Critical current density dependence of bulk textured Bi-2223 on thermal treatments and cold intermediate pressing

V. Garnier *, S. Marinel, G. Desgardin

Laboratoire CRISMAT, ESCTM UMR CNRS-ISMRA 6508, 6 Boul. du Maréchal Juin, 14000 Caen Cedex, France

Abstract

We report bulk Bi-2223 transport measurements for samples of various synthesis conditions. The measurements were carried out at 77 K in self-field and the critical current density, \( J_C \), reaches up to 12 000 A/cm\(^2\) depending on the sample’s preparation. The samples were prepared using a polymer matrix method. The same calcination procedure was performed on all the samples. After milling and pelleting, calcined pellets were either directly sinter-forged (838 °C/20 h/10 Mpa) or sintered at 838 °C for various times (25–50–75–100–200 h) before being sinter-forged under the same conditions as the just-calcined pellets. The resulting hot-forged discs were analysed in terms of texture quality (50% of the Bi-2223 grains are aligned within an angle ranging from 6.2° to 3.9°) and Bi-2223 phase percentage (ranging from 26% to 95%). The discs were cut into bars which were either directly annealed (825 °C/100 h + 1 °C per hour to 800 °C under 7.5% O\(_2\) balance N\(_2\)) or subjected to various (1–3) sintering steps (838 °C/50 h) with or without intermediate cold pressing (1 GPa) before annealing. Transport measurements were performed on these samples, and the results are discussed in terms of Bi-2223 phase percentage, texture quality, grain boundaries, effect of cold intermediate pressing and post sintering. This novel type of bulk synthesis process is similar to the one used for tapes, and it allows the preparation of more reproducible bulk samples with high \( J_C \) values and better grain connectivity.

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1. Introduction

Most of the work on Bi-2223-based materials has been performed on Ag/Bi-2223 tapes because of their capability to carry a large critical current density, \( J_C \). But this material can also be used as a bulk sample thanks to its good mechanical properties and smooth resistive transition. The sinter-forging process [1] seems to be the most appropriate method to prepare bulk samples. This process is a pressure sintering method in which the sides of a sample are unrestrained during uniaxial pressing at high temperature, allowing us to obtain, by the grain creeping, a very good texture. Optimized sinter-forging parameters have resulted in a critical current density of up to 11 500 A/cm\(^2\) in 1994 [2]; however since then the \( J_C \) values have
not been improved but only reproduced around 10 000 A/cm$^2$ [3–5]. The limits of these researcher’s preparation method seem to have been reached. Previous studies using the sinter-forging process have used fully reacted powder as a starting material before texturation, while the best Ag/Bi-2223 tapes are made from partially reacted powder. In fact, there are two distinct possible liquid phases in the Bi–Pb–Sr–Ca–Cu–O system. One comes from the partial decomposition of the Bi-2223 phase at high temperature. This partial melting induces an important secondary phase segregation which cannot be re-transformed into Bi-2223 with further annealing, and therefore induces bad grain boundaries and limits the $J_C$ values. The other liquid phase is obtained, using classical sintering temperatures, by the eutectic reaction between the Bi-2212 phase and the other secondary phases. Using this liquid phase, the grain creeping is allowed and the Bi-2223 phase could be formed.

The goal of this study is to utilize, for the first time, different precursor powders before sinter-forging which have varying Bi-2223 phase content and therefore different potential liquid phase amounts. This research proposes the use of post sinter-forging thermomechanical treatments to increase the $J_C$ values, as is classically done in the powder in tube (PIT) process.

2. Experimental

Starting from the stoichiometric composition Bi$_{1.8}$Pb$_{0.35}$Sr$_2$Ca$_2$Cu$_3$O$_{10.1}$ and using the corresponding metal acetates, the powder precursor was prepared by a liquid phase process, the polymer matrix method [6], which allows a homogeneous mix of the commercial powder at the atomic scale. The resulting powder was calcined for 24 h at 820 °C in air [7], milled and pellitized (3 g, 1.5 tons cm$^{-2}$, $\varnothing$ 16 mm). Some calcined pellets were either directly sinter-forged or sintered at 838 °C for various times (25–50–75–100–150–200 h) in air before being sinter-forged under the same conditions as the just-calcined pellets. Pellets were sinter-forged under 10 MPa, the same stress as generally used by other groups [8–13], for 20 h at 838 °C. The forged temperature is lower than that commonly used because fully reacted Bi-2223 powder was not used and this phase decomposition occurs at temperature higher than 850 °C [14]. The liquid phase, in our case, is supplied by the eutectic reaction between the Bi-2212 and the other intermediate phases (Ca$_2$CuO$_2$, CuO, Ca$_3$PbO$_4$). After sinter-forging, hot forged discs of about 0.5 mm thickness were obtained and XRD analysis was performed. Quantitative analyses of the texture degree were also performed on these discs by pole figure measurements, with a Philips X’pert diffractometer, using the {0 0 1 0} peak of the Bi-2223 phase. Each disc was then cut into bars with a wire saw. Some of these forged bars (F) were subjected to different post sinter-forging thermomechanical treatments (S: sintering: 838 °C/50 h and P: cold uniaxial pressing: 1 GPa) described as follows: F + S; F + S + P; F + S + P + S; F + S + P + S + P + S. All these samples were then annealed for 100 h/825 °C + 1 °C/h $\rightarrow$ 800 °C under 7.5% O$_2$–92.5%N$_2$ atmosphere. Their transport critical current densities were measured using the four probe method from a DC current–voltage using a 1 μV cm$^{-1}$ criterion under self-field and 77 K.

3. Results and discussion

Table 1 summarizes the sample characterization. Before the sinter-forging process, the precursor pellets contained different amounts of the Bi-2223 phase proportional to the sintering time, but no Bi-2223 phase was present in the calcined pellet. After sinter-forging, the Bi-2223 phase content increased for all the samples, reaching more than 90% for the precursor pellets sintered for more than 100 h. Thus our sinter-forging parameters continue to allow Bi-2223 phase formation during the texturation step.

Bi-2223 grain orientation analysis, performed on the forged discs, revealed different alignment qualities as a function of the type of precursor pellet. Since the precursor pellets contained various amounts of Bi-2223, the quantity of the liquid phase may also have varied. In addition, the Bi-2223 phase grain size is increasingly important since the precursor pellets have been sintered for a
long time. The best Bi-2223 grain alignment corresponds to the best compromise between the Bi-2223 phase content and the Bi-2223 phase grain size. The best texture quality has been obtained with a precursor pellet sintered for 100 h at 838 °C before sinter-forging. 50% and 90% of the grains have their c-axis aligned between 0–3.9° and 0–12.3°, respectively, with respect to the sinter-forging applied pressure axis. The texture quality is worst for the calcined precursor pellet which has the most potential for liquid phase formation and a smaller grain size. The grain alignment of the precursor pellet sintered 200 h before sinter-forging is worse than that of the 100 h sintered pellet, although its grain size is the largest. This can be explained by a low liquid phase quantity, which prevents a good grain creeping during the texturation process.

The transport critical current density, $J_C$ (77 K, 0 T), of the just-forged samples (F) is directly dependent on the texture quality; the sample with the best $J_C$ corresponds to a precursor pellet sintered 200 h at 838 °C. After the first post sinter-forging thermal treatment (F + S), the $J_C$ values decreased, because of the retrograde densification [15] induced by the Bi-2223 phase formation. This decrease is most pronounced for the calcined precursor pellet. However, for the 200 h precursor sintered pellet, the $J_C$ increased due to better grain connectivity of the already completely formed Bi-2223 phase. After the second post sinter-forging thermomechanical treatment (F + S + P), the $J_C$ values decreased for the samples containing the lowest Bi-2223 phase amount, as those samples seem to be more sensitive to the formation of microcracks. However, the critical current density increased continuously from the precursor calcined pellet to the 100 h sintered pellet, and then decreased for longer sintering time. The $J_C$ seems to be less sensitive to the pressing step for samples having the largest Bi-2223 grain size. With an additional thermal treatment (F + S + P), the $J_C$ values increased for the samples sintered no longer than 150 h and decreased for the others. This second post sinter-forging sintering step has allowed the microcracks induced by the previous pressing step to heal and has also permitted more of the Bi-2223 phase to form, resulting in increased $J_C$ values. Conversely, the $J_C$ values have

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F: forging; S: sintering; P: pressing.

*Means that 50% or 90% of the Bi-2223 grains have their c-axis aligned between 0° and x° with respect to the direction of sinter-forging applied pressure.
decreased for the precursor samples sintered more than 50 h. In this case, the liquid phase is no longer available to heal the microcracks, because the cumulative sintering time has allowed the formation of fully reacted Bi-2223 samples, and not enough secondary phases are present to produce the necessary liquid phase.

4. Conclusion

This work describes a novel type of bulk synthesis process similar to that generally used for the synthesis of Bi-2223 tapes. Using non-fully reacted powder before the sinter-forging process and different combinations of post sinter-forging thermomechanical steps, high $J_C$ values have been obtained with a better reproducibility than the classical sinter-forging process which used fully reacted powder. Critical current densities of 8300 A/cm$^2$ minimum up to 12 000 A/cm$^2$ have been obtained for precursor pellets having the best compromise between Bi-2223 grain size and Bi-2223 phase content. These characteristics allow good grain alignment, and adequate liquid phase encourages the increased formation of Bi-2223, heals the microcracks and improves the grain connectivity. Although the post sinter-forging thermomechanical treatments only slightly improve the $J_C$ values compared to the forged samples, the results described in this work are encouraging, and a better control of the post sinter-forging thermomechanical treatments conditions will improved the $J_C$ values.

References