# CRITICAL CURRENT DENSITIES IN Bi–2223 SINTER FORGINGS*

B. L. Fisher, K. C. Goretta, N. C. Harris, U. Balachandran
Energy Technology Division
Argonne National Laboratory, Argonne, IL 60439–4838, U.S.A.

Norimitsu Murayama
National Industrial Research Institute of Nagoya
Nagoya 462–8510, Japan



July 1999



Submitted to Proceedings of the 1999 International Cryogenic Materials Conference, Montreal, July 12–16, 1999.

*Work supported by the U.S. Department of Energy (DOE), Energy Efficiency and Renewable Energy, as part of a DOE program to develop electric power technology, and by the Argonne Division of Educational Programs with funding from DOE, under Contract W–31–109–Eng–38; and by the Agency of Industrial Science and Technology, Ministry of International Trade and Industry, Japan.

## DISCLAIMER

## DISCLAIMER

# CRITICAL CURRENT DENSITIES IN Bi–2223 SINTER FORGINGS

B. L. Fisher,[1] K. C. Goretta,[1] N. C. Harris,[1] U. Balachandran,[1]
and Norimitsu Murayama[2]

[1]Energy Technology Division, Argonne National Laboratory,
Argonne, IL 60439–4838, U.S.A.
[2]National Industrial Research Institute of Nagoya, Nagoya 462–8510,
Japan

## ABSTRACT

$(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ (Bi–2223( bars, prepared by sinter forging, exhibited good phase purity and strong textures with the c axes of the Bi–2223 grains parallel to the forging direction. The initial zero–field critical current density ($J_c$) of the bars was $10^3$ A/cm$^2$, but because the forged bars were uncoated, this value decreased with repeated thermal cycling. $J_c$ as a function of applied magnetic field magnitude and direction roughly followed the dependencies exhibited by Ag–sheathed Bi–2223 tapes, but the forged bars were more strongly dependent on field strength and less strongly dependent on field angle.

## INTRODUCTION

Good $Bi_2Sr_2CaCu_2O_x$ (Bi–2212) and $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ (Bi–2223) conductors exhibit excellent phase purity and texture.[1-3] These bulk, Bi–based, high–temperature superconductors have exhibited high transport critical current density ($J_c$). However, $J_c$ values of Bi–2212 and Bi–2223 can decrease substantially in applied magnetic fields that have a component in the c direction of the predominant texture, especially at temperatures approaching 77 K.[2,4]

The microstructures of sinter–forged Bi–2212 and Bi–2223 bars appear to be nearly identical to those of Ag–sheathed tapes.[4-10] $J_c$ values of forged Bi–2223 bars have been reported to reach $10^4$ A/cm$^2$ at 77 K in self field, with critical current ($I_c$) values of up to several hundred amperes. Tapes have achieved transport $J_c$ values of $\approx 10^5$ A/cm$^2$ at 77 K.[11] The difference of nearly one order of magnitude between these two conductors is at least partly related to the higher self fields of the larger, sinter–forged Bi–2223 conductors.

The effect of angle of applied magnetic field on $J_c$ has been quantified and modeled successfully for Ag–sheathed Bi–2223 tapes at 75 K.[2] The goal of this study is to determine whether forged Bi–2223 bars exhibit in–field $J_c$ properties similar to those of the sheathed tapes.

## EXPERIMENTAL PROCEDURES

Powder of nominal composition $Bi_{1.84}Pb_{0.34}Sr_{2.03}Ca_{1.9}Cu_{3.06}O_y$ was prepared by solid–state reaction of $Bi_2O_3$, PbO, $SrCO_3$, $CaCO_3$, and CuO powders. The powders were mixed in ethyl alcohol, dried, and then fired in air and ground repeatedly. The heat–treatment schedule was 750°C for 5 h, 800°C for 5 h, 850°C for 20 h, and 850°C for 50 h.

Compacts, each with a length of 60 mm, a width of 10 mm, and a thickness of 4.2 mm, were prepared by cold uniaxial pressing at 9.8 MPa for 60 s. Each compact was placed between two MgO plates, sinter–forged in air at 850°C and 2.0 MPa for 5 h in the first stage, and then at 860°C and 9.8 MPa for 20 h in the second stage. Temperatures were maintained along each sample to ±2°C; the distance between the sample and a thermocouple was ≈3 mm.

The MgO plates became bonded to the Bi–2223 bars during forging. They were removed by polishing. The thickness of each polished sinter-forged sample was ≈1.4 mm. Four sinter–forged samples were stacked and forged again in air at 860°C and 9.8 MPa for 20 h to produce a final sample thickness of ≈3 mm.

Sample density was obtained from mass and dimension measurements. Phase purity and texture were evaluated by X–ray diffraction, which was conducted parallel with the sinter–forging direction, and by scanning electron microscopy (SEM).[12]

To measure the $I_c$ of the forgings, three 20–A Kepco current amplifiers were placed in parallel to obtain a maximum current of 60 A. The output from the amplifiers was connected to a 1–mΩ resistor and the sample in series. This resistor was used to determine the current passing through the sample. A Hewlett–Packard voltage source was used to pulse the Kepco units (the pulsing unit is capable of current pulse widths of <100 ms). A Hewlett–Packard nanovoltmeter was used to measure the voltage drop of the sample and a Hewlett–Packard multimeter was used to measure the voltage drop across the resistor. All instruments were controlled by a computer through a GPIB interface. LabVIEW software was used for control and data collection.

Current was passed through the sample in both directions, and averages of these two readings were taken to cancel any thermal offset. The sample was placed in a liquid–$N_2$ dewar that was positioned in a uniform magnetic field from an accelerator magnet. $I_c$ values were measured in magnetic fields from 0 to 0.4 T; orientations were 0 to 90° with respect to the forging direction.

Figure 1 is a schematic diagram of the $I_c$ measurement system. Transport current was determined from the voltage drop across a calibrated resistor in series with the Bi–2223 sample. $I_c$ was determined with a standard 1–μV/cm criterion. All measurements were conducted at 77 K. The magnetic field was applied by a large–bore accelerator magnet capable of 0.4 T.

## RESULTS AND DISCUSSION

The density of the forged Bi–2223 samples was 6.1 g/cm$^3$. SEM examinations of fracture surfaces and polished cross sections indicated that the samples consisted of >95% Bi–2223 phase. Individual grains exhibited large aspect ratios and significant texture (Fig. 2). X–ray diffraction confirmed strong texture (Fig. 3), with the c axes of the grains aligned parallel to the forging direction and the a–b planes along the direction of current transport. It has been shown that the texture of good sinter–forged bars is sufficiently strong to produce near–single–crystal elastic properties.[13,14] One expects that such a microstructure should be well suited to high–current applications.

The initial transport $J_c$ values were $10^3$ A/cm$^2$, with $I_c$ values reaching ≈50 A. These values are lower than have been achieved by the best forged Bi–2223 materials.[5,8-10] After a series of tests, in which the sample was repeatedly cooled in liquid $N_2$ and then heated to room temperature, $J_c$ and $I_c$ values dropped significantly. The samples were uncoated and the degradation was attributed to effects of moisture.[15]

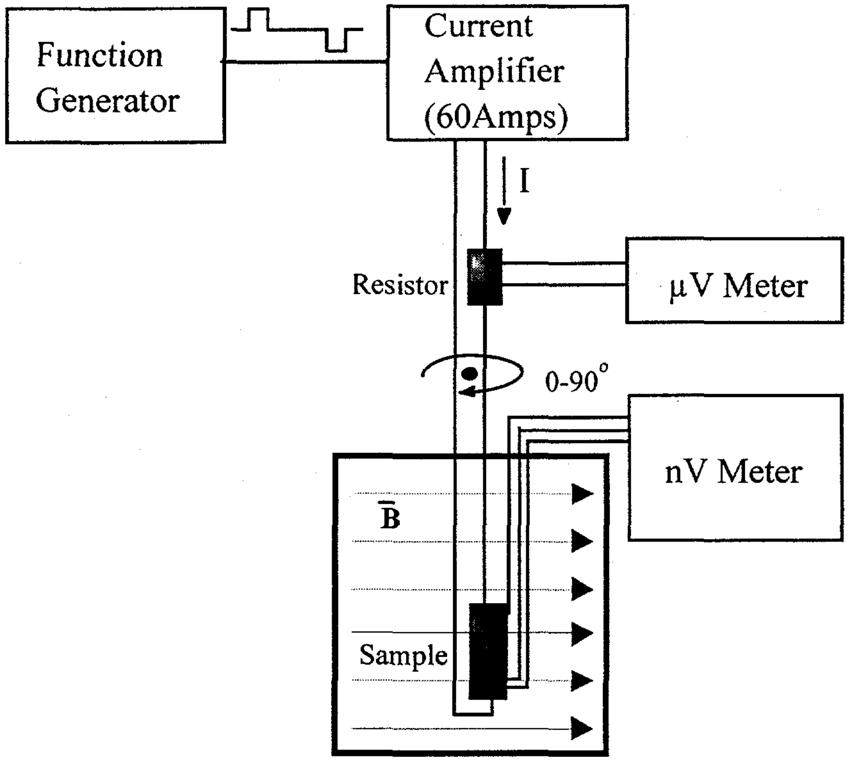


**Figure 1.** Schematic diagram of system used to measure $I_c$ vs. applied field strength and angle.

In–field transport measurements were conducted on single specimens in single campaigns to minimize environmental effects. Results for normalized $I_c$ vs. applied field parallel and perpendicular to the predominant c–axis texture are shown in Fig. 4. For comparison, the data of Willis et al.[2] from Ag–sheathed Bi–2223 tapes are also shown. In general, the $I_c$ values of the forged Bi–2223 decreased more with applied field than did those of the sheathed tape.

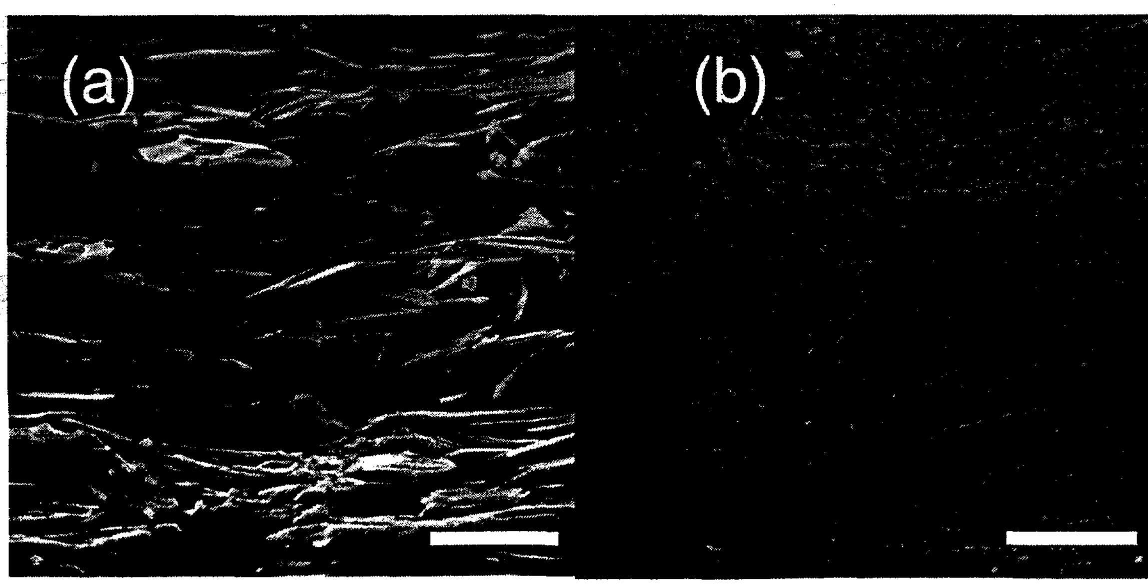


**Figure 2.** SEM photomicrographs of sinter–forged Bi–2223 sample: (a) fracture surface and (b) polished cross section; bars = 10 μm.

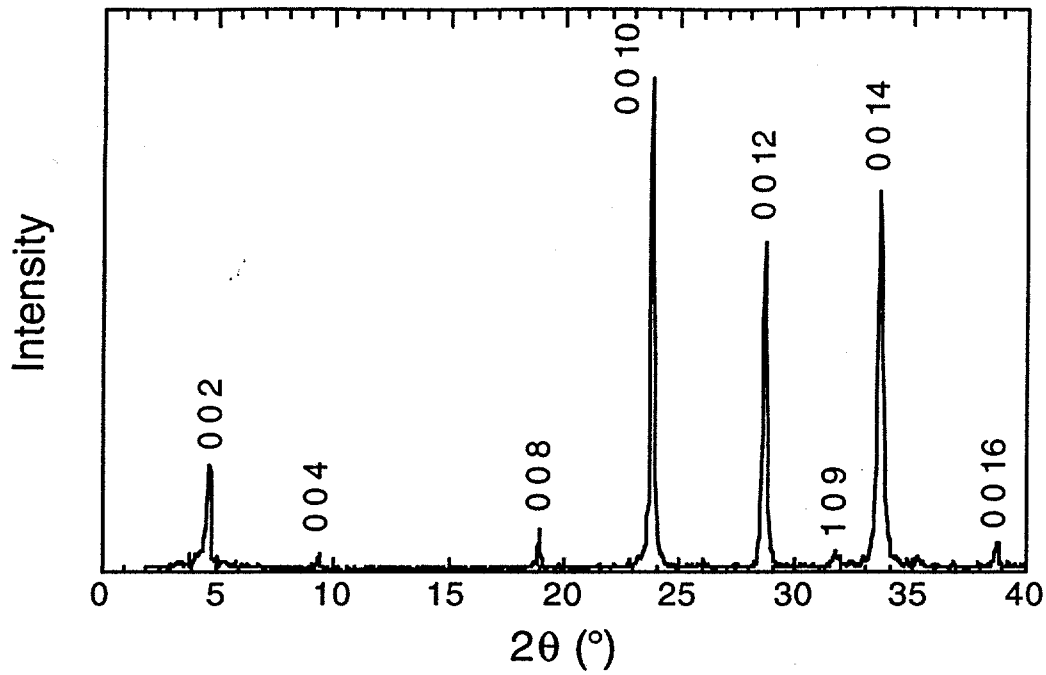


**Figure 3.** X–ray diffraction spectrum from top surface of sinter–forged Bi–2223 sample; as shown by the labeled Bi–2223 peaks, the Bi–2223 c–axis texture is very strong and there is little second phase.

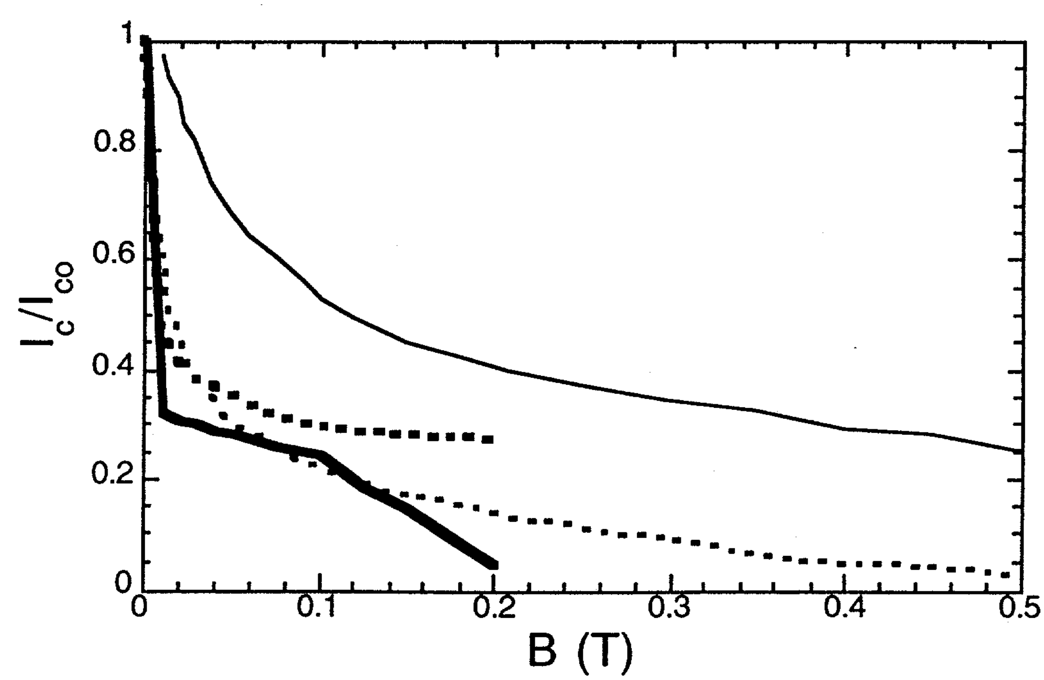


**Figure 4.** Measured $I_c$ vs. initial $I_c$ ($I_{co}$) values for sinter–forged Bi–2223 sample (bold lines) and representative Ag–sheathed Bi–2223 tape (fine lines);[2] solid lines denote field within nominal a–b plane and dashed lines indicate field in nominal c direction.

The effect of angle in various applied magnetic fields was complex (Fig. 5). For Ag–sheathed Bi–2223 tapes, Willis et al.[2] found that a simple cosθ relationship fit their data rather poorly. They constructed a model based on the c–axis component of the applied field and a statistical distribution of grains; it fit their data well.

The data in Fig. 5 exhibited a key difference between the sinter forgings and the sheathed tapes. The forged samples were relatively insensitive to angle of applied field in the range of 0 to ≈25°. The most likely explanation for this is that the textures of the forged samples were not, on average, as strong as those of the sheathed tapes. Theoretical fitting to the data could probably follow the approach of Willis et al.,[2] but effort would be required to quantify the texture through orientation distribution functions or other statistical descriptions.[3,5]

In general, it can be concluded that at 77 K, as expected, applied magnetic fields with large components in the nominal c direction of sinter–forged Bi–2223 samples caused $I_c$ to decrease significantly. Even fields as low as 100 G reduced $I_c$ by ≈30–35%. The forged bars were less sensitive to angle of applied field than were Ag–sheathed tapes, probably because of weaker crystallographic texture.

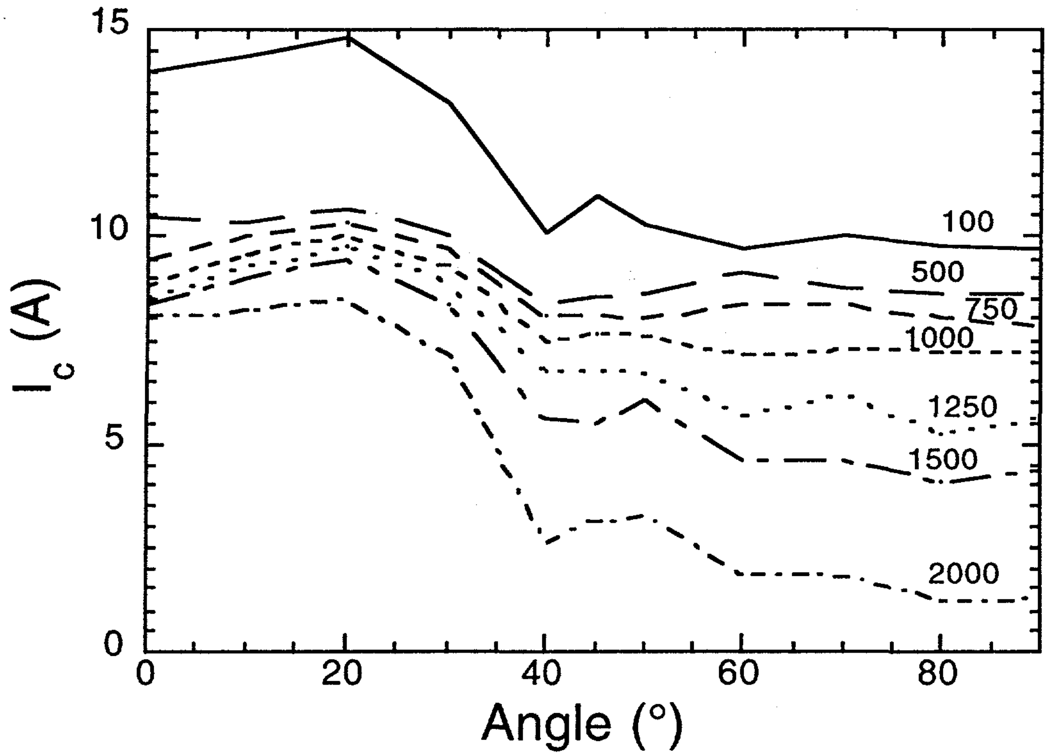


**Figure 5.** Measured $I_c$ vs. angle of applied field for sinter–forged Bi–2223 sample; field magnitude values in gauss are shown and 0° corresponds to applied field within the nominal a–b plane.

## CONCLUSION

Sinter–forged $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ bars exhibited high density, good phase purity, and strong textures with the c axes parallel to the forging direction. The initial zero–field critical current density of the forged bars was $10^3$ A/cm$^2$. Decreases $I_c$ values with applied magnetic field magnitude were greater in the forged bars than in Ag–sheathed tapes, but $I_c$ of the forged bars was less sensitive than the sheathed tapes to the angle of applied field.

## ACKNOWLEDGMENTS

We thank Professor M. T. Lanagan for helpful discussions. This work was supported by the U.S. Department of Energy (DOE), Energy Efficiency and Renewable Energy, as part of a DOE program to develop electric power technology, and by the Argonne Division of Educational Programs with funding from DOE, under Contract W–31–109–Eng–38; and by the Agency of Industrial Science and Technology, Ministry of International Trade and Industry, Japan.

## REFERENCES

1. E. E. Hellstrom, Important considerations for processing Bi–based high–temperature superconducting tapes and films for bulk applications, *MRS Bull.* 17[8]:45 (1992).
2. J. O. Willis, J. Y. Coulter, E. J. Peterson, G. F. Chen, L. L. Daemen, L. N. Bulaevskii, M. P. Maley, G. N. Riley, W. L. Carter, S. E. Dorris, M. T. Lanagan, and B. C. Prorok, Dependence of the angular anisotropy of the critical current density on texture in silver-sheathed Bi-2223 tapes, *Adv. Cryo. Eng.* 40:9 (1994).
3. K. C. Goretta, N. Chen, A. C. Biondo, B. L. Fisher, M. T. Lanagan, and J. S. Kallend, Fabrication and texture characterization of bulk $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ and $Bi_2Sr_2CaCu_2O_x$ superconductors, *Adv. Cryo. Eng.* 42:753 (1996).
4. S. X. Dou and H. K. Liu, Ag–sheathed Bi(Pb)SrCaCuO superconducting tapes, *Supercond. Sci. Technol.* 6:297 (1993).
5. N. Chen, A. C. Biondo, S. E. Dorris, K. C. Goretta, M. T. Lanagan, C. A. Youngdahl, and R. B. Poeppel, Sinter-forged $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ superconductors, *Supercond. Sci. Technol.* 6:674 (1993).
6. K. C. Goretta, L. J. Martin, D. Singh R. B. Poeppel, N. Chen, C.-Y. Chu, J. L. Routbort, and R. A. Gleixner, Mechanical properties of fiber–reinforced $Bi_2Sr_2CaCu_2O_x$ and $YBa_2Cu_3O_x$ bars, *Adv. Cryo. Eng.* 40:63 (1994).
7. J. Guo, J. Schwartz, Y. S. Cha, C.-T. Wu, and K. C. Goretta, Effects of hot rolling on powder–in–tube BSCCO tapes, *Adv. Cryo. Eng.* 40:169 (1994).
8. N. Murayama and J. B. Vander Sande, Hot forging with heat treatment of Bi-Pb-Sr-Ca-Cu-O superconductors, *Physica C* 241: 235 (1995).
9. A. Tampieri, G. Calestani, G. Celotti, R. Masina, and S. Lesca, Multi-step process to prepare bulk BSCCO (2223) superconductor with improved transport properties, *Physica C* 306:21 (1998).
10. N. Murayama and W. Shin, Decomposition of (Bi,Pb)-2223 phase during sinter forging, *Physica C* 312:255 (1999).
11. M. Lelovic, P. Krishnaraj, N. G. Eror, A. N. Iyer, and U. Balachandran, Transport critical current density above $10^5$ A $cm^{-2}$ at 77 K in $Bi_{1.8}Pb_{0.4}Sr_{2.0}Ca_{2.2}Cu_{3.0}O_y$ superconducting tapes made by the Ag wire-in-tube method, *Supercond. Sci. Technol.* 9:201 (1996).
12. J.-H. Park, K. C. Goretta, and N. Murayama, Chemical diffusion of oxygen in dense, textured $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$, *Physica C* 312:269 (1999).
13. Chang Fanggao, P. J. Ford, G. A. Saunders, Li Jiaquing, D. P. Almond, B. Chapman, M. Cankurtaran, R. B. Poeppel, and K. C. Goretta, Anisotropic elastic and non–linear acoustic properties of very dense textured $Bi_2Sr_2CaCu_2O_x$, *Supercond. Sci. Technol.* 6:484 (1993).
14. K. C. Goretta, D. S. Kupperman, S. Majumdar, M. W. Such, and N. Murayama, Elastic constants of dense, textured $(Bi,Pb)_2Sr_2Ca_2Cu_3O_y$, *Supercond. Sci. Technol.* 11:1409 (1998).
15. A. Barkatt, H. Hojaji, V. R. W. Amarakoon, and J. G. Fagan, Environmental stability of high $T_c$ superconducting ceramics, *MRS. Bull.* 18[9]:45 (1993).