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Preparation and Anisotropic Elastic Property of Bi-Sr-Ca-Cu-O Whiskers

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Abstract

Flexible and superconducting Bi-Sr-Ca-Cu-O whiskers have been prepared by sintering the melt-quenched glassy powder at 830°C in oxygen atmosphere. X-ray diffraction and a.c. susceptibility measurements reveal that the whiskers belong to 2212 phase with T_c = 79k. The whiskers possess high strength and flexibility along c-axis but were found to be weak and brittle when bent normal to the c-axis. The anisotropic micromechanical elastic property of the whiskers have been demonstrated under a low power optical microscope.

1. Introduction

Since the discovery of Bi-Sr-Ca-Cu-O superconducting whiskers in sintered pellets and powder samples, a fairly large number of reports have appeared in scientific literature during the last decade [1-3]. The high $T_c$ceramic superconductors being rigid and brittle are difficult to bend. This poses serious difficulty for wire applications, which must be overcomed to build superconducting motors, generators, SQUIDs and electromagnets. Moreover, the general tendency of high $T_c$ceramic superconductors to crystallize in the form of porous structures make the problem more tedious. Similarly, since fibers and tapes fabricated by melt-quenched technique [4, 5] are polycrystalline in nature, they contain large number of grain boundaries which not only makes the superconductor structurally weak but also scatter the charge carriers during the supercurrent flow [6]. So, intragrain current density is found to be three orders of magnitude higher than the intergrain current density.

The whiskers, which are filamentary shaped single crystals, have no grain boundaries thus provide remedy for the above mentioned problems. The values of J_c as high as 1.0 x 10^5 A/cm^2 have been achieved for Bi-system whiskers while J_c for the conventional bulk samples do not exceed 2 x 10^4 A/cm^2 [7].

It has also often been presumed that specimens of very small dimensions ought to have a much larger range of elastic strain than their bulk counterparts. It is either because they are free from dislocations or because the few dislocations present cannot be multiply sufficiently to give an observable amount of slip. Thus facilitates to enhance J_c [8].

In this paper we report the superconducting and anisotropic elastic properties of Bi-Sr-Ca-Cu-O whiskers made from the glassy powder precursor.

2. Experimental

Reagent grade Bi_2O_3, $SrCO_3$, $CaCO_3$ and CuO powders (all from Johnson Matthey Chemicals) were mixed in the ratio Bi: Sr: Ca: Cu = 2: 2.1: 1.8: 3. The mixture was thoroughly ground in an agate mortar and pestle and calcined at 800°C for 20 hours in a muffle furnace. This gave final composition of Bi: Sr: Ca: Cu = 2: 2.: 1: 2. The well ground carbonate free powder was then loaded and melted in an alumina crucible at 1100°C for 30 min. The melt was quickly quenched between two stainless steel plates to give 0.5 mm thick black colored glassy sheets. The sheets were then crushed to obtain a fine glassy powder. The powder was once again given a heat treatment in a tube furnace at 830°C for 72 hours. This yielded whiskers with nominal dimensions as length= 0.2-2 mm, width =6-10 μm, thickness=1-4 μm. The whiskers were separated from the reacted polycrystalline powder with the help of a stainless steel mesh susceptibility (ACS) measurements. XRD pattern for such a representative whisker is shown in the Fig. 1. The whisker composition was measured by a JEOL electron probe micro analyzer (EPMA) while surface morphology was investigated by optical and electron microscopy. To study the elastic properties two batches, each consisting on five straight whiskers, were mounted on a quartz plate. One end of each whisker was fixed with epoxy and the open end was bend by a fine stainless steel needle in a well illuminated optical microscope. Resistivity measurements, from room temperature to 77 K, were also made for few long whiskers, but generally XRD and ACS measurements were found sufficient for the purpose of phase identification. One batch of ten fine whiskers was saved for elastic property investigation and the rest were crushed to fine powder for X-ray diffraction (XRD) and a.c.

3. Results and Discussion

Figure 2 shows ACS curves for the Bi-Sr-Ca-Cu-O whiskers and polycrystalline powder. ACS measurements indicated a $T_c$zero at 79 K for the whiskers under discussion. The polycrystalline powder separated from the whiskers had T_c onset at 95 K, but superconducting volume fraction was only 35 % while for the whiskers it is 83% of the total volume. Higher T_c onset value for the polycrystalline powder is due to presence of the minute quantities of 2223 phase. For wire applications high $J_c$is the most desired parameter and it is directly proportional to the superconducting volume fraction of the sample. So whiskers appear as better candidates for such applications.

XRD data elucidated that the whiskers consist mainly 2212 phase, having orthorhombic unit cell with dimensions as : a =5.41 Å, b =5.38 Å, and c =30.0 Å. It further confirmed the whiskers to be single crystals and crystallographically identical. The flat side of the whiskers constitutes the 'ab' plane and the c-axis is perpendicular to the flat side [9]. This is in accordance with the observation that whiskers always position on the flat side and always cleave parallel to the flat side or 'ab' plane [10].

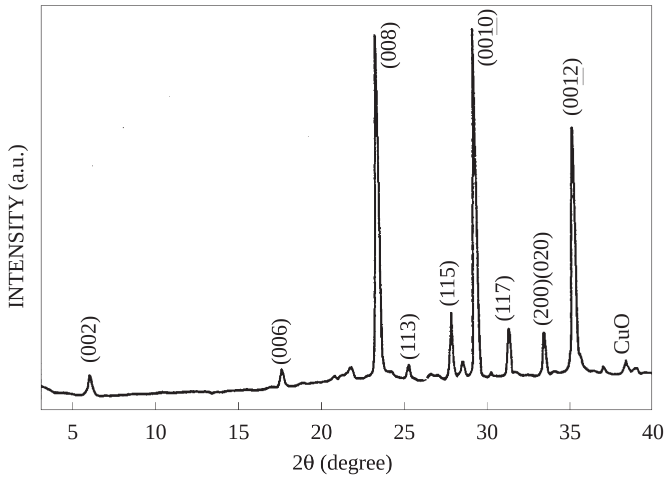

Figure 1. X-ray diffraction pattern of superconducting Bi-Sr-Ca-Cu-O whiskers

Remarkable elastic properties were observed for the Bi-system whiskers. A 1.5 mm long whisker was bent into varying radii of curvature. After each bending experiment the whisker was allowed to relax elasticaly by restoring to its original form. Along the flat side, the whisker was successfully bent up to 130°. The smallest radius of curvature through which whisker could be bent without fracture or plastic deformation was 0.25 mm. Attempts to from sharper bends caused fracture. Occasionally plastic deformation in the form of a permanent kink was also observed. Along a direction normal to the flat surface, which corresponds to the 'ab' plane, the whisker could be bent to a maximum angle of 26°, and any attempt to bend further ended in fracture.

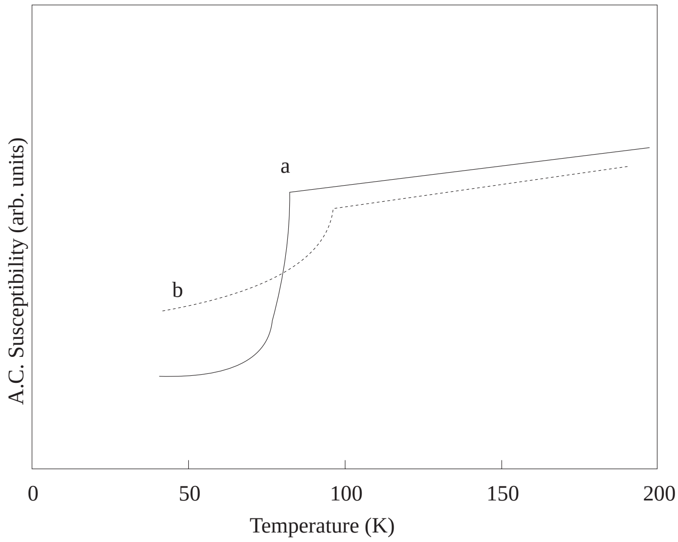

Figure 2. ac susceptibility curves for: (a) Bi-Sr-Ca-Cu-O whiskers and (b) Bi-Sr-Ca-Cu-O Polycrystalline powder.

This anisotropic bending behavior of the whisker is schematically shown in Fig. 3. The strain caused by bending has a maximum positive value on the outer convex side of the whisker while maximum negative value appeared on the inner concave side which is given by $t/2r$, where t is the thickness of the whisker and r is the radius of curvature. Typically the radius of curvature for a whisker bent at 130° was 0.25 mm. The resulted strain comes out to be between 2 and 3.5% . Due to bending stress micro cracks were also seen on the outer convex side of the whisker. Whisker was kept in bending state along the flat side for 1 hour, at 130°. On release it recovered its original shape. In this direction whiskers demonstrate high value of elasticity but along the normal to the flat side a small compression breaks the whisker easily. Micrographs of a whisker before bending and fracture caused after bending are shown in Fig. 4(a) and Fig. 4(b), respectively. The true reason for this anisotropic elastic property is not yet clear and requires further investigation.

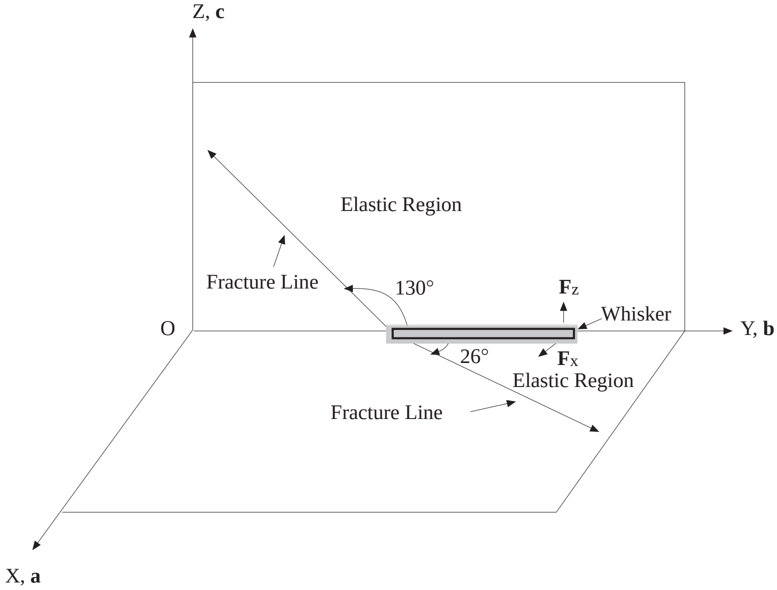

Figure 3. Schematic representation of anisotropic bending property of aBi-Sr-Ca-Cu-O whisker. X, Y, Z represent Cartesian axes while a, b, c denote crystal axes.

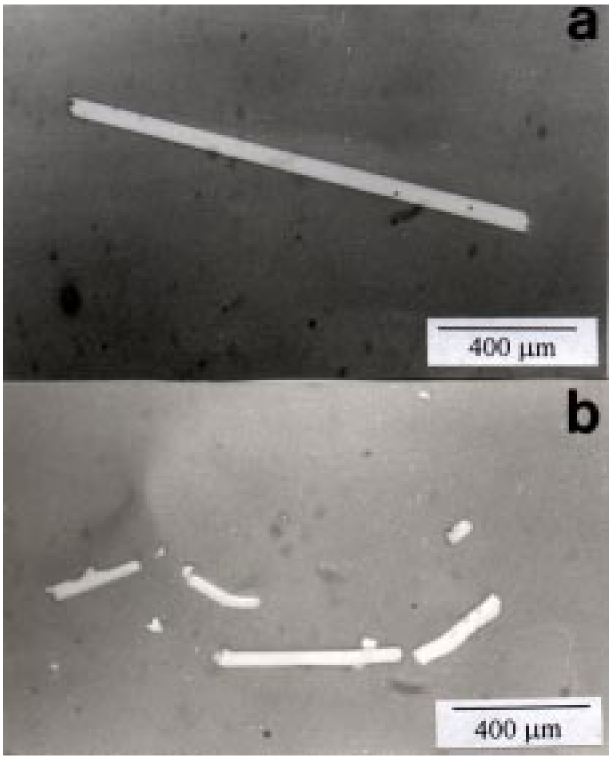

Figure 4. Optical micrographs of , (a) an isolated Bi-Sr-Ca-Cu-O whisker before bending and (b) Fragments of the same whisker fractured after bending.

4. Conclusion

In conclusion, Bi-Sr-Ca-Cu-O superconducting whiskers have been grown from melt-quenched glassy powder. The micro-mechanical bending property has been found to be anisotropic in nature. The whiskers showed high elasticity when bent along the flat side but found to be quite brittle and weak when bent normal to the flat side.

Acknowledgments

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