



Fabrication of single crystalline Bi-2212 whisker with addition of Sb_2O_3 into the $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$ system and their thermal, structural and superconducting properties

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ABSTRACT

Single crystalline Bi-based superconducting whiskers have been fabricated from a melt quenched $\text{Bi}_2\text{SbSr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$ precursor using suitable heat treatment cycles. Whiskers fabricated in this study have 6–15 μm thickness, 60–95 μm width and 1–3 mm length. Crystallization activation energy of the material fabricated was calculated to be 609 kJmol^{-1} by Kissinger method. Structural and micro-structural changes with the heating treatment cycles were analyzed by XRD and SEM-EDX. It was found that whiskers have pure *c*-axis oriented single crystalline $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ (Bi-2212) phase with very smooth surface and without cracks and dislocations. Sb ions did not diffuse directly to the structure of whiskers. But Sb ions added to the system caused to decrease the number of whiskers. Temperature dependence of resistivity was analyzed along both the *a*- and *c*-axis. The superconducting transition temperature, T_c , of the whiskers was found to be 93.8 K for the *a*-axis resistivity and 90.9 K for the *c*-axis resistivity, respectively. The transport critical current density, J_c^{trans} was found as $2.6 \times 10^5 \text{ A/cm}^2$ at 10 and 1.1×10^5 at 70 K. Magnetization hysteresis (*M*–*H*) was investigated at three different temperatures (10, 20 and 30 K). Magnetic critical current densities, J_c^{mag} , of whiskers were determined using the Bean's Model.

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1. Introduction

After developments in the superconductivity technology in last decades, importance of single crystalline superconducting materials with low dimensions has increased due to their flexible structure, transportation of high current density, producing high magnetic field. Single crystalline superconducting materials are used in some electronic applications such as Josephson effect based devices or THz applications [1,2]. The $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ (Bi-2212) superconducting whiskers is one of candidates for recent developing technology. They can be fabricated in 1–10 mm length by different growth technique and starting compositions [3–5]. Other advantages of the single crystalline Bi-based whiskers compared to the polycrystalline superconducting materials can be given as layered crystallographic structure and pancake and Josephson vortex structure [6,7]. Applied magnetic field causes two different vortex formations in the single-crystalline $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ system: When a magnetic field is applied parallel to the *a*–*b* plane, the elongated Josephson vortices forms between the planes [8]. In the

other hand, when a magnetic field is applied parallel to the *c*-axis, a pancake–vortex structure forms within the conducting planes. The pancake vortices play a role on pinning of the Josephson vortices, which enhance nonlinearity in current–voltage (*I*–*V*) characteristics. By using this formations, new functional devices based on the vortex motion depending applied magnetic field have been developed [9].

In order to fabricate long whiskers, different fabrication techniques have been developed such as “direct growth from the melt”, “seeded route” and “glass-precursor” [3,10,11]. But, the longest whiskers are obtained only by using glass-precursor technique [12, 13]. The technique has some advantages: Firstly, in the stage of glass material production, energetically meta-stable and dense glass structure is obtained and controlled crystallization can be carried out by careful heat treatments. Secondly, glassification creates some regions with high stress in the material [14,15]. Stress can behave as a driving force for ionic motion in the material. During heat treatment, thermal energy increases the velocity of ions and therefore whisker growth becomes easier.

There are several studies in the literature about Sb effect on the superconducting properties of the Bi–Sr–Ca–Cu–O (BSCCO) system [16–20]. It was reported that Sb ions incorporated the interstitial sites in the system rather than occupation of the sites

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substituted by Sb. Superconducting transition temperature, T_c , was found between 78 K and 90 K, depending on the Sb concentration. It was revealed that Sb substitution at 10–20% lead to the achievement of the optimum oxygen content and thereby to improvement in the superconducting properties. However, there is no study on the Bi-2212 whisker formation in Sb substituted BSCCO system in the literature.

It is known that Sb ions have an important effect on glass materials due to its glass making ability. In this study, we have investigated the effect of Sb on the whisker formation in the $\text{Bi}_2\text{SbSr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+x}$ ($\text{Bi}_2\text{Sb1-223}$) system. The Bi-2212 whiskers have been fabricated using glass precursor method and investigated their thermal (Differential Thermal Analysis (DTA) and Thermo Gravimetric Analysis (TG)), structural (X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray analysis (EDX)), electrical (Temperature dependence of Resistance ($R-T$), Current–Voltage Characteristic ($I-V$)) and magnetic (applied magnetic field dependence of magnetization ($M-H$)) properties.

2. Experimental details

Appropriate amount of high purity (99.9%) Bi_2O_3 , Sb_2O_3 , SrCO_3 , CaCO_3 and CuO powders were weighted to give a nominal composition of $\text{Bi}_2\text{SbSr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+x}$ and mixed for 2 h in a mortar. The mixture was melted in an $\alpha\text{-Al}_2\text{O}_3$ crucible at 1250 °C for 3 h. The molten material was rapidly quenched between two cold copper plates. So, ~0.7–1.4 mm thick quenched plates (glass materials) were obtained.

Heat treatments were carried out between 400, 500, 600, 700, 800 °C for 50 h and 820, 825, 830, 835, 840, 845, 850, 860 and 880 °C for 50–150 h with intervals of 10 h used each heat treatment temperatures, respectively. The new glass pieces taken from same glass batch were used for each heat treatment. Heating and cooling rates during heat treatment were chosen as 10 and 1 °C/min, respectively. At heat treatment temperatures above 820 °C, it was observed that whiskers on the surface of the material were grown in different lengths. Heat treatment details are presented in Table 1.

Crystallization kinetics and mass changes were investigated by using differential thermal analysis (DTA) with Al_2O_3 reference material and Thermo Gravimetric (TG) results under oxygen atmosphere. Shimadzu TA-50 and Shimadzu TA-60 system are used for DTA and TG measurements, respectively. Activation energy for crystallization was calculated by Kissinger method. The Avrami parameter was calculated using the crystallization activation energy and DTA data.

The structural characterization of the glass and the heat treated samples was investigated by X-ray diffraction (XRD). Scan speed was selected as 2° min^{-1} in the range of $2\theta = 2-60^\circ$. Automated Rigaku RadB Dmax X-ray diffractometer with $\text{CuK}\alpha$ ($\lambda = 1.5405 \text{ \AA}$) radiation was used for the XRD analysis.

The micro-structural and compositional characterization of both the glass and the heat-treated material were performed with Leo EVO-40 VPX scanning electron microscope (SEM) and Bruker detector 4010 energy dispersive x-ray spectroscopy (EDX).

$M-H$ measurements were carried out by Cryogenic Q-3398 vibrating sample magnetometer (VSM) at three different temperatures, 10 K, 20 K and 30 K up to 5 T. The measurements were performed on whiskers in the same batch which were also used for $R-T$ measurements. An amount of whiskers on the main matrix (~90 mg) was collected and placed into a non-magnetic capsule. The capsule was inserted into the VSM system and then the measurement was performed.

$I-V$ characteristics, temperature dependence of resistivity ($R-T$) and the magnetic properties ($M-H$) of the whiskers fabricated were

Table 1

Summary of the growth conditions of the whiskers.

Heating rate (°C/min)	Cooling rate (°C/min)	Heat treatment conditions		Whisker length (in the range of)
		Temperature (°C)	Time (h)	
10	1	400	50	–
10	1	500	50	–
10	1	600	50	–
10	1	700	50	–
10	1	800	50	–
10	1	780	50	Not observed
10	1	780	150	Not observed
10	1	820	50	Not observed
10	1	820	150	Not observed
10	1	825	50	~95nm–0.01 mm
10	1	825	150	~200nm–0.1 mm
10	1	830	50	~0.05–0.1 mm
10	1	830	150	~0.09–0.5 mm
10	1	835	50	~0.06–0.3 mm
10	1	835	150	~0.1–1.2 mm
10	1	840	50	~0.1–1.3 mm
10	1	840	150	~0.9–2.0 mm
10	1	845	50	~0.1–2.1 mm
10	1	845	140	~1.5–3 mm
10	1	845	150	~1.4–2.8 mm
10	1	850	50	~0.1–1.6 mm
10	1	850	150	~1.4–2.8 mm
10	1	855	50	~0.1–1.7 mm
10	1	855	150	~1.4–2.8 mm
10	1	860–880	50	Not observed
10	1	860–880	150	Not observed

carried out by closed cycle Leybold LT-10 cryostat system and Cryogenic Q-3398 vibrating sample magnetometer (VSM) and Quantum Design Physical Property Measurement System (PPMS-7) under the magnetic field between 0 and 5 T. The in-plane (a -axis) resistivity and out-of-plane (c -axis) resistivity were carried out for the whiskers fabricated at 845 °C for 140 h (optimum whisker growth condition as seen in Table 1). The studies on whiskers showed that the growth direction, or the axis along the length of the superconducting whiskers which is perpendicular to the surface of the main matrix, is the a -axis. Otherwise, the axis parallel to the surface, or the axis along the thickness of the whiskers, is the c -axis [3]. In this context, the contacts were made as follow: For a -axis measurement, the four electrodes were contacted the upper surface of whisker. Otherwise, for c -axis measurement, the two electrodes were contacted the upper surface of whisker and the others contacted the bottom of surface of whisker. All the contacts for both a -axis and c -axis resistivity measurements were made with silver and gold deposition as described in [21].

From $I-V$ curve, Transport critical current density, J_c^{trans} was determined by increasing the current applied to the whisker, using the voltage criteria of 1 μV . Magnetic critical current density, J_c^{mag} was calculated by using Bean's equation (equation (4)). Details have been explained in Section 3.4.

3. Results and discussion

3.1. DTA and TG analysis

Fig. 1 shows DTA patterns of the $\text{Bi}_2\text{SbSr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+x}$ system at the heating rates, α , of 5, 10, 15 and 20 °C/min under oxygen atmosphere and DTA data obtained are presented in Table 2. Glass transition temperature, T_g , were found between 382 and 420 °C and the first crystallization temperature, T_{x1} , between 460 °C and 473 °C, depending on the heating rate. We believe that T_{x1} corresponds to nucleation assisted first crystallization process.

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