

Synthesis of Y_2BaCuO_5 nano-whiskers by a solution blow spinning technique and their successful introduction into single-grain, YBCO bulk superconductors



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ARTICLE INFO

Keywords:

Single-grain superconductor
Nano-whiskers
Solution blow spinning
Bulk YBCO
High temperature superconductor
Nano- Y_2BaCuO_5
 $YBa_2Cu_3O_{7-x}$

ABSTRACT

The ability of single grain Y-Ba-Cu-O (YBCO) bulk superconductors to trap large magnetic fields is due generally to the presence of embedded, non-superconducting Y_2BaCuO_5 (Y-211) phase particles, in optimum amount, in the superconducting $YBa_2Cu_3O_{7-\delta}$ (Y-123) phase matrix, which increases flux pinning and, hence, critical current density of the bulk material. The presence of smaller particles of Y-211 could be expected to improve further the superconducting properties of single grain, bulk YBCO samples, although the smallest Y-211 particles that have been engineered to date within the bulk microstructure are around 800 nm in size. It is extremely challenging to reduce the Y-211 particle size any further due to problems of particle agglomeration that originate, inevitably, from the effects of surface energy and reactivity at relatively high processing temperatures. In this work, we report a novel approach to the fabrication of Y-211 pinning centres in bulk YBCO superconductors in the form of nano-whiskers manufactured by a solution blow spinning technique. The Y-211 nano-whiskers, of which 62% were smaller than 500 nm, were added to the YBCO precursor powders to produce a single grain bulk sample by the buffer-aided top-seeded melt growth (BA-TSMG) processing technique. The resulting YBCO single grain, of diameter 20 mm, was able to trap a magnetic field of 0.63 T at 77 K with an associated critical current density of 3.9×10^4 A/cm² in self-field. The results of this study demonstrate clearly that the use of Y-211 nano-whiskers is a promising route for enhancing flux pinning in bulk YBCO single-grains, which is potentially significant for the development of high field engineering applications.

1. Introduction

Bulk, single-grain RE-Ba-Cu-O [(RE)BCO] where RE is a rare earth element or yttrium, high-temperature superconductors (HTS), such as YBCO, have significant potential for a variety of engineering applications, such as rotating electrical machines, flywheel energy storage systems, magnetic bearings and trapped field magnets [1–4]. For such applications to be feasible, however, single grain bulk superconductors are required generally to trap high magnetic fields and to carry large critical currents (J_c) over relatively large areas. The controlled addition of discrete, non-superconducting Y_2BaCuO_5 (Y-211) phase particles to the superconducting $YBa_2Cu_3O_{7-\delta}$ (Y-123) phase matrix generally enhances flux pinning in the bulk microstructure, which, in turn, leads to a direct improvement in the superconducting properties of the large single grain [4–10].

Significantly, both the content [8,11–14] and size [15–21] of embedded Y-211 particles play an important role in the control and enhancement the superconducting properties of YBCO single-grains. An important issue within this context is the engineering of finer Y-211 particles, for which several methods have been developed and applied successfully over a number of years. These techniques include the addition of a grain-refining agent such as Pt [22,23] and/or CeO₂ [24–26], which inhibits the coarsening of Y-211 particles during the peritectic reaction and single grain growth process, and, consequently, reducing the Y-211 particle size in the as-processed bulk microstructure. An alternative method is the use of small-sized Y-211 particles in the precursor powder [16,18], although to date Y-211 powders with grains not smaller than 800 nm have been used in large grain fabrication processes due primarily to challenges associated with the agglomeration of fine Y-211

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<https://doi.org/10.1016/j.ceramint.2018.11.068>

Received 4 September 2018; Received in revised form 15 October 2018; Accepted 10 November 2018

Available online 14 November 2018

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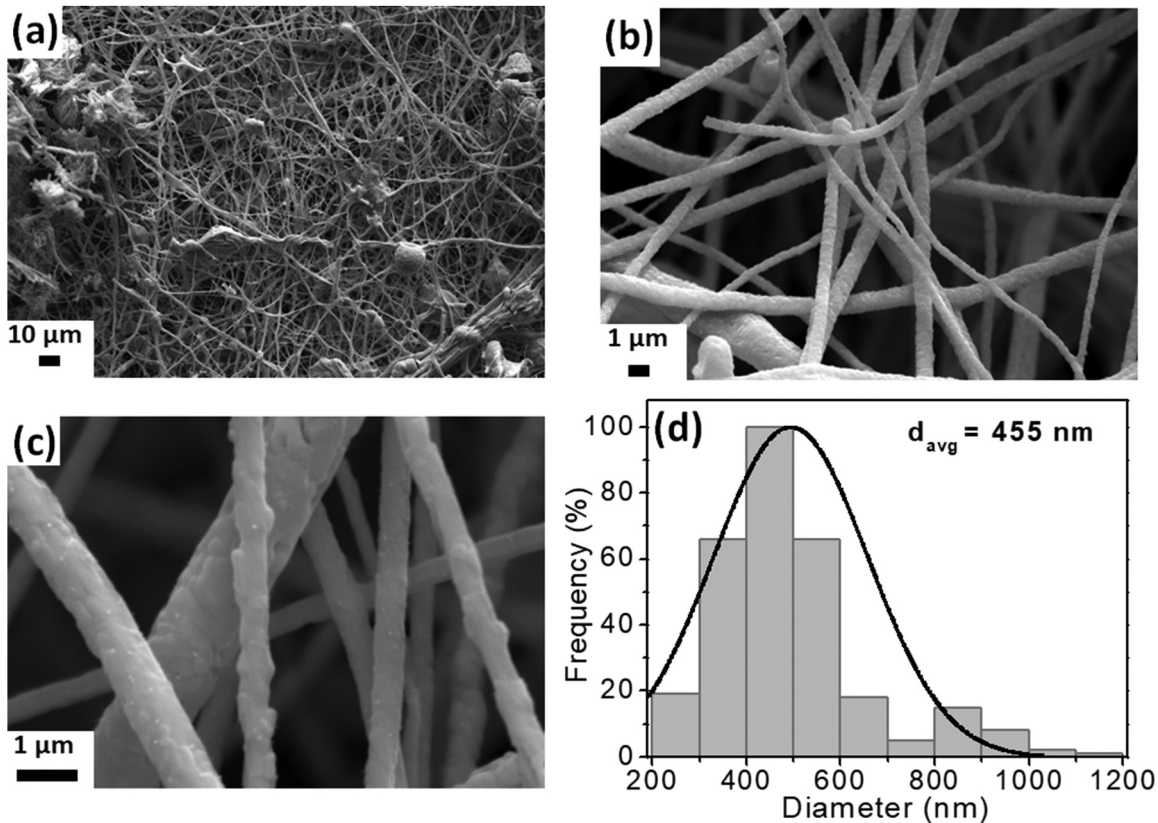


Fig. 1. SEM micrographs of the Y-211 nano-whiskers (a)–(c), and (d) shows their size distribution. The nano-whiskers obtained were found to have a granular morphology and were of an average diameter of 455 nm.

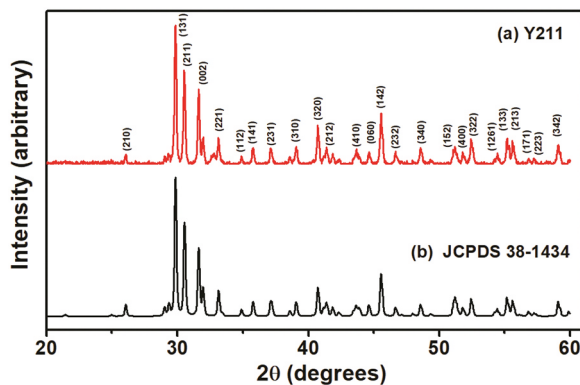


Fig. 2. (a) XRD spectra of Y-211 nano-whiskers produced by the solution blow spinning technique. The data correlate well with the JCPDS-38-1434 reference spectra in (b).

powder at elevated temperatures. Furthermore, optimum flux pinning in bulk superconductors is achieved generally by matching the size of the particle inclusions with the coherence length of the superconducting charge carriers (i.e. Cooper pairs), which is typically a few tens of nm at 77 K in YBCO [27–29]. This is clearly orders of magnitude smaller than the 800 nm Y-211 particle inclusion size achieved to date in the bulk, single grain microstructure, which represents relatively inefficient flux pinning and limited J_c .

In this work, we report a novel approach to the introduction of fine Y-211 particle inclusions in single grain YBCO bulk superconductors based on the fabrication of nano-whiskers with an average diameter of 455 nm via a solution blow spinning (SBS) technique [30,31]. The

nano-whiskers were mixed with commercial YBCO powders and the resulting precursor powder compacted into a pellet. A single-grain sample was produced subsequently using a buffer-aided top-seeded melt growth (BA-TSMG) process, as described elsewhere [32–37]. A standard YBCO sample (i.e. containing no added Y-211 nano-whiskers) was also prepared by BA-TSMG for purposes of comparison. The magnetic and microstructural properties of both resulting single grains were investigated in detail and are discussed.

2. Experimental details

2.1. Production and characterization of Y-211 nano-whiskers

Acetates (Ac) of Y, Ba, and Cu in the stoichiometric ratio of 2:1:1 were weighed in the appropriate proportions to yield the required Y-211 phase composition, along with polyvinyl pyrrolidone (PVP, molecular weight 360,000 g/mol) in an acetate: PVP weight ratio of 5:1 for the production of Y-211 nano-whiskers by the solution blow spinning technique. These reagents were then dissolved in a solution of 30% of propionic acid, 20% of acetic acid and 50% of methanol to yield a PVP concentration of 5 wt% in the final polymer solution. The precursor solution was loaded into a syringe and ejected onto a collector plate at a rate of 50 μL/min. The collector plate was positioned at a working distance of 30 cm from the tip of the syringe and rotated with an angular velocity of 100 rpm for continuous collection of the nano-whiskers. The procedure followed here was very similar to that described in detail elsewhere [31].

Ceramic, Y-211 whiskers were obtained by heat treating the polymer products obtained via solution blow spinning technique at 500 °C for 3 h to burn-out the PVP. These were heated subsequently

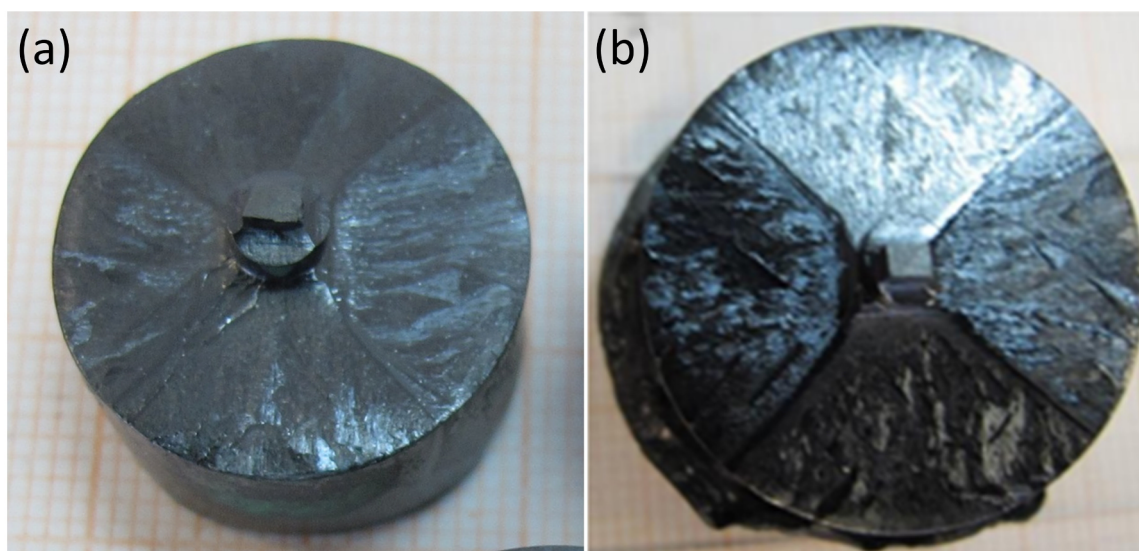


Fig. 3. Photographs of YBCO samples fabricated by the buffer-aided TSMG technique (BA-TSMG). (a) A standard sample and (b) a sample containing Y-211 nano-whiskers.

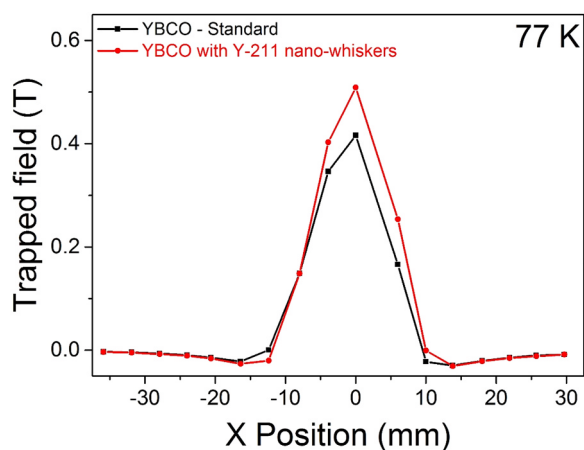


Fig. 4. Trapped field profiles measured at the top surface of the field-cooled YBCO samples. Standard sample (black in color) and the sample containing Y-211 nano-whiskers (red in color). The solid lines are a guide for the eye (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.).

in a tube furnace under flowing oxygen, initially at 850 °C for 14 h followed by 900 °C for 2 h. Both the heating and cooling steps of the burn-out process were carried out using a heating/cooling rate of 2 °C/min. Images of the product i.e. whiskers were obtained by scanning electron microscopy (SEM) using an EVO LS15 Zeiss SEM operating at 20 kV. X-ray diffraction measurements were performed using a Shimadzu XDR-6000 diffractometer with Cu-K α radiation, for a displacement (2θ) varying from 20° to 60° at a scan rate of 0.02°/min.

2.2. Fabrication of single grain YBCO via BA-TSMG

Primary precursor powders of Y-123 and Y-211 (99.9% pure, Toshiba Manufacturing Co. Ltd., Japan) corresponding to 75 wt% Y-123 + 25 wt% Y-211 by composition and 0.5 wt% CeO $_2$ (99.9% pure, Sigma Aldrich) were mixed thoroughly using an automixer for 3 h prior to the addition and further mixing of 0.5 wt% of Y-211 nano-whiskers, produced by the solution blow spinning technique. The mixture was

then calcined at 800 °C for 1 h to remove any alcohol / organic binders used to carry the nano-whiskers which prevented micro-cracking occurring later in the process. The resulting powder was then compacted into a pellet of 25 mm diameter using a uniaxial press. A buffer pellet of composition 75 wt%Y-123 + 25 wt% Y-211 was placed at the centre of the upper surface of the product green body and then capped with a dense Mg-NdBCO generic seed crystal [38]. A standard reference YBCO sample was also prepared under a similar sample arrangement and thermal processing conditions. The precursor sample assemblies were heat treated in a box furnace using a standard heat profile as described elsewhere [32,34]. Typically, the YBCO samples (melt processed with and without Y-211 nano-whiskers) were heated to a temperature of 1055 °C, which is well above the peritectic temperature (T_p) of the Y-123 phase (1005 °C in air) in order to achieve incongruent melting of Y-123 phase, and slow-cooled through T_p to promote the reaction of Y-211 with the BaCuO $_2$ + CuO liquid phase to form the Y-123 phase via heterogeneous nucleation and the growth of a single grain. A small degree of undercooling was maintained throughout the active growth region to minimize the probability of unwanted satellite grain nucleation. The as-grown single grain YBCO samples were oxygenated in a tube furnace to obtain an optimally doped orthorhombic, superconducting crystal structure. The critical temperatures (T_c) and critical current densities (J_c) of the samples were measured using a SQUID magnetometer. J_c was calculated from the width of the magnetic hysteresis loops using the extended Bean critical state model [39]. The field dependence of J_c was determined by applying magnetic field of up to 6 T at 77 K.

3. Results and discussion

3.1. Y-211 nano-whiskers

The initial heat-treatment burns-off the polymers from the product and results in the formation of a fabric-like structure of ceramic nano-whiskers, as shown in the SEM images in Fig. 1(a)–(c). The entangled nano-whiskers can be seen in Fig. 1(a) along with beads and other structures. The nano-whiskers exhibit a granular morphology, as can be seen in Fig. 1(b) and (c). Fig. 1(d) shows the size distribution of the nano-whiskers. It is worth noting that, despite exposing these whiskers to relatively high temperatures of 850 °C and 900 °C for reasonable time

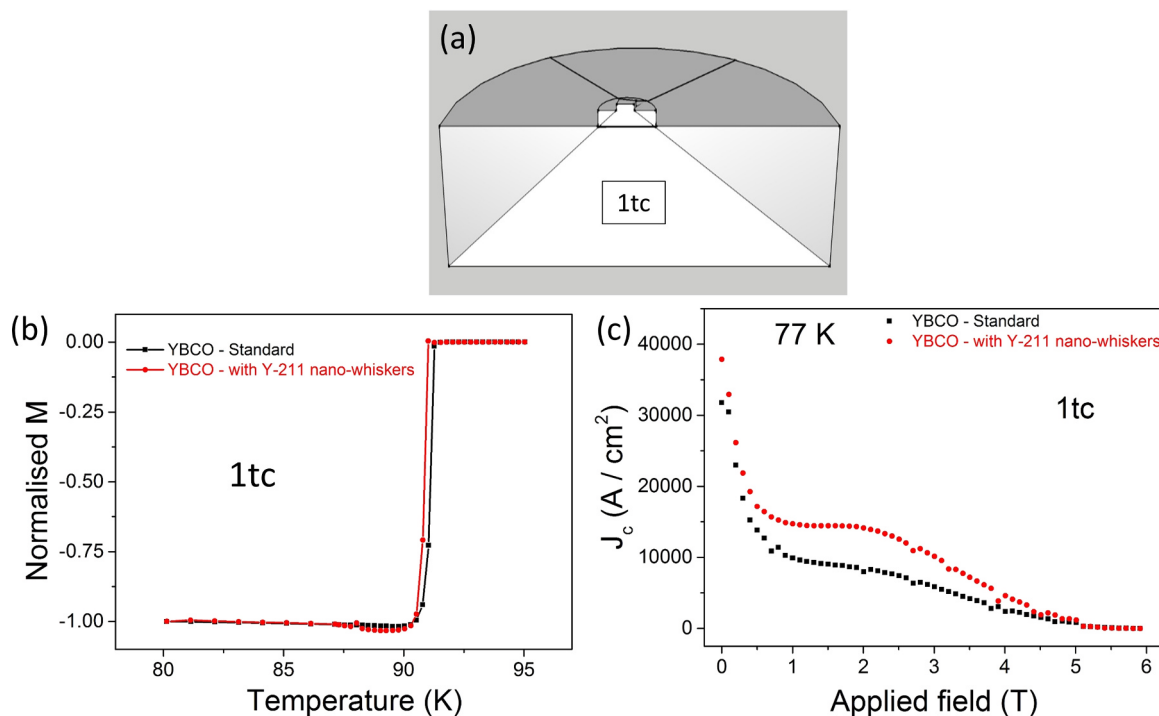


Fig. 5. (a) Schematic illustration of the bulk single grain and the location ‘1tc’ from where the sub-specimens were extracted for magnetic measurements. (b) Normalized magnetization as a function of temperature for the two sub-specimens. A critical temperature (T_c) of 91 K with a transition width < 0.7 K was observed for both samples. (c) J_c (calculated employing Bean extended critical state model [39]) as a function of applied field at 77 K for both the samples.

duration (14 h and 2 h, respectively), 62% of the nano-whiskers are smaller than 500 nm with an average diameter of 455 nm.

The XRD pattern of the nano-whiskers is shown in Fig. 2(a). The presence of secondary phases is not observed in the spectra and the characteristic peaks of the Y-211 phase are in good agreement with the reference pattern [JCPDS-38-1434 in Fig. 2(b)]. These data indicate that the Y-211 nano-whiskers obtained via the solution blow spinning technique are of high quality and that the heat treatment employed was effective in producing a pure Y-211 phase.

A standard YBCO sample and a sample containing Y-211 nano-whiskers were fabricated using the BA-TSMG processing technique, as described in Section 2, and the samples obtained are shown in Fig. 3.

The top and bottom surfaces of the as-grown bulk single grains were polished to obtain parallel, flat faces as required for the trapped field measurements. The samples were cooled in the presence of an applied magnetic field of 1.3 T to 77 K. The trapped magnetic flux density was measured using a rotating scanning array of 19 Hall sensors. The trapped field contours obtained from the surface of each of these samples are shown in Fig. 4. It can be seen that the sample containing Y-211 nano-whiskers trapped a higher field compared to the standard sample, indicating that the flux pinning strength in the bulk single grain is enhanced significantly by the addition of Y-211 nano-whiskers.

Small sub-specimens of approximate size $1.6 \text{ mm} \times 2.0 \text{ mm} \times 1.5 \text{ mm}$ were extracted from the central location of each single grain sample, indicated as ‘1tc’ in Fig. 5(a). A Superconducting Quantum Interference Device (SQUID) was used to determine the superconducting transition temperature of each sub-specimen in the presence of an applied magnetic field of magnitude 2 mT, as shown in Fig. 5(b). The measured T_c was ~ 91 K for both samples with a transition width < 0.7 K. The observed sharp transition to the

superconducting state establishes further the high quality of the samples. The measured field dependence of J_c for both the samples at 77 K is shown in Fig. 5(c).

The optical images obtained under low ($50\times$) and high ($1000\times$) magnification of the sample, and corresponding to location ‘1tc’ in the bulk single grain [as indicated in Fig. 5(a)], are shown in Fig. 6, from which the presence of pores and a homogenous distribution of Y-211 particles can be seen. The presence of two different sized Y-211 particles is also apparent in Fig. 6(b), including that of very fine Y-211 inclusions ($\sim 200\text{--}300$ nm in size), observed using SEM at higher magnification (Fig. 6(c)), generated from the Y-211 nano-whiskers following reaction with the liquid phase. These very fine Y-211 particles can enhance the flux pinning strength of YBCO at 77 K as indicated by the trapped field and $J_c(B)$ measurements.

4. Conclusions

High quality Y_2BaCuO_5 (Y-211) nano-whiskers have been fabricated by the solution blow spinning technique. SEM images show a granular morphology of the nano-whiskers with an average diameter of 455 nm. The solution blow spinning method employed has been shown to enable the reliable production of much finer Y-211 nano-whiskers than other fabrication techniques. The nano-whiskers were added to conventional precursor powders, which were then used to fabricate successfully a single grain bulk YBCO sample by the buffer-aided, top seeded melt growth technique. A sharp superconducting transition was obtained in this sample, which exhibited a self-field current density in excess of $3.9 \times 10^4 \text{ A/cm}^2$ at 77 K. Furthermore, we have demonstrated that these nano-whiskers can be incorporated successfully into bulk single grain YBCO to enhance the critical current performance of the bulk superconductor. The addition of Y-211 nano-whiskers is clearly a potentially valuable and novel approach to

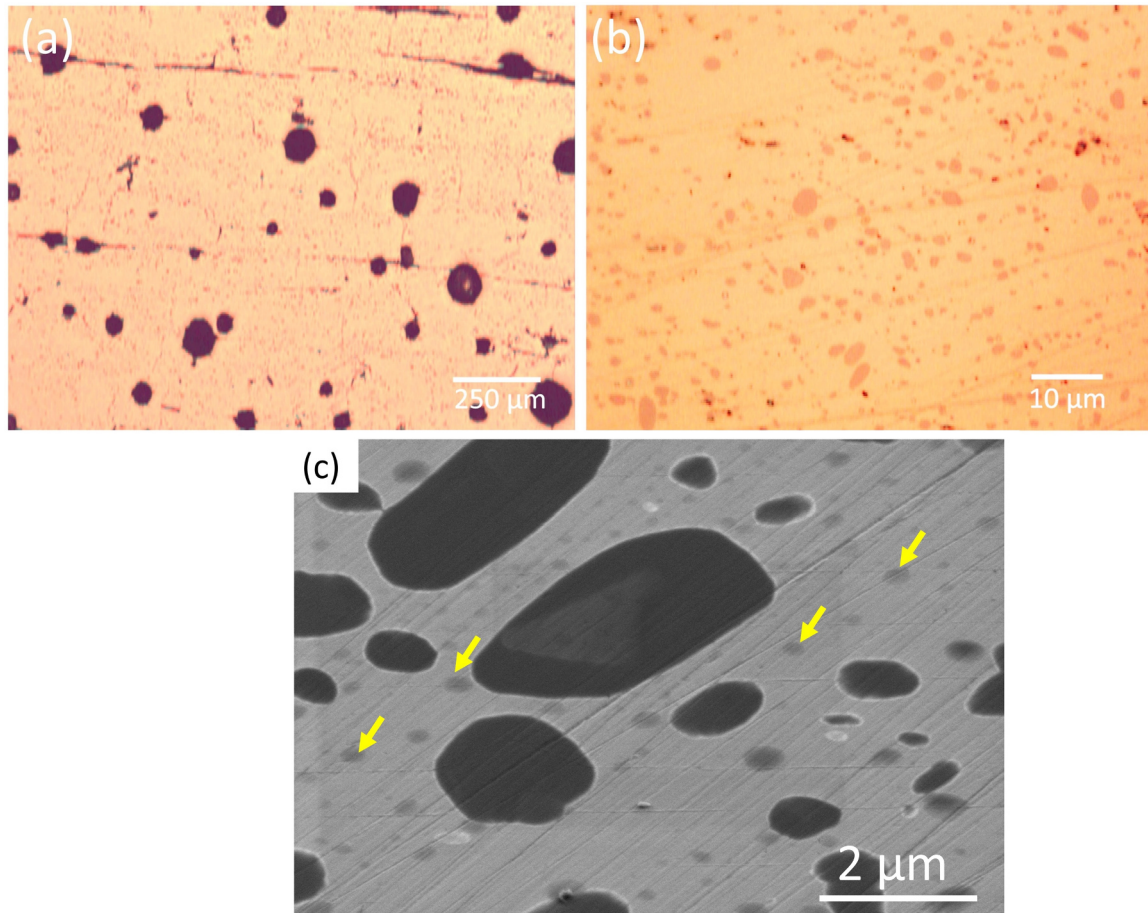


Fig. 6. (a) and (b) show optical micrographs obtained at different magnifications ($50\times$ and $1000\times$) from the centre [1tc, see Fig. 5(a)] of the YBCO bulk single grain containing Y-211 nano-whiskers. The same sample observed under higher magnification using a scanning electron microscope, as in (c), reveals the presence of fine-sized Y-211 particles ($\sim 200\text{--}300\text{ nm}$ in size) as a direct result of the addition of the Y-211 nano-whiskers.

improve the performance of bulk YBCO superconductors for high field engineering applications.

Acknowledgments

The authors from São Paulo State University (UNESP) acknowledge São Paulo Research Foundation (FAPESP) grant 2016/12390-6 for financial support. Authors also acknowledge EPSRC support (grant number EP/P00962X/1) and KACST. Additional data related to this publication is available at <https://doi.org/10.17863/CAM.26420>, in the University of Cambridge data repository. All other data are available directly within the publication.

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