

Research paper

The effect of resolidification on preform optimized infiltration growth processed (Y, Nd, Sm, Gd)BCO, multi-grain bulk superconductor

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ABSTRACT

Controlling the microstructure of superconductors by incorporating the flux pinning centers and reducing the macro-defects to improve high field performance is the topic of recent research. In continuation, the preform optimized infiltration growth (POIG) processed $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (YBCO) system, Y-site substituted with three mixed RE (Nd, Sm, Gd) elements is investigated. 20 wt.% of (Nd, Sm, Gd) $_2\text{BaCuO}_5$ were mixed with Y_2BaCuO_5 and POIG processed in reduced oxygen atmosphere to obtain YNSG superconductor. No seed is employed for crystal growth; hence the processed samples are multi-grained. Microstructural and compositional investigations on YNSG revealed the presence of different phases in the matrix as well as in precipitates which are of the order of submicron to 4 μm . A large fraction of macro-defects (~6% of porosity) was observed in the YNSG sample. For reducing the unwanted macro-defects and refine the non-superconducting precipitates, processed YNSG sample is pressed and resolidified (by infiltrating the liquid phases once again) in an argon atmosphere and the structural, microstructural, elemental and superconducting properties are compared with YNSG and undoped samples. Due to spatial scatter in superconducting critical temperatures, caused by the distribution of different REBCO unit cells in YBCO, superconducting transition curve is sharp in YNSG, whereas the resolidified sample showed the broad transition due to solidified liquid phases.

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1. Introduction

Among the cuprate superconductors, $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (REBCO or RE-123, where RE = rare earth elements such as Nd, Sm, Gd, etc.) has drawn more attention because of low flux creep. Even though the flux pinning properties of REBCO materials are better than $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (YBCO or Y-123), the YBCO superconducting composites is found to be more interesting because of ease of preparation. Due to similar ionic radii of RE^{3+} and Ba^{2+} , $\text{RE}_{1-x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-\delta}$ (RE/Ba -ss) type, solid solutions will form if REBCO materials processed in an air atmosphere. The solid solutions are regions where the RE atoms have substituted either for RE' (where RE and RE' are different rare earths) or Ba within a unit cell, forming $(\text{RE}_{1-x}\text{RE}'_x)\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ (RE/RE' -ss) or RE-rich phase $(\text{RE}_{1+x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_y)$ respectively within the REBCO matrix. The RE/RE' -ss and RE/Ba -ss phase features a lower superconducting critical temperature (T_c) than the stoichiometric RE-123 materials [1].

Formation of a large fraction of RE/Ba -ss will be detrimental to superconducting properties [2,3].

To suppress the formation of RE/Ba -ss and promote REBCO, oxygen controlled atmospheres and the addition of extra Ba is developed. These processes not only remove the negative influence of the RE/Ba substitution on T_c but promotes the formation of RE/RE' -ss clusters which can act as compositional fluctuations [4–12], leading particularly to an improvement of pinning at intermediate and high magnetic fields. However, it is reported that formation of RE/Ba -ss in mixed rare earth superconductors cannot be suppressed completely even processed in oxygen controlled atmospheres [13,14]. The literature on ternary mixed rare earth superconductors are reported strong flux pinning due to stress fields arising from the presence of different RE-123/RE'-123 unit cells in the matrix [3,13–18]. The enhanced pinning is originated by the substitution of low- T_c RE-123 in the high- T_c 123 matrix sites, due to which the lattice mismatch arises among the neighboring unit cells containing RE-123/RE'-123 and enhances J_c up to high fields.

Chen et al. have proposed the infiltration and growth (IG) process [19] which is an alternative technique for melt growth (MG) technique. This method yields obtaining high-density YBCO/REBCO materials with refined Y/RE $_2\text{BaCuO}_5$ (Y/RE-211 or 211) particles

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well dispersed in the final microstructures. In contrast to the conventional MG process; IG process starts with a 211 preform fabricated through any suitable methods for ceramic powder, such as uniaxial compaction, cold isostatic pressing, etc. This 211 preform is then provided with a liquid phase (BaO + CuO) from a liquid source such as a RE-123 pellet. The preform pellets of liquid phase source and 211 kept in contact, upon heating above peritectic temperature; liquid phases will infiltrate into the 211 preform and subsequent cooling below T_p reacts to form a RE-123 matrix with left back 211 phase particles. The IG process possesses several advantages compared to the MG process: (1) near-net shape fabrication and (2) the refinement of 211 inclusions in the final RE-123 products and (3) minimization of macro-defects, etc., [20,21]. In the IG process, no liquid outflow occurs, and RE-123 does not undergo decomposition, as it does in MG process, and hence the effect of dopants on the growth and properties of RE-123 can be studied effectively.

However, IG process suffers from serious problems like inhomogeneities in the distribution of 211 particles and hence often results in non-uniform current densities across the volume of the samples [22–25]. Preform optimized IG (POIG) process recently developed in our group offers products with negligible macro defects along with homogeneously distributed finer Y-211 phase particles in the final YBCO composites. In this process, an optimized pressure of 460 MPa is applied to make the preforms, and the sintering temperature is optimized to strengthen the preforms to support liquid phase infiltration. Processed products support high J_c up to higher fields owing to enhanced flux pinning caused by the presence of submicron sized Y-211 particles and widespread twinning with twin widths of 25–100 nm present throughout the YBCO sample [26–32].

In literature, the studies on RE-123 systems with single, two and three RE elements compounded on the RE'-site is well established [33–46]. There is relatively less work reported on IG processed mixed REBCO superconductors. It is reported in the literature [47,48] that, 20% and 80% doping of RE in RE' has caused enhancement of superconducting properties. This is attributed to the creation of optimized stress field density associated with nanoscale compositional fluctuations. The aim of the present report is to study the effect of (i) the addition of 20 wt.% of Nd, Sm and Gd elements on Y-site in the POIG processed YBCO (referred to as YNSG) and (ii) the resolidification process on the POIG processed YNSG. The results on both POIG processed (Y, NSG)BCO superconductors are compared with YBCO sample processed in an argon atmosphere.

2. Experimental details

Precursor powders of Y-123, Y-211, and ($\text{Nd}_{0.66}\text{Sm}_{0.66}\text{Gd}_{0.68}$) BaCuO_5 (NSG-211) were prepared by a chemical route using citrate precursors. 20 wt.% of NSG-211 was added to 80 wt.% of Y-211 powder, tumbled into the intimate mixture. The preform powders are pressed into pellets under an optimized uniaxial pressure of 460 MPa to arrive Y-123, Y-211 and (Y, NSG)-211 preform pellets. The YNSG sample is synthesized with (Y, NSG)-211 preform under commercial grade Ar atmosphere using thermal cycle as shown in Fig. 1. In order to evaluate the role of processing conditions realistically, YBCO was also POIG processed under argon without the addition of any RE maintaining the same heat treatment schedule as shown in Fig. 1 and the final product referred to as YBCO-Ar. With an aim to eliminate the macro-porosity and refine the 211 phase particles in the YNSG sample, after processing, the YNSG sample was pressed at 540 MPa and once again processed by thermal cycle as shown in Fig. 1. The resultant sample after resolidification process is referred to as YNSG-R. Sample assembly (pressed

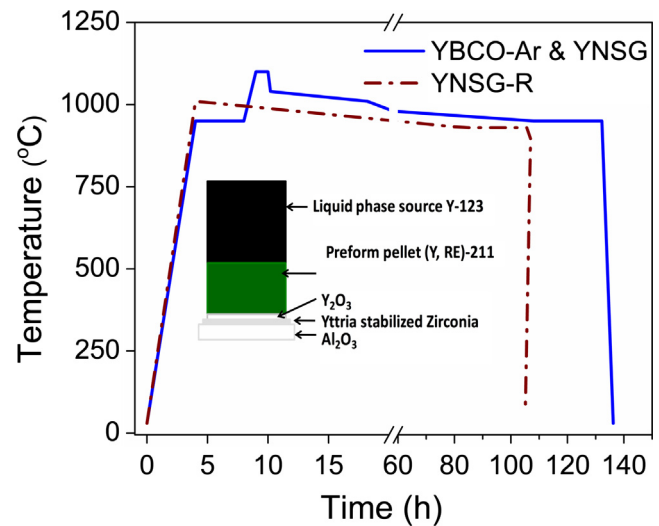


Fig. 1. Thermal cycles employed for POIG processing the YBCO-Ar and (Y, NSG)BCO samples.

YNSG sample + Y-123 pellet (liquid phase source)) was allowed to a temperature just above peritectic temperature of NSG-123 in 1% O_2 in Ar (i.e. $\sim 1040^\circ\text{C}$) [16] for infiltrating the liquid phases (BaO + CuO) into the pressed YNSG sample and from there slowly cooled with a cooling rate of 1°C/h through the peritectic temperature to a temperature of 930°C , which is much below the T_p of YBCO in argon atmosphere [49]. Then it was maintained there for 24 h to complete the grain growth, and the furnace was cooled to room temperature. No seed is employed for any of the studied samples. Hence, the processed samples are observed to be multi-grain with grain size up to ~ 3 mm.

After processing the samples, oxygenation was done for 110 h at 460°C for the structural phase transition to take place from non-superconducting tetragonal phase to superconducting orthorhombic phase. Superconducting critical temperature of the oxygenated composites have been determined by ac susceptibility ($\chi_{ac}(T)$) measurements in the temperature range $40\text{ K} \leq T \leq 100\text{ K}$ at driving ac field of rms amplitude 10 Oe with a frequency of 33 Hz employing a physical property measurement system (PPMS) based AC magnetometry system (ACMS; Quantum Design). Structural studies on oxygenated samples have been done by recording

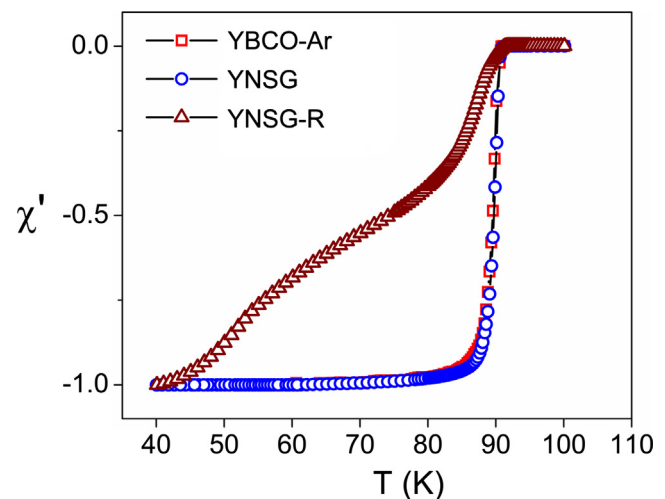


Fig. 2. Variation of the normalized real part of ac susceptibility (χ') with temperature. Sharp superconducting transitions are observed for YBCO-Ar and YNSG samples.

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