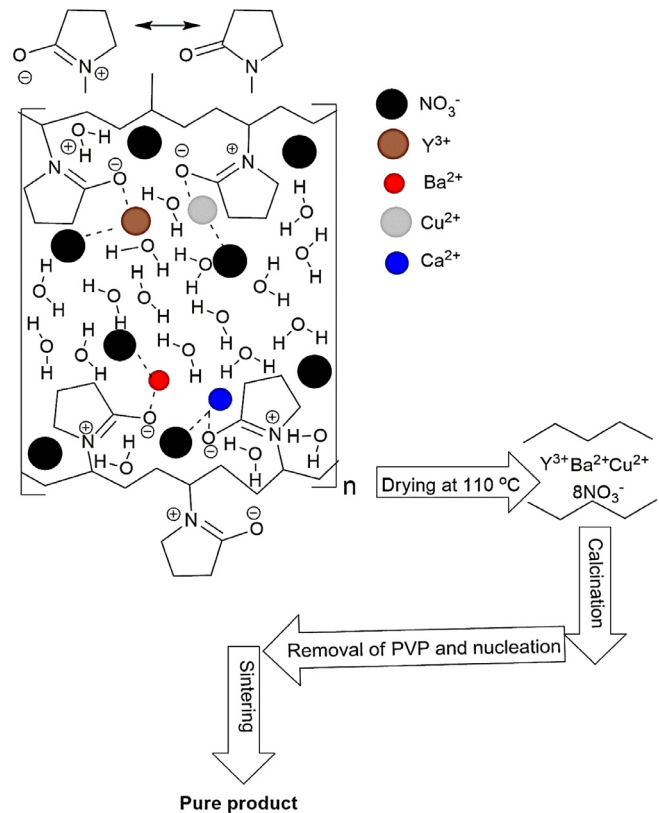


Fig. 1. A proposed mechanism of the interaction between PVP and metallic ions.

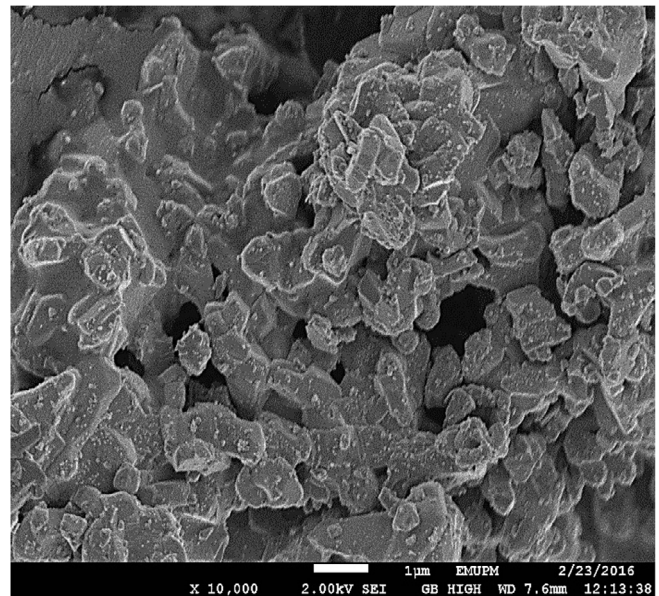


Fig. 2. FESEM images of pure Y123 powders after calcination at 910 °C.

distributed randomly with no alignment in certain direction and have variants of crystallinity along the sample surface.

FESEM images for the samples sintered at 980 °C taken on fractured surface of pellets for $Y(Ba_{1-x}Ca_x)_2Cu_3O_{7-\delta}$ with $x = 0.00, 0.04, 0.08, 0.1$ and 0.125 are displayed in Fig. 3 respectively. The pure sample has an average grain size of $0.65 \mu m$, with some voids and holes observed (Fig. 3 ($x = 0.00$)). It was observed that the

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