## Effect of tensile strain on grain connectivity and flux pinning in $Bi_2Sr_2Ca_2Cu_3O_x$ tapes

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The grain-to-grain connectivity in  $Bi_2Sr_2Ca_2Cu_3O_x$  tapes is still poorly understood, even though they have been commercially available in long lengths for several years. This letter explains the effects of tensile strain on the grain-to-grain connectivity in  $Bi_2Sr_2Ca_2Cu_3O_x$  tapes. The different length scales at which damage to the grain structure occurs are studied with magneto-optical imaging, scanning-electron microscopy, and transport current. These data show that the initial degradation in critical current when strain exceeds the irreversible strain limit is caused by microcracks (~100–500 nm in width) that form mainly at high-angle grain boundaries. Filament-wide cracks (~5–10  $\mu$ m in width) form at locations of lower grain density in the filaments at strains far exceeding the irreversible strain limit. However, in contrast to previous reports, a careful analysis of the pinning force as a function of tensile strain, taking into account current sharing with the normal matrix by using the offset criterion, shows that intragranular flux pinning is not affected by strain in any significant way. © 2006 American Institute of Physics. [DOI: 10.1063/1.2165090]

The rate of improvement in current carrying capabilities of Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> (Bi-2223) tapes has decreased recently. This is partly due to a shift of emphasis to other aspects of the preparation process, including yield and cost, and partly to the complex and still poorly understood grain-to-grain connectivity in Bi-2223 tapes. Focus has shifted to other materials and production techniques in which the effects of granularity are better understood and thus can be more easily overcome.<sup>1</sup> Nevertheless, Bi-2223 and Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>x</sub> (Bi-2212) tapes are currently the only high-temperature superconductors that are commercially available in long lengths, so that they are the materials of choice for a wide variety of applications, even though their critical current density  $(J_c)$  is generally understood to be limited by their grain-to-grain connectivity. Granularity also affects the electromechanical properties of Bi-2223 tapes. It is well known that the critical current  $(I_c)$  of high-temperature superconductors degrades irreversibly due to damage to their grain structure when the applied strain exceeds the irreversible strain limit  $(\varepsilon_{irr})^{2-5}$ Micrographic studies show that this damage occurs on both a submillimeter length scale, observed with magneto-optical imaging (MOI),  $^{6-8}$  as well as on a micrometer length scale, observed in scanning-electron microscopy (SEM) studies.9,10

A number of authors have reported a possible increase in intragranular flux pinning in Bi-2223 tapes due to the formation of micro-cracks under tensile strain.<sup>11–13</sup> In this letter, we argue that microcracks will not act as additional pinning centers. The size of effective pinning centers has to be of the

same order of magnitude as the coherence length of the superconductor (1.5 nm in the *ab* plane for Bi-2223),<sup>1</sup> whereas the initial cracks occur on a micrometer scale; a mismatch of at least three orders in magnitude. Also, intragranular flux pinning is not enhanced when a Bi-2223 tape is ground into a single-grained powder,<sup>14</sup> which, from a mechanical viewpoint, is a much more severe process than the application of tensile strain to a conductor.

This letter also clarifies the different length scales at which strain induced damage to the grain structure occurs in Bi-2223 tapes. The location of micro-cracks responsible for the initial degradation of  $I_c$  and their influence on the electromechanical properties of the tape are determined using SEM and transport current measurements. A careful analysis of the current-voltage curves explains the apparent shift in macroscopic pinning force as function of tensile strain, which has been erroneously interpreted as the source of the additional intragranular flux pinning.

The Bi-2223 tapes are 4-mm-wide multifilamentary tapes, produced by the oxide powder-in-tube method. They consist of either 65 filaments (sample B-1), 55 filaments (sample B-2), or 19 filaments (sample B-3), embedded in a pure silver matrix. Their  $I_c$  at 77 K varies between 20 and 135 A. Although samples from different manufacturers are used, the difference in quality does not influence the results presented in this letter.

Damage to the grain structure on a submillimeter length scale due to tensile strain in sample B-1 is visualized with MOI.<sup>8</sup> The sample was zero-field cooled to  $30\pm1$  K on the cold finger of a helium flow cryostat. An external magnetic field of 25 mT (2% accuracy) was applied perpendicular to the surface of the tape to visualize the spatial variation in critical current density of the filaments. Tensile strain was

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FIG. 1. (a) MO image of part of a filament of tape B-1 before strain is applied. The filament runs horizontally and is partly shielded from the external magnetic field of 25 mT (dark areas). (b) Less dense areas form filament-wide cracks after strain is applied, starting at a strain of ~0.61%. The scale bar is 125  $\mu$ m long.

applied to the superconductor *in situ*, and the change in flux shielding due to the formation of cracks was visualized. Part of the silver matrix is etched away to expose the top layer of filaments, refining the resolution of MOI to approximately 10  $\mu$ m.

The *in situ* formation of microcracks under the influence of tensile strain in sample B-2 was visualized with SEM at room temperature. Part of the silver sheath was etched away also to expose the top layer of filaments.

The electromechanical properties of sample B-3 were measured on a U-shaped bending spring at the boiling point of liquid nitrogen at sea level (77 K).<sup>5</sup> The sample (3 cm in length) was soldered to the brass bending spring. The dependence of the critical current on magnetic field (up to 280 mT applied perpendicular to the tape surface) was measured at each value of the applied tensile strain (accuracy of ±0.005% strain). The critical current was determined within 1% accuracy using both a voltage criterion and an offset criterion<sup>15</sup> at 1  $\mu$ V/cm.

Cracks spanning the width of the Bi-2223 filaments, occurring about 150  $\mu$ m apart, were observed in sample B-1 at an applied strain of 0.6%, which is approximately 1.5 times  $\varepsilon_{irr}$ . The magneto-optical image shows that an applied magnetic field of 25 mT partly penetrates the grain structure even before strain is applied [Fig. 1(a)]. These locations correspond to areas in the filaments that are less dense due to sausaging or pre-existing inhomogeneities caused by intermediate deformation steps in the production process, which form cracks when strain is applied [Fig. 1(b)]. Although the cracks at high strain levels form major barriers to a transport current, they do not explain the initial irreversible degradation in critical current when the applied strain just exceeds  $\varepsilon_{irr}$ .

The damage to the grain structure that is responsible for this initial degradation of  $I_c$  occurs at a micrometer scale, which is far below the resolution of MOI. Microcracks form at strains exceeding  $\varepsilon_{irr}$ , before filament-wide cracks appear (Fig. 2). Here, the formation of cracks as a function of *tensile strain* is observed *in situ* by SEM, as opposed to *bending strain* in previous studies.<sup>10,11</sup> A crack approximately 10  $\mu$ m in length runs roughly horizontally in the center of Fig. 2, perpendicular to the applied strain. The crack runs mainly along grain boundaries but also cuts through grains themselves. A number of smaller cracks can occur as well, for instance in the top left corner of the image.



FIG. 2. SEM image of the grain structure of tape B-2 showing crack formation after tensile strain exceeding  $\varepsilon_{irr}$  is applied *in situ*. The arrows indicate some of the cracks. The exact amount of applied strain is unknown due to the partial etching of the tape surface.

Detailed information regarding the effect of microcracks on the critical current is obtained by studying the dependence of  $I_c$  on applied magnetic field and tensile strain at 77 K. Current at low magnetic field (below  $\sim 60 \text{ mT}$  for sample B-3) is carried by a network of grains that are connected at angles larger than  $\sim 4$  deg, in parallel with a backbone of strongly linked grains that are connected at angles below  $\sim 4 \text{ deg.}^{16,17}$  Current is carried only by the strongly linked backbone at high magnetic fields (above 60 mT). Tensile strain above  $\varepsilon_{irr}$  (~0.5% for sample B-3) affects high-angle grain boundaries more than grains that are connected at low angle as evidenced by a faster decrease in  $I_c$  at low magnetic fields compared to high magnetic fields [Fig. 3(a)]. For example, the critical current at a strain of 0.83% degraded by  $\sim$ 75% in self-field, whereas it only degraded by  $\sim$ 40% at 280 mT. The difference in strain sensitivity is even more apparent from the degradation in *n*-value [Fig. 3(b)]; the degradation in self-field was 65% compared with only 10% at 280 mT. The higher strain sensitivity of high-angle grain



FIG. 3. (a) Normalized critical current of tape B-3 as function of tensile strain at 77 K for different magnetic fields applied perpendicular to the wide side of the tape, showing a greater strain effect at low magnetic fields. (b) Normalized n value as a function of the tensile strain and magnetic field.



FIG. 4. (a) Macroscopic pinning force of tape B-3 as function of magnetic field for different applied strains when the critical current is determined with a voltage criterion of 1  $\mu$ V/cm. The arrow indicates the shift of the maximum pinning force to higher magnetic field as the strain exceeds  $\varepsilon_{irr} = 0.53\%$ . (b)  $F_p$ , when the critical current is determined with an offset criterion at 1  $\mu$ V/cm, shows the absence of any shift when current sharing is corrected.

boundaries is also supported by the SEM image in Fig. 2. High-angle grain boundaries are not only characterized by weaker flux pinning, but are also mechanically weaker than low-angle grain boundaries. This conclusion is supported by a recent report regarding over-pressure sintered Bi-2223 tapes, with nearly fully dense filaments and a highly aligned grain structure, which shows a near-doubling of the irreversible strain limit with respect to their standard processed counterparts.<sup>18</sup>

The question whether tensile strain affects intragranular flux pinning in Bi-2223 tapes is addressed by studying the strain dependence of the macroscopic pinning force  $(J_c)$  $\times B$ ). A clear shift from  $\sim 100$  to  $\sim 150$  mT in the magnetic field  $(B_{\text{peak}})$  at which the macroscopic pinning force is maximum occurs when tensile strain exceeds  $\varepsilon_{irr}$ , with  $I_c$  determined with a 1  $\mu$ V/cm voltage criterion [Fig. 4(a)]. Note that a voltage criterion is an inaccurate determination of the critical current at relatively low currents and low n values, especially when the superconductor is shunted by a large amount of normal conducting material.<sup>15</sup> The offset criterion takes the shunting of current into account, enabling a more accurate determination of the critical current even at low nvalues. The shift in  $B_{\text{peak}}$  as a function of tensile strain does not appear when  $I_c$  is determined with an offset criterion of 1  $\mu$ V/cm [Fig. 4(b)]. Thus, the shift in  $B_{\text{peak}}$  is not caused by an increase in flux pinning, but by current sharing at low currents and low *n* values. These findings also explain the shift in  $B_{\text{peak}}$  reported previously,<sup>11–13</sup> since the data were analyzed with a voltage or electric-field criterion.

In summary, the different length scales at which damage occurs in the grain structure of Bi-2223 tapes as function of tensile strain were investigated with magneto-optical imaging, scanning-electron microscopy, and transport current. The initial degradation in critical current when the applied strain exceeds  $\varepsilon_{irr}$  is due to the formation of microcracks on a micrometer length scale, located mainly at high-angle grain boundaries. Filament-wide cracks form on a submillimeter scale at strains far exceeding  $\varepsilon_{irr}$ . The location of the cracks is correlated with areas where the grain structure is less dense, possibly due to sausaging or other production-related damage. By accurately analyzing the transport data with the offset criterion, it is concluded that intra-granular flux pinning in Bi-2223 tapes is not affected by tensile strain, in contrast to previous studies.

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- <sup>1</sup>D. C. Larbalestier, A. Gurevich, D. M. Feldmann, and A. A. Polyanskii, Nature (London) **414**, 368 (2001).
- <sup>2</sup>J. W. Ekin, D. K. Finnemore, Q. Li, J. Tenbrink, and W. Carter, Appl. Phys. Lett. **61**, 858 (1992).
- <sup>3</sup>R. Passerini, M. Dhallé, E. Giannini, G. Witz, B. Seeber, and R. Flükiger, Physica C **371**, 173 (2002).
- <sup>4</sup>H. Kitaguchi, K. Itoh, H. Kumakura, T. Takeuchi, K. Togano, and H. Wada, IEEE Trans. Appl. Supercond. **11**, 3058 (2001).
- <sup>5</sup>B. ten Haken, H. H. J. ten Kate, and J. ten Brink, IEEE Trans. Appl. Supercond. 5, 1298 (1995).
- <sup>6</sup>M. Polak, J. A. Parrell, A. A. Polyanskii, A. E. Pashitski, and D. C. Larbalestier, Appl. Phys. Lett. **70**, 1034 (1997).
- <sup>7</sup>M. R. Koblischka, T. H. Johansen, and H. Bratsberg, Semicond. Sci. Technol. **10**, 693 (1997).
- <sup>8</sup>D. C. van der Laan, H. J. N. van Eck, B. ten Haken, H. H. J. ten Kate, and J. Schwartz, IEEE Trans. Appl. Supercond. **13**, 3534 (2003).
- <sup>9</sup>R. Passerini, M. Dhallé, B. Seeber, and R. Flükiger, Supercond. Sci. Technol. 15, 1507 (2002).
- <sup>10</sup>M. T. Malachevsky and C. A. D'Ovidio, Supercond. Sci. Technol. 18, 289 (2005).
- <sup>11</sup>Y. K. Huang, B. ten Haken, and H. H. J. ten Kate, IEEE Trans. Appl. Supercond. 9, 2702 (1999).
- <sup>12</sup>J. Horvat, Y. C. Guo, and S. X. Dou, Physica C 297, 10 (1998).
- <sup>13</sup>S. Nishimura, T. Kiss, M. Inoue, M. Ishimaru, M. Kiuchi, M. Takeo, H. Okamoto, T. Matsushita, and K. Ito, Physica C **372–376**, 1001 (2002).
- <sup>14</sup>M. Dhallé, D. C. van der Laan, H. J. N. van Eck, L. Vargas, B. ten Haken, H. H. J. ten Kate, U. P. Trociewitz, and J. Schwartz, IEEE Trans. Appl. Supercond. **13**, 3702 (2003).
- <sup>15</sup>J. W. Ekin, Appl. Phys. Lett. **55**, 905 (1989).
- <sup>16</sup>M. Dhallé, M. Cuthbert, M. D. Johnston, J. Everett, R. Flükiger, S. X. Dou, W. Goldacker, T. Beales, and A. D. Caplin, Supercond. Sci. Technol. 10, 21 (1997).
- <sup>17</sup>D. M. Feldmann, J. L. Reeves, A. A. Polyanskii, G. Kozlowski, R. R. Biggers, R. M. Nekkanti, I. Maartense, M. Tomsic, P. Barnes, C. E. Oberly, T. L. Peterson, S. E. Babcock, and D. C. Larbalestier, Appl. Phys. Lett. **77**, 2906 (2000).
- <sup>18</sup>K. Sato, Proceedings of the 3rd Workshop on Mechano-Electromagnetic Properties of Composite Superconductors, July 2005, Kyoto, Supercond. Sci. Technol. (in press).