Intragrain pinning strength depth dependence of 2223 (Bi,Pb)-based high
critical $T_c$ superconducting ceramics made by a vitreous route

An Dang, P. A. Godelaine, and Ph. Vanderbemden
SUPRAS, Institut d’Electricité Montefiore, B28, Université de Liège, B4000 Liège, Belgium

R. Cloots
SUPRAS, Institut d’Electricité Montefiore, B28, and Institut de Chimie B6, Université de Liège,
B4000 Liège, Belgium

M. Ausloos$^a$
SUPRAS, Institut de Physique B5, Université de Liège, B4000 Liège, Belgium

(Received 13 September 1994; accepted for publication 20 December 1994)

Campbell’s method for measuring the critical current in superconductors has been used to obtain the
critical current density and the pinning strength in Bi$_{1.7}$Pb$_{0.2}$Sr$_2$Ca$_2$Cu$_3$O$_{10-\gamma}$ ceramics synthesized
by a vitreous route. The intragrain critical current is much higher than $10^9$ A/cm$^2$ at 40 K in zero dc
magnetic field. A large increase of the pinning strength is observed near the grain surface. The
decrease with depth is hyperbolic. The role of the precursors in the synthesis route is emphasized for
introducing specific pinning centers. The analysis takes into consideration the ceramics granular
nature, i.e., the existence of intergrain and intragrain currents. © 1995 American Institute of
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It is of great importance to increase the critical current of
bulk 2223-based BiSrCaCuO ceramics (BSCCO) for many
practical applications. The pinning strength of such BSCCO
is known to be relatively low due to the lack of appropriate
pinning centers in the bulk of the materials in contrast to
point or line defects in YBCO ceramics. We have decided to
use some material prepared by the so-called “multicompo-
nent powder glass route.”$^1-5$ This allows us to have some
control on the geometry and also of the chemical nature on
pinning centers, and on their distribution in the material.

We have measured the internal pinning strength inside
the grains by applying a modified Campbell’s method,$^6$ first
obtaining the so-called flux profile$^7$ and then the critical
current density. The effect of an external dc field has been in-
vestigated. The pinning strength is seen to decrease strictly
hyperbolically in the grains. The role of surface precipitates
is thus underlined. This points toward interesting possibilities
for optimizing the pinning mechanism in such BSCCO sys-
tems. The obtained values are close to record high.

The vitreous route has already been used by our group in
order to synthesize various 2223 Bi-based superconducting
ceramics.$^1-5$ Other authors have also used the same kind of
procedure. A large (nonexhaustive) list of references can be
found in Ref. 5.

The process involves a so-called “crystalline precursor
matrix” method based on the synthesis of separately
quenched intermediate phases in a two-powder process. This
procedure corresponded to “route 2” in Ref. 5.

The x-ray diffraction pattern of the resulting 2223 mate-
rial showed a $\sim 10\% - 90\%$ mixture of 2212 and 2223
phases. However, the 2223 phase was the main phase in the
system.$^5$ The electrical resistivity curve$^5$ as a function of
temperature showed a percolation path at 108 K, a residual
resistivity of $\sim 4 \mu \Omega \cdot m$, and a linear resistivity coefficient of
0.1 $\mu \Omega \cdot m/K$.

Microstructural characterizations were performed on a
Hitachi S2500 scanning electron microscope. The micro-
graphs showed that the materials can be considered as a mul-
titude of needle-like 2223 crystals of regular dimensions.
(One should note that the 2212 phase only is present is the
single phase SrCaCu$_3$O$_5$ crystalline precursor is prepared at
995 °C$^5$). Close observation showed the presence of micro-
scopic impurities incorporated in the main phase (indicated
by a cross in Fig. 1). They are thought to be the 2212 inter-
grown phase in 2223 grains. Electron dispersive x-ray analy-
ysis can precisely determine the previous observations. It was
found that precipitates at grain boundaries and in grains are
Sr-, Ca-, and Cu-rich phases.$^5$

In Ref. 5 we have reported the electrical, thermal, and
thermoelectric properties of such systems. We concentrate
here on the $J_c$ and pinning strength values, their depth depen-
dence and the related mechanism.

Campbell’s method$^6$ consists in measuring the flux pen-
netration (so-called “flux profile”) for a cylindrical sample
inserted in a magnetic field composed of a dc component and
a small alternating superimposed signal.

The penetration depth $\rho$ is defined as the difference be-
tween the radius $R$ of the sample and the position reached by
the magnetic flux in the sample. This penetration depth is
easily obtained by considering the flux modification as a
function of the magnitude of the ac field. We obtain the so-
called Campbell’s formula$^6$:

\[
(p/R) = 1 - \left[ 1 - (dS_{\text{supra}}/dh_{\text{ac}})/(2\pi\mu_0R^2) \right]^{1/2},
\]

where $S_{\text{supra}}$ is the signal measured by a small coil surround-
ing (wound on) the sample and $h_{\text{ac}}$ the magnitude of the ac
applied field. The inverse graph $h_{\text{ac}}(p)$ is known as the “flux
profile.”

$^a$Corresponding author; E-mail: U2150MA@BLULG11.BITNET

0021-8979/95/77(7)/3560/3/$36.00 © 1995 American Institute of Physics
In order to take into account the granular nature of the ceramics, we have extended the above model. We have shown that the flux profile has a knee structure from which the critical current $J_{c_k}$ can be found in the grains of mean radius $R_g$, as

$$J_{c_k} = (d h_{sc}/dp)_{p=p^*}(R/R_g)(1-p^*/R),$$

(2)

where $p^*$ is the intersection of the straight line fitted to the high $h_{ac}$ data with the $h_{sc}=0$ axis. The value of $p^*$ gives the order of magnitude of the penetration depth averaged over the grains. In order to do so the critical state model is used. One should also point out a recent article where the critical state model has been applied to granular superconductors. The analysis is however limited to describe the susceptibility without extracting quantitative data.

The critical current $J_{c_j}$ in the weak links can also be found from

$$J_{c_j}^{(0)}(d h_{sc}/dp)_{p=0}(1/R)(p^*/R)[2-(p^*/R)],$$

(3)

i.e., from the data lowest value range. Furthermore, the grain superconducting fraction $f_g$ can be obtained from

$$f_g = (1-(p^*/R))^2.$$  

In so doing the flux profile inside the grain can be extracted from the global flux profile. In other words, we can measure the penetration of the magnetic flux on the average inside the grains. The critical current of the grains as a function of the distance $p_g$ inside the grain,

$$p_g R_g = 1-[(1-p^*/R)/(1-p^*/R)],$$

can thus be obtained.

In order to obtain these critical currents we need a large number of points since the derivative should be computed. We have used a multiple profile acquisition scheme with five runs of twenty points and a shift in the initial value of the ac field in order to obtain a set of one hundred data points.

The flux profile for the Bi$_2$2Pb$_2$Sr$_2$Ca$_2$Cu$_3$O$_{10-y}$ sample prepared by the vitreous route was taken at 77 and 40 K in view of the superconductivity transition and of our crystal limitations. For this measure the dc field was perpendicular to the ac field; the latter is maintained along the axis of the sample. The value of $h_{ac}$ is at most 10 G. The 40 K results are shown in Fig. 2. Four flux profiles were taken for dc fields ranging between 0 and 0.3 T. The knee structure mentioned above is well visible in this graph. The slope in the low-field region is proportional to the critical current $J_{c_k}$ inside the grains.

In Fig. 3 the calculated critical currents in the weak links and in the grains are shown for both 77 and 40 K temperatures. The weak link critical current is greater than 600 A/cm$^2$ at low dc field and larger than 100 A/cm$^2$ at relatively large dc field at 40 K. The grain critical current is in the record high range for bulk materials (see values quoted for films in Refs. 9 and 10) between $10^4$ and $5 \times 10^4$ A/cm$^2$ at 77 K.

![FIG. 3. Calculated critical currents in the weak links and the grains at 40 K and in the grains at 77 K. In all curves, an important increase of the intra-grain critical current is seen at low dc field.](image-url)
K and B = 0. In all curves, a large increase of the intragrain critical current is well marked at low dc field.

We also made other flux profile acquisition runs but with high ac fields up to 30 G. Furthermore, a large dc field was applied in the perpendicular direction. Obviously, the higher the dc field, more deeply the ac field influence is marked inside the grains. Figure 4 shows these results. A marked curvature of the flux profile in the grain response can be observed after the knee. We argue that such a feature is due to a reinforcement of the pinning mechanism near the surface of the grains, and confirm the importance of local micro and macrostructure 10 for improving pinning strength values. In order to do so, we subtract the weak link region and analyze the data. Since the derivative of the flux profile curves is proportional to the critical current $J_{c_{\parallel}}$ and if we identify $J_{c_{\parallel}}$ with the pinning strength $P_v$, by $P_v = J_{c_{\parallel}} B$, where $B$ is the dc flux density, we obtain the data of Fig. 5 which allows us some information on the depth dependence of $J_c$ and of the pinning strength. It is seen that $P_v$ increases when the dc field increases, as expected. More interestingly the very large rise of $P_v$ near the grain surfaces should be noticed. We argue that the increase in pinning strength only arises from the presence of SrCaCuO$_2$ precipitates near the grain surface. These particles may thus act directly or indirectly as pinning centers.

The inverse of the pinning strength $P_v$ vs $P_g$ is almost a straight line given by $1/P_v = \kappa P_g + \psi$. The values of $\kappa$ and $\psi$ decrease when the dc field increases. In Fig. 5 we have shown the resulting data fit with these $\kappa$ and $\psi$ parameters. The pinning strength near the grain surface reaches a value $\sim 2.66 \times 10^{9}$ N/m$^3$ at 0.04 T.

We have thus demonstrated that a reinforcement of the pinning strength near the surface of grains is observed. This is likely due to the presence of a SrCaCuO$_2$ crystalline phase near the grain boundaries.

Part of this work has been financially supported through the Belgium Federal Services for Scientific, Technical and Cultural (STSC) Affairs under contract SU02/013. R.C. is a research fellow of the FNRS (Brussels). We thank Professor H. W. Vanderschueren for allowing us to use the Measurement and Instrumentation in Electronics Laboratory (MIEL).

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