Improvement of pinning in Bi2212-based materials by hot plastic deformation

R R Daminov1,2, M Reissner1, M F Imayev2, W Steiner1, M V Makarova3 and P E Kazin3

1Institute of Solid State Physics, Vienna University of Technology, A-1040 Wien, Austria
2Institute for Metals Superplasticity Problems, Russian Academy of Sciences, Ufa, 450001, Russia
3Chemistry Department, Moscow State University, Moscow, 119992, Russia

E-mail: reissner@ifp.tuwien.ac.at

Abstract. Hot plastic deformation of bulk Bi2212 strongly improves pinning by introducing strong pinning centres accompanied by the formation of a high degree of texture. This is proved by an increase in critical current density and mean effective activation energy obtained from relaxation measurements, as well as from a large shift of the irreversibility line. The analysis shows that a simple interpretation of the $U(J)$-relation in terms of collective creep theory is not possible. Admixture of MgO particles gives similar, but less good results.

1. Introduction

Although the high transition temperature of high temperature superconductors (HTS) has set great hopes for new applications, the use of bulk material of HTS is often still limited by too low critical current densities. To improve high current carrying probabilities both a strengthening of the pinning and, because of the large anisotropy of these materials, the production of high texture is necessary. With hot plastic deformation a new path of preparation was trod, which can fulfil these requirements [1]. In a specially constructed equipment samples can be deformed simultaneously by uniaxial pressure and torsion under high temperatures, thus producing a well defined defect structure with high texture [1]. The mechanism responsible for the high degree of basal plane texture in YBa$_2$Cu$_3$O$_x$ could be identified: rotation and stacking of plate-like grains in liquid grain-boundary films [1, 2]. Investigations of the influence of the deformation temperature on the superconducting properties of Bi$_2$Sr$_2$CaCu$_2$O$_{8+x}$ (Bi2212) samples have shown that basal texture higher than 90% can be achieved and critical current densities $J_c$ (at 30 K and 1 T) about 15 times higher than in the undeformed sample are possible [3]. One benefit of the hot plastic deformation method by torsion under pressure is the high strain, due to torsion, which favors high density of dislocations and formation of low angle grain boundaries extended over large areas. Details about the microstructural changes during deformation will be described in a future paper [4].

In two preliminary investigations [3, 5] we have shown that highest $J_c$ values and highest degree of texture are obtained for deformation temperatures between 875°C and 905°C. In this paper we present
results of a more detailed investigation of the pinning properties on two samples prepared at $T_d = 895\,^\circ\text{C}$, one with (DM895) and one without (D895) admixture of MgO particles, which might act as additional pinning centres. The results are compared with those obtained for an undeformed sample (S855).

2. Experimental
The samples were chosen from two series prepared from commercial Bi2212 powder from Hoechst, one without (series D) and one with (series DM) admixture of 3.45 wt% MgO particles (approximately 20 nm mean diameter). The powders were pressed into pellets of 10 mm diameter and 2 mm height and sintered in air at 855$^\circ\text{C}$ for 24 h, followed by hot plastic deformation. The deformation was performed by both uniaxial pressure, and simultaneous torsion caused by rotation of one of the anvils around the compression axis. During the deformation process the apparatus was held at a fixed temperature ($T_d$). At the end the samples were annealed in air at 850$^\circ\text{C}$ for 96 h. In an extended study the relevant parameters, deformation temperature $T_d$ (795$^\circ\text{C}$ to 940$^\circ\text{C}$), loading pressure $p$ (5 to 45 MPa), twist angle $\alpha$ (45 to 90$^\circ$), and rotation speed $v$ (1.5 $\times$ 10$^{-3}$ to 3.0 $\times$ 10$^{-3}$ rpm) were systematically changed to find the optimal conditions leading to highest texture in combination with best superconducting properties with regard to pinning [4].

The microstructure of the samples was checked by SEM and X-ray diffraction. The superconducting properties were investigated by magnetic measurements in a vibrating sample magnetometer between 2.5 K and $T_c$ in fields up to 7 T parallel to the compression axis. From hysteresis measurements the critical current density $J_c$ was calculated by the Bean formula [6] assuming that the mean grain size $d$, determined as the average length of cuts through the grains at arbitrary positions perpendicular to the field direction, is the characteristic length for the supercurrent flow. In addition, the time dependence of the magnetic moment was measured at 1, 3, and 5 T in the whole temperature range for up to 1 h, starting both from the field increasing and decreasing branch of the hysteresis loop. These relaxation measurements were analyzed within the Anderson-Kim model [7] of flux creep to get mean effective activation energies. Additionally the energy-current relation was determined after Maley et al [8].

3. Result
The improvement by hot plastic deformation is clearly seen in the temperature dependence of the critical current density (figure 1). At 4.2 K and 1.5 T $J_c$ is 245% higher for sample D895 than for the undeformed sample S855. This difference increases to 355% at 5.5 T. Sample DM895 shows higher

![Figure 1](image-url)
current density too, but the improvement at 4.2 K is only 28% also slightly increasing with applied field. Especially at higher temperatures and fields the improvement becomes best. This is indicated by the hump which appears in the temperature dependence of \( J_c \) for both deformed samples, which is best seen in the normalized representation (inset figure 1). Accompanied is this behaviour by a strong shift of the irreversibility line (IL) to higher temperatures. In figure 2 irreversibility lines are shown for sample S855 and D895 determined for different current criteria. For a maximal current density of \( 1 \cdot 10^6 \text{ A/cm}^2 \) the irreversibility temperature \( T_{irr} \) for 4 T is shifted from 13 to 25 K. The shift is even higher for larger current densities; e.g. for \( 5 \cdot 10^6 \text{ A/cm}^2 \) \( T_{irr} \) shifts from 3.7 to 21 K for 4 T. The lines are nearly coincident for both samples, if a current criterion of \( 5 \cdot 10^6 \text{ A/cm}^2 \) and \( 2 \cdot 10^7 \text{ A/cm}^2 \) is chosen for S855 and D895, respectively.

According to Anderson and Kim [7] mean effective activation energies \( <E> \) are calculated from the relaxation curves by \( <E> = kT[lS + \ln(\pi\tau_0)]\), with \( S = M(t_0)^{-1}(dM/d\ln t) \) the normalized creep rate, \( t_0 \) the starting time of the relaxation, and \( \tau \) the intrinsic relaxation time, which was assumed to vary between \( 10^{-6} \) and \( 10^{-12} \) s. Within measuring accuracy there is no significant difference between \( <E> \) determined in increasing and decreasing field, indicating that the relaxation is mainly due to bulk pinning. Therefore in figure 3 only the mean values between both measurements are shown versus temperature for the relaxations at 1 T. Up to 17 K a strong increase in \( <E> \) with temperature is observed. This increase is caused by the fact that \( <E> \) is the value of the centre of gravity of a probable existing activation energy distribution, caused by a distribution of pinning energies in the sample. By increasing the temperature, more and more of the low energy part of this distribution is not seen in the time dependence, because relaxation over the low energy barriers is finished before the relaxation measurement is started. Hence, the mean of the distribution \( <E> \) has to increase with \( T \). The increase in slope and the much higher absolute values of \( <E> \) for the deformed samples indicate that the activation energy distributions are shifted to much higher energies. This leads to the conclusion that hot plastic deformation introduces pinning centres (mainly intragranular dislocations and low angle grain boundaries) into the material which are much stronger than those in the undeformed sample.

To gain some information about the character of the pinning from the relaxation data also the energy-current relation \( U(J) \) was determined. According to Maley et al [8] \( U = -kT[\ln(dM/dt) - \ln(HQX/2\pi d)] \), with \( H \) the applied field, \( \nu \) the attempt frequency, \( X \) the hopping distance and \( d \) the sample thickness. Assuming that the second logarithm is in first approximation a constant \( C \), \( U \) can be directly determined from the relaxation data. Choosing an appropriate value for \( C \), and taking into

**Figure 2.** Irreversibility lines for sample S855 (open symbols) and D895 (full symbols) determined for different current criteria.

**Figure 3.** Temperature dependence of mean effective activation energy at 1 T. Lines are only guides to the eye.
account a temperature correction $g(T) = 1 - (T/T_c)^2$ (as proposed by Tinkham [9]), the $U$ values for the different temperatures can be plotted versus irreversible magnetization $M_{irr}$, according to Bean [6]. $M_{irr}$ is proportional to $J_c$, leading finally to the searched $U(J)$-relation. Figure 4 shows the result for 1 T. Whereas for S855 and DM895 a $C$ value of 20 is found for D895 a smooth curve is only found if $C = 20$ is used for the temperatures up to 11 K. For higher temperatures $C = 40$ is obtained. In this temperature range the $U(J)$-relation shows a strange behaviour. Instead of the usually concave curvature as found for sample S855, a convex curvature is present between 11 and 26 K. This behaviour exists also in case of sample DM895, but much less pronounced. The change in curvature indicates that a simple interpretation of the $U(J)$-relation is not possible in terms of the collective creep theory, which discusses pinning of weak, randomly distributed pinning centres, and which well describes pinning in standard Bi2212 samples as pinning of 2D pancakes.

**References**

[1] Imayev M F, Kabirova D B, Korshunova A N, Zagitov A S, Val’kovsky S N and Kaibyshev O A 1999 Proc. Fourth Int. Conf. On Recrystallization and Related Phenomena ed T Sakai and H G Suzuki (The Japan Institute of Metals) p 899

[2] Imayev M F, Kaibyshev O A, Musin F F, Yamalova M 1993 Mater. Sci. Forum 113-115 585

[3] Daminov R R, Imayev M F, Reissner M, Steiner W, Makarova M V and Kazin P E 2004 Physica C 408-410 46

[4] Imayev M F, Daminov R R, Reissner M, Steiner W, Makarova M V and Kazin P E to be published

[5] Reissner M, Daminov R R, Imayev M F, Steiner W, Makarova M V and Kazin P E 2003 Proc. 6th European Conf. On Applied Superconductivity ed A Anderson, C P Pepe, D Christiano and G Masullo IOP Conf. Ser. Nr. 181 p 2195

[6] Bean C P 1962 Phys. Rev. Lett. 8 250

[7] Anderson P W, Kim Y B 1964 Rev. Mod. Phys. 36 39

[8] Maley M P, Willis J O, Lessure H and McHenry M E 1990 Phys. Rev. B 42 2639

[9] Tinkham M 1988 Phys. Rev. Lett. 61 1658