

## **Bulk textured Bi2212/MgO with high critical current densities at low temperatures**

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**ABSTRACT:** Bulk Bi2212 superconductors containing MgO are textured by solidification in a high magnetic field (MMP) followed or not by hot forging (HF). The texture degree is studied by neutron diffraction. The combination of MMP and HF yields highly textured bulk materials: the c-axis alignment is of 16.3 m.r.d. Transport  $J_c$  of 11 kA/cm<sup>2</sup> is reached at 40 K. At 4 K,  $J_c$  values higher than 160 kA/cm<sup>2</sup> are magnetically measured with a record value of 165 kA/cm<sup>2</sup>.

### **1. INTRODUCTION**

To reach high critical current density in bulk BiSrCaCuO superconductors, the weak link problem has to be overcome. Texturing processes have thus been developed on Bi2212 compounds: the floating zone method (Kubo 1989 and Angurel 1997), the directional isothermal growth (Holesinger 1993), the melt-casting process (Bock 1989) or the composite reaction texturing (Soylu 1992). Magnetic melt processing (Noudem 1996) as well as sinter forging (Noudem 1994, Rouessac 1998) have proved to be efficient in texturing Bi2223 compounds. In this paper, Bi2212 bulk superconductor containing MgO is textured by solidification in a high magnetic field followed or not by hot forging. Textures of the bulk materials are studied using neutron diffraction. Critical current densities ( $J_c$ ) are obtained by transport measurements for temperatures between 77 and 40 K. Magnetic measurements of  $J_c$  are also performed at 4 K.

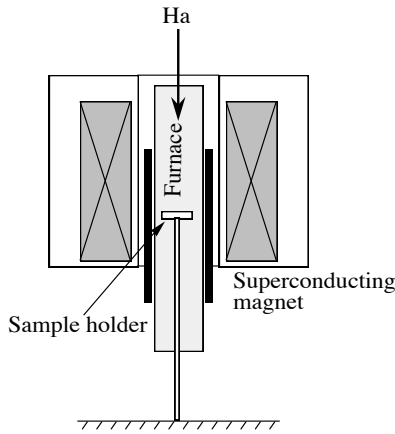
### **2. TEXTURING OF BULK Bi2212/MgO BY MAGNETIC MELT PROCESSING AND HOT FORGING**

#### **2.1 Effect of adding MgO**

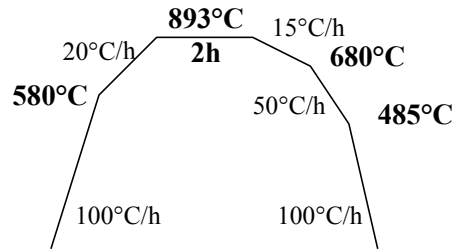
Bi2212 *Hœchst* precursor added with 10 wt.% MgO powder from *Aldrich* (grain size < 44  $\mu\text{m}$ ) is used as starting powder. It is uniaxially cold-pressed into pellets of 20 mm diameter and 5-7 mm thickness. Adding MgO does not change the onset of melting (Pavard 1998). After melt-processing (MP), MgO is located in MgO-rich submicronic inclusions well distributed throughout the Bi2212 matrix. By comparing MP samples containing MgO or not, it is found that adding MgO raises the critical current density  $J_c$  at 77 K by 140%, reduces the  $J_c$  depression in an external magnetic field and increases the irreversibility field by a factor 2 at various temperatures. These enhancements are correlated to the submicronic MgO-rich inclusions which can introduce better indirect vortex pinning. Moreover, MgO adding allows to keep the pellet's shape after MP whereas a Bi2212 pure sample takes the exact shape of the crucible. This is due to an increase in the global viscosity during MP induced by MgO. The effects of adding MgO are reported in Pavard 1998.

#### **2.2 Magnetic melt texturing**

For magnetic melt processing (MMP), the Bi2212/MgO pellets are placed in a furnace inserted in a high magnetic field magnet (see fig. 1). The temperature can reach 1100°C and the vertical magnetic field, 8 Tesla. The magnetic field exerted on the pellets presented in this paper is 5.7 Tesla. The thermal treatment used is shown in fig. 2. Magnetic susceptibility measurements indicate that the onset of melting is 877°C (see Pavard 1998).

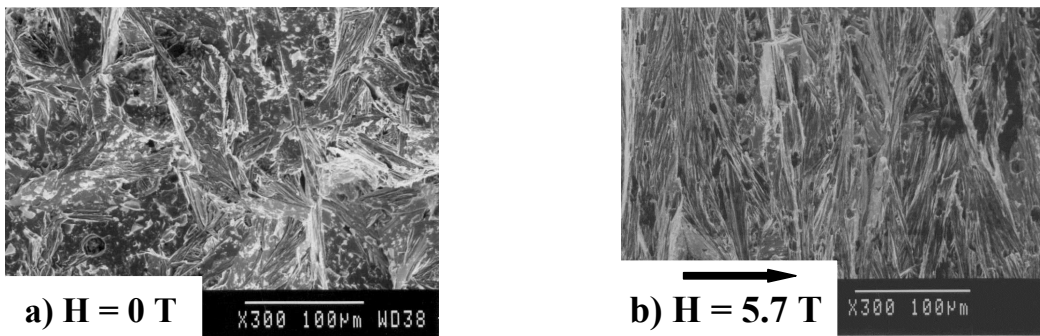


**Figure 1:** Schematic view of the experimental set-up



**Figure 2:** Magnetic melt processing thermal treatment

The orientation induced by the magnetic field applied during MMP is shown in fig. 3 where the microstructures of two samples, one processed in zero magnetic field (MP) and the other processed in 5.7 Tesla (MMP) are compared. No preferred orientation of the platelets occurred in the MP sample whereas they are preferentially oriented with their c-axes parallel to the magnetic field direction in the MMP one. The MMP induced texture explains the difference in  $J_c$  measured on 15 mm<sup>2</sup> section bars at 77 K: respectively 65 and 160 A/cm<sup>2</sup> for the MP and MMP samples. For the MMP sample,  $J_c$  is 500 A/cm<sup>2</sup> on a 1.8-mm<sup>2</sup> section bar ( $J_c$  value obtained without optimization of annealing treatment under reduced atmosphere).

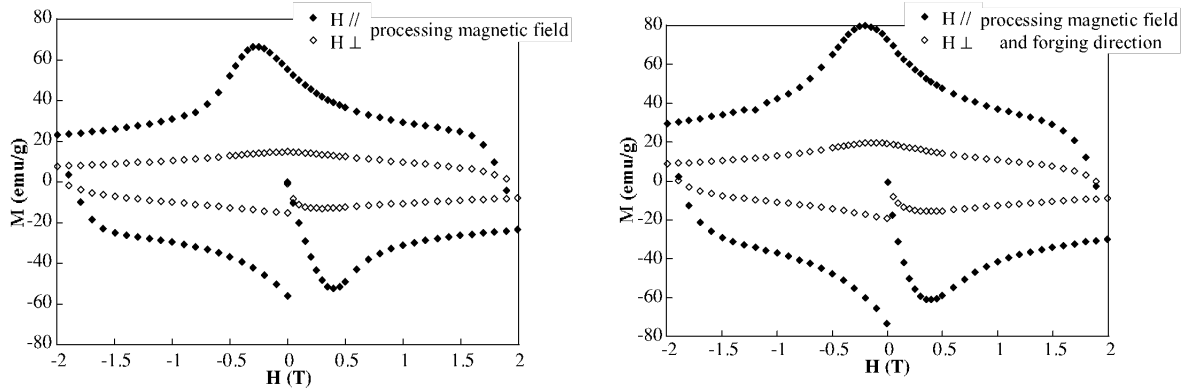


**Figure 3:** SEM micrographs of cleaved surface of bulk Bi2212/MgO melt processed (a) in zero magnetic field and (b) processed in 5.7 Tesla. In (b) the field direction is indicated.

### 2.3 Combination to hot forging

After MMP, bulk Bi2212/MgO materials still have a relative low density (around 80% of the theoretical density for Bi2212 + 10 wt.% MgO). Thanks to MgO, the pellet' shape is kept after MMP allowing further forging treatment on it. Hot forging (HF) at 25 MPa for 2 hours at 880°C applied after MMP increases the density up to 90%, and  $J_c$  at 77 K by 10%: it is 550A/cm<sup>2</sup> on a 2-mm<sup>2</sup> section bar. No phase composition evolution in the bulk due to the HF is observed by SEM. Fig. 4 shows the magnetization loops measured at 4 K on a MMP and on a MMP+HF sample. The magnetic anisotropy  $A$  is defined by  $\Delta M_{//} / \Delta M_{\perp}$ , where  $\Delta M_{//}$  and  $\Delta M_{\perp}$  are the widths of the closed magnetization loops for an exciting magnetic field applied respectively parallel and perpendicular to

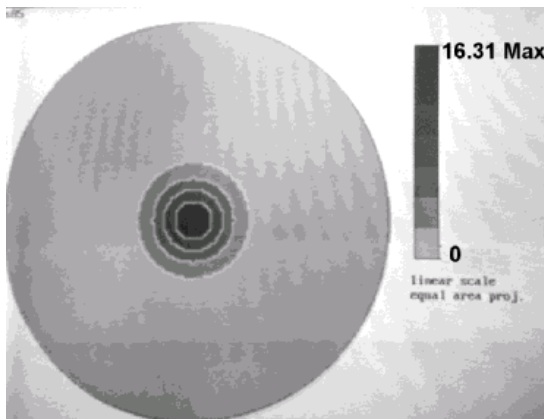
the preferential c-axis direction.  $A = 4.0$  for MMP and  $A = 4.4$  for MMP+HF: HF applied after MMP enhances the texturation degree.



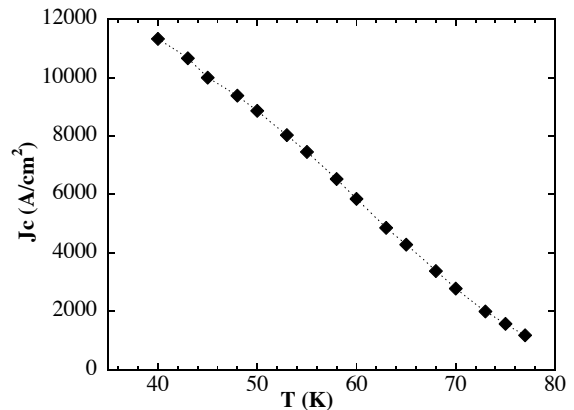
**Figure 4:** Magnetization loops for (a) MMP and (b) MMP+HF measured at 4 K. The exciting magnetic field  $H$  is applied either parallel or perpendicular to the c-axis preferential direction.

### 3. NEUTRON DIFFRACTION TEXTURE STUDY

Neutron diffraction studies on bulk Bi2212/MgO materials were done at the Institut Max von Laue Paul Langevin (ILL) in Grenoble, using the D1B instrument (Bunje 1982) where the sample can be placed on a eulerian cradle. The wavelength of the neutron beam is  $2.52 \text{ \AA}$ . Results obtained on a MMP pellet, a HF pellet (HF alone at 25 MPa and  $880^\circ\text{C}$  for 5 hours) and a MMP+HF pellet are given here. All these bulk samples show an axially symmetric texture: c-axes are oriented parallel to the magnetic field direction or to the forging direction in their whole volume. Pole figures for these three pellets have been calculated using fitting programs developed in Grenoble and in the University of Berkeley (Chateigner 1994, Wenk 1998). The c-axes alignment is evaluated in m.r.d. (multiple of random distribution). The HF pellet shows a texture with c-axis of 9.3 m.r.d, that is an alignment which is 9.3 times stronger than a uniform random orientation distribution. The texture is stronger for MMP: 10.6 m.r.d. The combination MMP+HF gives the best texture, with 16.3 m.r.d. The corresponding (001) pole figure is reported in fig. 5.



**Figure 5:** (001) pole figure for a MMP+HF pellet



**Figure 6:**  $J_c(T)$  for a  $0.3\text{-mm}^2$  section bar from a MMP+HF sample annealed in flowing argon.

### 4. CRITICAL CURRENT DENSITY

#### 4.1 Transport measurements

Transport critical current densities are measured using a criterion of  $1 \mu\text{V/cm}$ . At 77 K, a  $J_c$  of  $1900\text{A/cm}^2$  is reached on a  $5\text{-mm}^2$  section bar cut from a MMP+HF sample, after annealing 20

hours in air at 770°C and at 800°C and then 8 hours in flowing argon at 400°C.

The  $J_c$  evolution with temperature of a MMP+HF sample annealed in flowing argon at 400°C is reported fig. 6. The section bar is small (0.3 mm<sup>2</sup>) because the experimental device is limited in current to 40 A (Pavard 1999).  $J_c$  reaches 11330 A/cm<sup>2</sup> at 40 K.

## 4.2 Magnetic measurements

At 4 K,  $J_c$  is deduced from magnetization loops using the anisotropic Bean's model (Gyorgy 1989). From the loops of fig. 4 measured on cubic samples with 1.4 to 1.5 mm sides (Pavard 1999),  $J_c$  in the a,b planes is well above 100 kA/cm<sup>2</sup> for the MMP and MMP+HF samples.  $J_c$  is respectively 161 and 165 kA/cm<sup>2</sup>. To our best knowledge, these are the highest values reported for bulk Bi2212 superconductors.

## 5. CONCLUSION

Magnetic melt processing (MMP) applied Bi2212 containing MgO is efficient to obtain bulk superconductors with a high texture degree. MgO-rich inclusions enhance the superconducting properties. The texture induced by magnetic melt processing (MMP) is stronger than that obtained by hot forging (HF). Combination of MMP and HF improves the c-axis alignment. This is demonstrated by a neutron diffraction texture study on the whole pellets. The degree of the c-axes alignment is 16.3 m.r.d. for a MMP+HF pellet. MMP and MMP+HF samples show high critical current densities at low temperatures: at 40 K, transport  $J_c$  of 11.3 kA/cm<sup>2</sup> is reached. At 4 K,  $J_c$  higher than 160 kA/cm<sup>2</sup> are magnetically measured with a record value of 165 kA/cm<sup>2</sup> for a MMP+HF sample.

## ACKNOWLEDGMENT

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## REFERENCES

- Angurel L A, de la Fuente G F, Badia A, Larrea A, Diez J C, Pena J I, Martinez E and Navarro R 1997 *Studies of High Temperature Superconductors* **21** (Nova Science, Ed. A V Narlikar), 1
- Bock J and Preisler E 1989 *Solid State Commun.* **72**, 453
- Bunje H F, Wenk H R and Pannetier J 1982 *Textures and microstructures* **5**, 153
- Chateigner D, Germe P and Pernet M 1994 *J. Appl. Cryst.* **25**, 278
- Gyorgy E M, Van Dover R V, Jackson K A, Schennmeyer L F and Waszczak J V 1989 *Appl. Phys. Lett.* **55**, 283
- Holesinger T G, Miller D J, Viswanathan H K, Dennis K W, Chumbley L S, Winandy P W and Youngdahl A C 1993 *Appl. Phys. Lett.* **63**, 982
- Kubo Y, Mishichita K, Higashida Y, Mizumo M, Yokoyama H, Shimizu N, Inukai E, Kuroda N and Yoshida H 1989 *Japan. J. Appl. Phys.* **28**, L606
- Noudem J G, Beille J, Bourgault D, Sulpice A and Tournier R 1994 *Physica C* **230**, 42
- Noudem J G, Beille J, Bourgault D, Chateigner D and Tournier R 1996 *Physica C* **264**, 325
- Pavard S, Villard C, Bourgault D and Tournier R 1998 *Supercond. Sci. Technol.* **11**, 1359
- Pavard S, Bourgault D, Villard C and Tournier R 1999 *Physica C* **316**, 198
- Rouessac V, Poullain G, Desgardin G and Raveau B 1998 *Supercond. Sci. Technol.* **11**, 1160
- Soylu B, Adamopoulos N, Glowacka D M and Evetts J E 1992 *Appl. Phys. Lett.* **60**, 3183
- Wenk H R, Matthies S, Donovan J and Chateigner D 1998 *J. Appl. Cryst.* **31**, 262