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Laser induced Cylindrical Zone Melting of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ Superconductors[#]

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Received ...

Abstract

A new preparative route for $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ superconducting rods, based on the use of a modified Laser Zone Melting system, is presented. It allows to zone melt one or several cylindrical precursors simultaneously under an in-plane geometry. The resultant textured superconductors exhibit a microstructure similar to that of Laser Floating Zone melted fibres and cylinders. The transport critical current and current density values obtained for these products compare favourably with values typically reported in the literature for BSCCO superconductors.

[#]Dedicated to Professor Martin Jansen on the occasion of his 65th Birthday

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Keywords: Superconductor, $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$, Laser, Microstructure

Introduction

The discovery of high temperature superconductors two decades ago brought about a significant effort in the study of complex oxides, especially within the Y-Ba-Cu-O and Bi-Sr-Ca-Cu-O systems [1, 2]. Particularly, the discovery of $\text{Bi}_2\text{Sr}_2\text{CuO}_6$ (Bi-2201) [3] $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ (Bi-2212) and $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ (Bi-2223) [4, 5] provided systems which could be melt-processed with relative ease. Although these materials exhibit incongruent melting, the influence of microstructural defects on the superconducting properties is less severe than in $\text{YBa}_2\text{Cu}_3\text{O}_7$ materials. For this reason melt processing techniques can be conveniently applied to prepare Bi-2212 superconductors. Since these high temperature superconductors are intrinsically anisotropic in nature, directional solidification methods were soon applied in order to prepare them in a highly textured form. Among these methods, CO_2 Laser induced Float Zone (LFZ) melting [6, 7, 8] proved to be convenient to prepare polycrystalline fibers and rods ~~cylinders~~ which exhibited very high texture, with elongated grains preferentially aligned along the superconductor's structure a-b plane [9], curiously the one with the highest J_c [10].

Important advances in laser processing of Bi superconductor cuprates took place with the introduction of Nd:YAG lasers for Float Zone melting [11], which allowed to obtain a more uniform temperature distribution within the melt, as well as nearly flat melt-solidification interfaces [12]. After having improved the processing parameters, J_c values at 77 K as high as 5500 A/cm^2 were obtained in 1mm diameter rods [13], corresponding to $I_c(77\text{K})$ values of the order of 45 A. These values increased linearly with the sample diameter [14] and the current flowed in the outer region of the samples. Optimisation efforts identified the laser power as one of the most important processing parameters. The latter had to be adjusted in order to homogenize the radial phase distribution and to facilitate the use of the superconductor's full

cross-section to transport the current [14].

The rods thus obtained were used as elements in hybrid current leads, one of the first applications of these superconductors. 600 A hybrid current lead units, containing each four Bi-2212 bars, passed stringent tests at CERN [15]. One limitation of these materials was the difficulty to fabricate them in large series. It is precisely this point which provoked us to search for and develop new, more reliable and scalable preparation methods for these Bi-2212 superconductors.

Among the methods developed thereafter, two may be distinctively highlighted. The first would be the more classical-like in-plane Laser Zone Melting (PLZM), first published by Larrea et al [16] for ceramic eutectics. Here the difficulty lied in achieving a sharp, long laser line, which the Bi-2212 sample could displace through at an orthogonal direction in the plane, as described by Mora et al [17]. In order to achieve the necessary long, uniform laser line focus, a laser diode array emitting at 860 nm was used instead of a Nd:YAG laser in the latter work. Both laser emission wavelengths, 1064 nm and 860 nm, induce similar solidification behaviour within the superconductor oxide. The second method was very similar, using the same configuration, but with the support containing a series of Al₂O₃ rods in constant rotation, on top of which were placed superconductor cylinder preforms. This latter method, which may be called Cylindrical Laser Zone Melting (CLZM) will be introduced in this paper, along with the first results obtained for Bi-2212 superconductors.

Finally, a comparison will be carried out among the different types of products obtained using the three preparative techniques mentioned here. In essence, the three geometrical forms (LFZ, PLZM and CLZM) are obtained by laser-induced melting followed by directional solidification in a specific geometry.

Results and Discussion

Cylindrical Laser Zone Melting (CLZM) of Bi-2212 Bars

The most relevant processing parameters used in CLZM of oxides are laser power (W) and irradiance (W cm^{-2}), cylinder rotation rate (rpm) and traverse speed (mm h^{-1}). A Diode laser emitting at 860 nm is used as the energy source. A modified photograph of the apparatus developed for the present experiments is shown in Figure 1, where the molten zone is easily identified (yellow or bright vertical zone approx. 1 mm thick in the picture). To the left of the molten zone is the precursor initiating the melting process; to the right is a zone melted section of each cylinder, with a shiny black aspect and reduced diameter as a consequence of densification during solidification.

Microstructure

The transverse and longitudinal cross-sections of a large number of samples obtained by CLZM were observed by SEM. The micrographs shown in Figures 2-4 correspond to three representative samples. Lower laser powers and higher traverse speeds yield partially melted cylinders, as exemplified in the micrographs shown in Figure 2 (top, a and b). The sample has been processed using a laser power of 1.8 W, a transverse rate of 55 mm/h and a rotation rate of 40 rpm which leads to an irradiance of 17.6 W/cm^2 . Three distinct areas may be appreciated within the transverse cross section: (i) a molten-solidified skin of thickness ca. 100 μm , (ii) the heat affected zone (HAZ) of approx. 60 μm and (iii) the inner, unaffected core. The latter is basically the original compacted cylinder precursor. Some bubbles appear trapped within the solidified region, near the edge of the cylinder. They vary in diameter

approximately between 10 and 40 μm , and are expected from the closing of pores during densification of the porous precursor. The inhomogeneities observed previously in LFZ rods and fibres [18, 19] in terms of diameter, with a periodicity of less than 1 mm, now appear within the Molten-HAZ-Precursor interfaces. These are clearly observed in Fig. 2-b, forming periodic variation domains with respect to the molten-HAZ region thickness.

Phase composition changes drastically at the interface between the HAZ and the molten region. In fact, the HAZ has very similar composition to the core, although it is denser. The molten region microstructure is similar to what has been reported for LFZ rods [11, 13, 17]. $\text{Ca}_{1-x}\text{Sr}_x\text{CuO}_2$ and Bi-2201 are the primary crystallisation phases and they are initially found upon solidification, prior to annealing. These phases are acicular in habit with a pseudo square or rectangular cross-section, as observed in Fig 2-c and -d. This sample does not show a clear longitudinal texture. This can be understood taking into account that texture is mainly induced by the primary $\text{Ca}_{1-x}\text{Sr}_x\text{CuO}_2$ phase. With the processing parameters applied for this sample, the volume of the molten zone is very small and it partially resolidifies during rotation. That is, when the molten zone moves through the lower part of the sample it is screened from direct laser irradiation and solidifies at its surface, in contact with the support. For this reason, the solidification front does not reach the total sample cross-section at this laser power level.

Upon thermal annealing, the I_c value measured for this cylinder was 18 A, corresponding to a J_c value of 420 A/cm^2 considering the total sample cross-section. The critical current of the precursor at 77 K is zero and, in consequence, the electrical current should circulate mainly through the directionally solidified layer or crust, giving a local J_c value of 2400 A cm^{-2} and a critical current density per thickness of $180 \text{ mA}/\mu\text{m}$, taking into account only this molten/solidified superconductor layer.

Similar observations may be made in Figure 3, where micrographs corresponding to cylinders grown at a lower traverse rate of 45 mm/h are shown. In comparison with the previous sample, the irradiance has increased up to 19.9 W/cm². In this case, several differences may be pointed out. First, the molten zone thickness has increased to a value of approx. 360 μm on the average, while the HAZ is approximately 140 μm thick. Secondly, the bubbles trapped within the molten-solidified region are much larger, in the range 30-150 μm. In addition, a well textured region of approximately 100 μm in thickness is identified in the outer region of the sample (Figure 3-c and -d). This gradient in texture is similar to that reported in planar monoliths processed by PLZM techniques [17, 20, 21] and in LFZ rods [13, 14, 18]. This microstructure shows that a planar solidification interface is maintained throughout the external region of the sample. As the depth is increased towards the center of the cylinder, the texture changes from well aligned grains, parallel to the cylinder axial direction, to grains that grow inwards at an angle expected from the change in the solidification gradient [21]. This particular cylinder exhibited an I_c value of 34 A, that corresponds to a critical current density value of 1100 A/cm². Considering that the current is mainly confined in the well-textured external region, this value increases up to 3000 A/cm² and 340 mA/μm considering the well aligned melt-textured thickness. An important point to mention is that the bubbles are mainly concentrated bellow the well-textured region, where most of the critical current circulates. For this reason they have a low influence in the critical current values, although they are expected to affect the mechanical properties of these materials to a significant extent.

Increasing the laser irradiance to values of 22.1 W/cm² enables to melt the complete sample crosssection, as observed for the sample shown in Fig. 4. This sample was processed with a laser power of 2.0 W and the same traverse and rotation rates as the previous sample. The microstructure along a transverse cut of a completely molten cylinder is given in the

micrographs shown in Figure 4 (a and c) and the longitudinal section of the sample after annealing is presented in Figure 4 (b and d). Relatively large bubbles are observed throughout the sample, with sizes approaching 140 μm in diameter. After annealing, the primary crystallisation phases are distributed in the outer ring of the cylinder. They are practically absent from the inner core. A clear feature of this sample is the uniformity of the light grey matrix, which corresponds to the Bi-2212 superconductor phase that is obtained after annealing. The external well-textured region has a thickness of approximately 100 μm , as deduced from Figure 4 c. This thickness is very similar to that obtained in the sample presented in Figure 3, with an intermediate irradiance value. As expected from the larger volume of textured superconductor, the I_c value measured for this sample was 60 A, corresponding to a J_c of 2100 A cm^{-2} and 60 mA/ μm in terms of textured thickness.

The evolution of the sample microstructure in these materials demonstrates that an essential solid state reaction process takes place during annealing in all material forms of Bi-2212 so far studied and obtained from the melt, and that the presently reported CLZM cylinders are no exception. The primary phases which appear during initial solidification from the melt yield a multiphase product with microstructural features defined by the processing method, where $\text{Ca}_{1-x}\text{Sr}_x\text{CuO}_2$ (dark grey contrast grains) and Bi-2001(intermediate grey contrast) are present in significant amounts. These secondary phases then react with the remaining Bi-containing matrix (light grey) during the first step of the annealing treatment to form Bi-2212 superconductor as the major phase in the annealed product. Dispersed residual $\text{Ca}_{1-x}\text{Sr}_x\text{CuO}_2$ and $\text{Bi}_2\text{Sr}_2\text{CuO}_6$ (Bi-2201) secondary phases are still present after annealing; the former is clearly visible in the form of dark-contrast grains with a rectangular-like cross-section habit. The residual Bi-2201 secondary phase is necessary for the development of texture and appears inevitably intertwined with Bi-2212 grains. This is consistent with the fact that

preferential grain alignment and phase purity are competing processes in melt derived, directionally solidified Bi-2212 [22]. In the second annealing step, oxygen adjustment takes place and affects the final T_c value of the superconductor [7].

From the above micrographs, it may be observed that thin, well textured regions are important in order to attain high J_c values in CLZM cylinders. In addition, the technique allows to control the thickness of melt textured superconductor products by choosing adequately the processing parameters. In particular, the molten zone volume section induced in the precursor cylinders may be varied by changing the irradiance level: a 13% increase in irradiance (from 17.6 to 19.9 W/cm²) induces an increase of nearly 300 μm in molten cross section; a 25% increase (from 17.6 to 22.1 W/cm²) induces full melting of the cylinder.

Critical current values of textured Bi-2212 superconductors

The superconductor cylinders presented in this work compare favourably with other textured Bi-based high temperature superconductors obtained via Laser Melting or other variants, as demonstrated by the data summarised in Table I. This table collects data published in the literature throughout the last decade, which seems reasonable to highlight. The referenced work covers different texturing techniques: LFZ Bi-2212 bars [13, 20], electrically assisted LFZ Bi-2212 rods (EALFZ) [21], Bi-2212 rods fabricated using a casting process [23], PLZM Bi-2212 monoliths [24] and Bi-2212 tapes [25].

A texture gradient is obtained in all of the materials prepared using any of the melting processes. In the case of the cylindrical bars, the best texture is always associated with the external sample region, where the current flow is largely concentrated. The processing parameters have to be optimised in order to increase the thickness of this well textured layer and to use its full section for current transport. This has been achieved by designing optimization processes [14] or by inducing the texture using an electrical current during

solidification [23]. Obviously this can be easily attained in the case in thinner samples, where maximum $J_c(77\text{ K})$ values of ca. 5500 A/cm^2 have been reached in 1 mm diameter samples [13]. For diameters up to ca. 3 mm, critical current values increase linearly as a function of thickness [14]; for higher diameters this increase is slower, evidencing the limitations of the usual cylindrical laser melt processing techniques.

A similar texture gradient has been reported in planar monoliths processed by PLZM techniques [17, 24, 26]. In this case the well-textured region is placed close to the sample surface. The thickness of this region can reach several hundred micrometers, above that obtained by other processing techniques. Usually, $J_c(77\text{ K})$ values of the order of 1000 A/cm^2 are obtained in this geometry. They increase up to values of the order of 4000 A/cm^2 when local magnitudes are measured. Similar microstructure gradients have also been observed in Ag/Bi-2212 tapes [25], where the interface of the superconductor core with the Ag sheath is known to contain the highest texture and J_c value within the tape's cross-section.

In general, J_c values decrease in all of these materials when superconductor thickness or radius increases due to the microstructure gradient, regardless of the preparative method employed. An advantage of the CLZM texturing process presented in this work is that it can be used in cylindrical samples with larger sections, modifying only the external surface to attain higher local critical density values. After the texturing process, the sample core (essentially the original compacted powder) may be removed with relative ease to yield a hollow cylinder. This geometry could lead to an increase of the thermal stability of the sample, by increasing the ability of the ceramic superconducting sample to release heat. This is important in many electrical applications, such as resistive fault current limiters, where the avoidance of hot spots is crucial. The latter are related to the presence of defects in the material, reinforcing the idea of developing processing methods which may have a high throughput and may be highly repetitive. These are the underlying ideas behind the preparative method developed and reported here.

Conclusions

A method to prepare Bi-2212 superconductor cylinders with relatively high critical currents has been developed based on the use of a Diode Laser assisted Cylindrical Zone Melting (CLZM) technique. The method allows preparation of several cylinders simultaneously, and the results obtained are comparable to those published in the literature for textured BSCCO. In essence, as described in previous referenced work, the initial compacted superconductor precursor decomposes upon laser melting, yielding a mixture of $\text{Ca}_{1-x}\text{Sr}_x\text{CuO}_2$, $\text{Bi}_2\text{Sr}_2\text{CuO}_6$ and an amorphous matrix of frozen Bi rich liquid phase. This mixture reacts during thermal annealing to produce the final superconducting $\text{Bi}_2\text{Sr}_{2-x}\text{CaCu}_2\text{O}_8$ phase in the presence of a minor amount of secondary phases. The method is applicable to many other types of solids and allows to transform precursor metal oxide compacted cylinders into textured products such as cylindrical monoliths and tubes, by controlling the laser power and irradiance, as well as the rotation and traverse rates of the sample.

Experimental

Precursor cylinders were prepared by cold isostatic pressing (2 min at 200 MPa) a commercial stoichiometric precursor powder supplied by Nexans Superconductors. These powders, with chemical composition $\text{Bi}_{1.98}\text{Sr}_{2.01}\text{Ca}_{1.02}\text{Cu}_2\text{O}_x$, have a near 100% Bi-2212 phase, determined by X-Ray diffraction, and a particle size distribution characterized by $d_{50}=3.9\text{ }\mu\text{m}$. These precursors are cylinders of 10 cm in length and 2.3 mm in diameter with an average density of 4.8 g/cm^3 .

The apparatus used to carry out Cylindrical Laser Zone Melting (CLZM) experiments is described in Figure 1. It makes use of a 300 W Diode Laser (Monochrome S. A., Barcelona; $\lambda = 860$ nm, cw mode) source whose beam output is shaped into a thin, long line, by placing two cylindrical lens with opposing optical axes in the beam path. Alumina rods are placed in a rotation mechanism and serve as support of the superconductor cylinder preforms. The power of the laser, rotation rate and traverse speed are all kept constant throughout the CLZM experiment. As illustrated in Fig. 1, the direction of movement is orthogonal to the laser focus line, in order to achieve planar growth interfaces. The apparatus allows processing several cylinder preforms simultaneously under the same conditions. A 0.5 mm thin by > 5 mm wide molten zone was induced, which allowed to process two cylinders simultaneously at the same melting-solidification rate. Once the laser passed through the selected length of material, the laser beam was suddenly interrupted and the sample was allowed to cool for several minutes while rotating, before its removal for further processing and characterisation.

After the texturing process, samples had to be annealed in order to recover the Bi-2212 phase. A two-step thermal treatment of 60 h at 870°C followed by 12 h at 800°C was performed in air. This annealing was selected because it is the result of an optimization process that was defined for LFZ Bi-2212 textured rods with a similar phase distribution in the textured region [14].

Electrical transport measurements were performed using the conventional four-point probe configuration on 5 cm long samples immersed in liquid nitrogen. Silver contacts were placed on the samples before annealing. For the determination of the critical current values, samples were submitted to sinusoidal current ramps with periods of 2-6 s. Self-field critical current (I_c) values were determined at 77 K using the $1 \mu\text{V cm}^{-1}$ criterion. SEM observation was carried out with a JEOL 6400 microscope fitted with a LINK ANALYTICAL EDS system.

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Figure captions

1. Photographic illustration of the experimental setup used for the Cylindrical Laser Zone Melting (CLZM) experiments performed with rotating cylinder preforms obtained by cold isostatic pressing.
2. SEM micrographs obtained on an as-grown cylinder processed at 55 mm/h traverse rate, 40 rpm rotation rate and approximately 1.8 W of laser power. Transverse cross section cuts are shown in (a) and (c), while longitudinal cross section cuts are shown in (b) and (d). Transport values of 18 A (I_c), 5.8×10^3 A cm⁻² (J_c) and 180 mA μm⁻¹ ($J_{c,thickness}$) were determined for this sample after annealing.
3. SEM micrographs obtained on an as-grown a cylinder processed at 45 mm/h traverse rate, 40 rpm rotation rate and approximately 1.8 W of laser power. Transverse cross section cuts are shown in (a) and (c), while longitudinal cross section cuts are shown in (b) and (d). Transport values of 34 A (I_c), 3.0×10^3 A cm⁻² (J_c) and 85 mA μm⁻¹ ($J_{c,thickness}$) were determined for this sample after annealing.
4. SEM micrographs obtained on a superconductor cylinder processed at 45 mm/h traverse rate, 40 rpm rotation rate and approximately 2.0 W of laser power. Transverse cross section cuts of the as-grown sample are shown in (a) and (c), while longitudinal cross section cuts of the samples after annealing are shown in (b) and (d). Transport values of 60 A (I_c), 2.1×10^3 A cm⁻² (J_c) and 60 mA μm⁻¹ ($J_{c,thickness}$) were determined for this sample after annealing.

Figure 1 (Angurel et al)

Figure 2 (L. A. Angurel et al)

(a)

(b)

(c)

(d)

Figure 3 (L. A. Angurel et al)

(a)

(b)

(c)

(d)

Figure 4 (L. A. Angurel et al)

TABLE I. Published experimental Critical Current data at 77K for LFZ cylinders, PLZM in-plane monoliths and other textured superconductor forms, for comparison with that corresponding to the CLZM cylinders obtained in the present work. In the latter case, values associated with both, the full section and the well-textured region are presented.

Processing [Reference]	Planar LengthxCross section (mm ³)	Diameter (mm)	Effective SC section (mm ²)	v(mm/h)	Ω (rpm)	I _c (A)	J _c (A cm ⁻²)
CLZM [This study]	N/A	2.3	4.31 0.7	55	40	18	420 2400
CLZM [This study]	N/A	2.0	3.1 1.1	45	40	34	1100 3000
CLZM [This study]	N/A	1.9	2.8	45	40	60	2140
LFZ [20]	N/A	1.75	2.4	30	24	80	3330
LFZ [13]	N/A	1.00	0.8	15	24	45	5700
EALFZ [21]	N/A	≈ 1.75	2.40	15	N/A	68	2800
Bi-2212 casting [23]	N/A	10	78	N/A	N/A	437	556
PLZM [24]	100x8x0.4	N/A	3.2	15	N/A	42	1300
PLZM [24]	100x8x2	N/A	3.2	35	N/A	28	875
Bi-2212 tapes [25]	100x3x0.04	N/A	0.1	N/A	N/A	2.8	2800