Pinning Properties of Bi-2212 Single Crystal Whiskers

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Abstract. Bi-2212 single crystal whiskers have been fabricated on the surface of the bulk glass BSCCO pieces prepared by the melt-quenching process. The characterizations of the whiskers were made using XRD, SEM-EDX, M-T, M-H analysis. Pinning force calculations were performed using magnetic hysteretic loops taken at different temperatures and results obtained were analyzed using scaling law equation. The whiskers fabricated showed higher pinning at low temperature. The pinning force at 10 K was calculated to be 3.83x10⁻¹¹ N/m³ under 2 T applied magnetic field. The results obtained showed that a decrease on the flux pinning force decreases the critical current density of whiskers.

1. Introduction

Technological developments lead scientists to the fabrication of HTc materials in single crystal form with micro-nano dimensions which is essential to use in technological devices. Since discovery of high temperature (HTc) superconducting cuprates, many studies have been done on fabrication of the HTc materials in different dimensions by using different preparation techniques. The superconducting properties and technological applications of the fabricated materials have been widely investigated.

The Bi₂Sr₂CaCu₂O₈ (Bi-2212) whiskers have been discovered by Matsubara [1]. The single crystalline nature of the Bi-2212 whiskers has drawn great attention among the researchers who tried to produce the materials in different dimensions by using different preparation methods [2-4]. The fabrication of flexible whiskers without grain boundary and crystal defect is necessary for scientific researches and then integration to the devices [5, 6]. In future, it is believed that superconducting single-crystal whiskers will be most used materials in the technological applications such as micro/nano electronic switches, high current applications and Josephson junction based devices [7,8].

Flux pinning is one of the crucial problems in the development of HTc superconductors of high current density. In 1973, Krammer first suggested a mechanism of flux motion characterized by synchronous shear of the vortex line around flux lines which are too strongly pinned to be broken. The model was further developed owing to new insight into the physics of flux pinning, especially for the HTc superconductor cuprates [9, 10].

Grain boundaries, stacking fault, crystal defects and impurities behave as the primary flux-pinning centers in HTc superconductors. The applied temperature is very important for high current and high pinning force. At high temperature, thermal energy causes melting of vortex lattice while in

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the region of low temperature, \( H_{c1} \) materials generally show strong pinning. It is well known that \( H_{c2} \) materials in small dimensions also exhibit strong pinning. The behavior of the vortex state in Bi-2212 remains still controversial against many theories being purposed to explain experimentally observed results, such as collective pinning, vortex glass, flux lattice melting, flux creep and scaling law. Studies on pinning properties of \( H_{c2} \) materials showed that the scaling law can be easily applied to explain the pinning force \( F_p(H) \). Flux pinning, \( F_p(H) = \mu_0 H J_{c}(H) \), shows a characteristic field dependence proportional to scaling law \( h^{n} (1-h)^{n} \) where \( h \) is the reduced field \( H/H^* \) and \( H^* \) is the normalization field [11-13].

In this study, we fabricated the Bi-2212 whiskers using conventional glass-ceramic technique. Structural and microstructural properties of the whiskers fabricated have been characterized with XRD and SEM-EDX. The pinning properties were investigated by using the scaling law and experimental results obtained were compared with theoretical results.

2. Experimental Procedure

\( \text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_y \) (Bi-2212) whiskers were grown on BSCCO glass plates using the glass-ceramic technique. In this study, the starting composition was selected to be \( \text{Bi}_3\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y \) (Bi-3223). An appropriate amount of high purity (at least 99.9\%) \( \text{Bi}_2\text{O}_3 \), \( \text{SrCO}_3 \) and \( \text{CaCO}_3 \) CuO oxide powders was mixed in an agate mortar for 3–5 h to attain the starting composition. The mixture was melted in an \( \alpha-\text{Al}_2\text{O}_3 \) crucible at 1200\(^\circ\)C for 2-3 h in a high temperature furnace and then molten material were quenched between two cold coppers plates. Thus, rapidly quenched, dark, shiny and approximately 1–3mm thick amorphous materials were obtained. The whiskers were grown on the glass plates in oxygen atmosphere at 850\(^\circ\)C for 120 h.

Structural analysis of both main matrix and the whiskers fabricated were characterized by X-ray diffractometer (XRD). The Rigaku RadB system having CuK\(\alpha_1 \) radiation (\( \lambda = 1.5405 \) Å ) and scan rate of 3°/min were used during XRD analysis. Unit cell parameters were calculated by using XRD data obtained. Surface morphology of the whiskers and the main matrix were carried out by using a Leo Evo40VP scanning electron microscope (SEM).

Magnetization measurements were investigated by using Cryogenic Q-3398 vibrating sample magnetometer (VSM). Superconducting critical transition temperature, \( T_c \), of the whiskers fabricated was determined with M-T experiments. Pinning force calculation was performed by magnetic hysteretic loops taken at different temperatures and directions and the data obtained was fitted to scaling law equation. The results were compared with experimentally and theoretically obtained results.

3. Results and Discussion

X-ray diffraction (XRD) pattern of the whisker and the main matrix is showed in Figure 1. The whisker consisted of pure Bi-2212 phase without impurites. It was found that the whisker was oriented along (00l) direction. The crystal symmetry was obtained to be tetragonal and unit cell parameters to be \( a=b=5.4049 \)Å and \( c=30.6017 \)Å which corresponds to the previously obtained values for Bi-2212 single crystal materials [14,15]. For the main matrix, it was seen that intensity of diffraction peaks decreased significantly, compared to the XRD peaks for the whiskers. Some Bi-2212 peaks were detected, however, large number of impurity phases such as \( \text{Cu}_3\text{Sr}_2\text{O}_5 \), \( \text{Bi}_2\text{Sr}_2\text{Cu}_2\text{O}_6 \) was found in the main matrix. We believe that ionic diffusion from main matrix to whiskers takes place during the whisker formation and this ionic diffusion causes the multiphased main matrix.

Figure 2 shows the surface morphology of whiskers and the main matrix. It was observed that whiskers have grown on the local nucleation centers on the surface of the main matrix and almost perpendicular to surface, Figure 2.a. This suggests that whiskers grow by the time dependent nucleation-growth process. Figure 2.b shows the cross-section of a whisker. The SEM observations
revealed very smooth surface formation with no grain boundaries on the direction of the growth. Any impurity or substructures were not observed, indicating a clear and perfect microstructural formation. EDX analysis showed that composition of the whisker perfectly corresponded to the Bi-2212 phase.

![Figure 1](image1.png)

**Figure 1.** X-ray diffraction of whiskers and main matrixes.

![Figure 2](image2.png)

**Figure 2.** a) Main matrix with whiskers and b) whisker surface.
Temperature dependence of magnetization of both the whisker and the main matrix is shown in Figure 3. A sharp drop in the magnetization curve was obtained, suggesting a good diamagnetic property and single crystal nature of the whisker fabricated. The $T_c$ value for the whisker was found to be 89.4 K. However, the main matrix has a transition indicating intergranular connectivity. The diamagnetic signal increased and the $T_c$ value decreased. It was seen that the impurity concentration influenced the onset $T_c$ of the main matrix. The $T_c$ value for the main matrix was obtained to be 76.8 K, which is attributed to the existence of weakly coupled grains.

Under a fixed magnetic field, when transport current density, $J$, is passed through a HTc superconductor in the mixed state, there is an electromagnetic interaction between the electrons carrying the current and fluxes themselves. A Lorentz force acts on each flux at right angles to both the direction of the flux penetration and the transport current. Two regimes can take place depending on the competition between the Lorentz and pinning forces: flux flow when the Lorentz force is larger than the pinning and/or formation of pinning when the pinning force is stronger than the Lorentz force. The fluxes remain stationary and the sample continues to have zero resistance, as long as there is a pinning force at least equal to the Lorentz force acting to move the fluxes across the sample. However, when the Lorentz force acting on the flux exceeds the pinning force, the fluxes begin to move across the sample. The energy dissipated by the drag or viscous force opposing this motion is supplied by the transport current. Thus, when fluxes are in motion, the material becomes resistive [16]. In HTc superconductors containing various defects such as grain boundaries and impurities, vortices are pinned by the defects.

Change of the pinning force of whiskers and the main matrix with applied field was calculated using $F_p(H) = \mu_0 H J_c(H)$, where $H$ magnetic field and $J_c(H)$ the critical current density calculated by using Bean equation. The obtained maximum $F_p(H)$ values and the corresponding $J_c(H)$ and $H$ values were presented in Table 1. The maximum $F_p(H)$ value was found at 10 K for all the materials. It is obvious that $F_p(H)$ in the ab-axis of the whisker was higher than that of the c-axis. For the main matrix, it was determined that the $F_p(H)$ value was approximately 100 times lower than that of both c-axis and ab-axis of the whisker.

We also fitted to pinning force obtain from M-H curves by using scaling law. According to scaling law, the pinning force was given below equation [17]:

\[ F_p(H) = \mu_0 H J_c(H) \]
where \( h = H / H^* \) and \( H^* \) is normalization field determined by using equation \( J_c(H=H^*)=0 \). \( F, m, n \) are constants determined pinning force. The parameters obtained fitting of experimentally calculated pinning forces are shown in table 2.

\[
f = F \cdot h^m (1-h)^n
\]

(1)

| Materials                  | Temperature (K) | Max. Pinning Force (N/m³) | Magnetic Field (Tesla) | Critical Current Density (A/cm²) |
|----------------------------|-----------------|---------------------------|------------------------|---------------------------------|
| Bi-2212 Whisker (perp. to c-axis) | 10              | 3.13x10¹¹                  | 2.0                    | 0.2x10⁶                         |
| Bi-2212 Whisker (perp. to ab-axis) | 10              | 3.83x10¹¹                  | 5.5                    | 1.3x10⁶                         |
| Main Matrix                | 10              | 1.17x10⁹                   | 1.5                    | 2.7x10³                         |
| Bi-2212 Whisker (perp. to c-axis) | 20              | 2.39x10⁹                   | 0.3                    | 8x10⁴                           |
| Bi-2212 Whisker (perp. to ab-axis) | 20              | 2.73x10¹⁰                  | 1.0                    | 5x10⁵                           |
| Main Matrix                | 20              | 1.40x10⁸                   | 0.3                    | 1.1x10³                         |

Variation of the pinning force calculated by using equation (1) with the magnetic field applied along the c-axis of the whisker is shown in Figure 4.a. The pinning force rapidly increased at low magnetic field values. Maximum pinning force was obtained to be \( 3.13x10^{11} \) N/m³ under 2.0 T at 10 K. The pinning force decreased exponentially with increasing the magnetic field and also temperature. The magnetic field (applied along a-b axis) dependence of the pinning force is shown in Figure 2.b. Maximum pinning force value was found to be \( 3.83x10^{11} \) N/m³ under 5.5 T at 10 K. The \( F_p^{\text{max}} \) in the a-b axis was higher than that of the c-axis, indicating that vortices on the ab-plane were pinned stronger than the c-axis. We believed that this can be due to low dimensionality of a-b axis. Figure 4.c shows the pinning force variation with the applied magnetic field for the main matrix. Maximum pinning force value was found to be \( 1.17x10^9 \) N/m³ under 1.5 T at 10 K. It is clear that the variation of the pinning force with the applied magnetic field for the c-axis of the whisker displayed similar behavior as the main matrix. The pinning force in the main matrix reduces due to high density of impurity and so low critical current density is attained [18]. It was concluded from the results obtained that Bi-2212 single crystal whiskers showed strong pinning force and the impurities in the main matrix decreased the critical current density, \( J_c \), and pinning properites.

The experimental results were fitted to Scaling law and the results are given in Table 2. As a result of the fitting process, the normalization field for the c-axis and the a-b axis of the whisker and main matrix was determined and compared with \( H^* \) values. It was found that the a-b axis shows strongest pinning and highest \( H^* \) value.

| Materials   | Temperature (K) | \( m \)  | \( n \)  | \( F \)  | \( h(f_{p_{\text{max}}}) \) | \( H^* (T) \) |
|-------------|-----------------|---------|---------|---------|-----------------|--------------|
| Bi-2212 Whisker (c-axis) | 10              | 0.850   | 2.93    | 280.1   | 0.25            | 11           |
| Bi-2212       | 10              | 0.700   | 2.39    | 980.1   | 0.79            | 7.1          |
4. Conclusions

In this study, we have fabricated the single-crystal Bi-2212 whiskers and investigated pinning properties of the whisker fabricated. XRD and SEM investigations showed that the whisker fabricated was oriented along the c-axis. The whiskers have grown on the local centers by the nucleation-growth process. Pinning force was calculated using Scaling law. Maximum pinning force value was found to be $3.83 \times 10^{11}$ N/m$^3$ under 5.5 T at 10 K for the ab-axis of the whisker. However, it was seen that the pinning force values decreased to $3.13 \times 10^{11}$ N/m$^3$ under 2.0 T at 10 K and $1.17 \times 10^{9}$ N/m$^3$ under 1.5 T at 10 K for the c-axis and the main matrix, respectively. High pinning force is believed to be due to low anisotropy of the ab-axis. This clearly indicates that vortices were pinned on the ab-plane.

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