Improvement of critical current density of MgB₂ bulk superconductor processed by Spark Plasma Sintering

Jacques G. Noudem¹ | Yiteng Xing¹ | Pierre Bernstein¹ | Richard Retoux¹ 
Masaki Higuchi² | Srikanth S. Arvapalli² | Miryala Muralidhar² | Masato Murakami²

¹Normandie Univ, ENSICAEN, UNICAEN, CNRS, CRISMAT, Caen, France 
²Superconducting Materials Laboratory, Graduate School of Science & Engineering, Shibaura Institute of Technology, Tokyo, Japan

Correspondence
Jacques G. Noudem, Normandie Univ, ENSICAEN, UNICAEN, CNRS, CRISMAT, 14000 Caen, France. Email: jacques.noudem@ensicaen.fr

Abstract
Spark Plasma Sintering (SPS) is a promising rapid consolidation technique that allows a better understanding and optimization of the sintering kinetics and therefore makes it possible to obtain MgB₂ bulk superconductor with tailored microstructures consisting of grains with either spherical or elongated morphology. In this contribution, the role of the precursor powders on the superconducting properties of MgB₂ is investigated. Three sets of bulk MgB₂ material were processed from: (i) a commercially available MgB₂ powder; (ii) a mixture of Mg metal and amorphous B using a single-step solid-state reaction process and (iii) a mixture of amorphous boron coated with carbon and Mg metal. The samples were prepared in the same SPS processing conditions. The microstructure of the samples was investigated by X-ray diffraction, SEM and TEM and correlated to their superconducting properties. The critical current density of the best sample at 20K was $J_\text{c} = 500 \text{kA/cm}^2$ in self-field, which is one of the highest critical current density reported for MgB₂ bulk superconductors.

KEYWORDS
critical current density ($J_\text{c}$), flux pinning, MgB₂, microstructure, spark plasma sintering

1 | INTRODUCTION

Intermetallic MgB₂ of hexagonal structure is well known since 1953.¹ In 2001, its superconducting behavior up to $T_\text{c} = 39$ K, due to the existence of two different superconducting gaps, was discovered.² This material has several advantages for applications, especially its low cost, good mechanical properties,³,⁴ and large coherence length.⁵ Its upper critical field is higher than that of conventional NbTi, and Nb₃Sn superconductors. It shows no weak links and less anisotropic effects than superconducting cuprates.⁶,⁷ In addition, MgB₂ has a density of 2.6 g cm⁻³, which is much lower than that of any other superconductors, making it the most suitable candidate for lightweight applications.

Akin to YBa₂Cu₃O₇₋δ (YBCO) and (Rare-Earth) Ba₂Cu₃O₇ (REBCO) cryomagnets,⁸,⁹ bulk MgB₂ could be used for various applications such as the delivery of drug at specified locations in the human body using an external magnetic force,¹⁰ magnetic separation systems,¹¹ portable nuclear magnetic resonance (NMR) and magnetic resonance imaging (MRI) devices,¹² generators or motors¹³,¹⁴ etc… For all these applications, the trapped magnetic field of present MgB₂ bulks must be improved. According to the Bean model, the trapped field is proportional to the critical current density, $J_\text{c}$,
and depends on the size of the persistent current loops,\textsuperscript{15} that is linked to the sample size. As a consequence, to obtain high trapped fields it is necessary to make large superconducting bulks comprising effective pinning sites. For $J_c$ enhancement, different strategies have been investigated including irradiation,\textsuperscript{16-18} chemical doping,\textsuperscript{19-21} or substitution,\textsuperscript{22} particle size refinement by mechanical ball milling\textsuperscript{23} and other techniques. Among various chemical dopants, carbon was one of the most effective choice. While carbon as dopant via various sources, uniform carbon distribution was always an issue,\textsuperscript{24} leading to nonuniform superconducting performance that is not desired. Hence, research focusing on achieving uniform carbon distribution was carried out in recent decades. One famous recent technique to achieve this was usage boron that is coated with boron, also known as carbon coated boron (CCB). Several researchers have employed this CCB, which was prepared via specific pyrolysis technique, and attained uniform carbon distribution as well as $J_c$. Some studies reported that a low carbon wt% of around 1.5 wt% is optimum for producing best $J_c$.\textsuperscript{25,26} While other studies have incorporated Cu addition in conjunction to CCB powder, to ensure low temperature synthesis (600°C) by means of Cu activated sintering and enhance the $J_c$ via low MgO as uniform carbon doping.\textsuperscript{27} Furthermore, the Cu addition as a coating can help in obtaining slightly higher density MgB\textsubscript{2} wires produced via Internal Mg Diffusion (IMD) process.\textsuperscript{28} Classically, bulk MgB\textsubscript{2} is prepared by conventional sintering\textsuperscript{29-31} or by infiltration processes.\textsuperscript{32} However, the density of the obtained bulks is quite low, about half the value of the theoretical density. Fortunately, some processing techniques can be used to prepare dense MgB\textsubscript{2} samples, especially (i) hot isostatic pressing (HIP),\textsuperscript{33} (ii) hot compaction or pressing (HP),\textsuperscript{34,35} (iii) high-pressure sintering (HPS),\textsuperscript{36,37} and (iv) spark plasma sintering (SPS).\textsuperscript{38-40} This last technique SPS, offers the possibility to prepare samples with a complex shape and/or a large size.\textsuperscript{38,39} In this study, SPS was employed for obtaining sintered polycrystalline MgB\textsubscript{2} bulks. The influence of the starting powder on the superconducting properties of MgB\textsubscript{2} has been investigated. By the optimization of both the starting powder and the SPS processing parameters as well as sintering conditions, the $J_c$ of the samples was improved as a result of the controlled microstructure and the increase of their density and flux pinning capability.

In section 2, we describe the experimental procedures we have used for the fabrication and the characterization of the samples. The results are reported in section 3.

## 2 | EXPERIMENTAL PROCEDURE

Three groups of samples differing by their precursor materials were fabricated: (i) the first group was fabricated with a commercially available (MgB\textsubscript{2}, purity >97%, 100 meshes) powder made by PAVEZYUM, Advanced Chemicals, Turkey, (ii) the second with a mixture of Mg metal (99.9% purity, 200 meshes) and amorphous boron (99% purity, 300 meshes) (Mg + 2B) using a single-step solid-state reaction process and (iii) the third one with a mixture of amorphous boron coated with carbon and Mg metal (Mg + B/C). High-purity Mg metal (99.9% purity, 200 meshes) was purchased from Furu-uchi Chemical Corporation, Japan, while crystalline boron powder (98.5% purity) and 1.5 wt% carbon-encapsulated amorphous boron powder (98.5% purity, 250 nm size particles) were purchased from PAVEZYUM. The process used for mixing the powder components is reported elsewhere\textsuperscript{24}.

Graphite foils were wrapped alongside of inner walls of graphite mold that was filled up with the powder to sinter and the mold was located in the spark plasma sintering set-up (FCT System GmbH, HD25, Rauenstein, Germany). Details on the shaping and densification of the samples are given elsewhere.\textsuperscript{33} Briefly, the SPS was operated in DC mode with high intensity current pulses (2000 A, 4 V) and a 50 MPa uniaxial pressure under dynamic vacuum (10\textsuperscript{-3} bar). The sintering temperature was in the 800-1000°C range. During the whole process, the temperature, the applied pressure, the shrinkage curve as well as the piston speed were recorded. After the sintering step, the as-prepared samples of 20 mm diameter and 8 mm height were polished to remove the carbon traces due to the graphite foils. We present below the results obtained with three samples whose characteristics are typical of each group. They are named as follows: S1 is related to the bulks fabricated with the commercial MgB\textsubscript{2} powder, S2 to the samples made by mixing Mg and B powders (Mg + 2B), and S3 to the bulks made with the Mg + B/C powder.

The density of the samples was determined with ethanol by the Archimedes method. X-ray diffraction was carried out with a Philips 0-20 diffractometer using the monochromatic Cu-K\textsubscript{α} radiation. The microstructure of the samples was analyzed with a Carl Zeiss (Supra 55, Oberkochen, Germany) scanning electron microscope (SEM) and transmission electron microscopy (TEM) was used to investigate the finer microstructural features. Energy Dispersive Analysis and High Resolution Electron Microscopy observations were performed at room temperature on a 200 kV JEOL 2010 FEG STEM electron microscope (tilt ±42°) equipped with an EDS (Energy Dispersive Spectrometer, Si/Li detector) EDAX and fitted with a double tilt sample holder. Cross sections of densified samples for TEM observations were prepared by ion milling at low voltage (Argon gas) to prevent heat damages using a JEOL Ion Slicer.

For investigating the superconducting properties, small specimens with dimensions in the range 2 x 2 x 2 mm\textsuperscript{3} were cut from the bulks in order to measure their critical temperature, $T_c$, and to record their magnetization hysteresis loops ($M$-$H$ loops) in the 10 K-30 K range with a SQUID magnetometer.
Their critical current density as a function of the applied field was estimated from the M–H loops with the extended Bean critical state model for rectangular samples. The spatial trapped field distribution at 20 K was mapped 20 minutes after cooling the sample to the same temperature 10 mm away from a 45 mm diameter permanent magnet using a cryogen-free cryo-cooler. During these measurements, the temperature was monitored with two sensors. One was located on the cold head of the cryo-cooler and the other on the sample surface. The Hall probe used for mapping the field was located above the cryostat 10 mm away from the superconductor. Levitation force measurements were also carried out at 20 K after cooling down the samples at 35 mm from the permanent magnet.

3 | RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction diagrams (XRD) of the processed samples. Almost all the peaks in the XRD patterns correspond to MgB₂. S1 shows traces of MgO, compound already present in the as-supplied commercial MgB₂ powder. The formation of a small amount of MgO visible around 62° in the XRDs of S2 and S3 is due to the high reactivity of Mg with residual oxygen during the processing step. Using Rietveld refinements, the MgO amount was evaluated at 1.35 and up to 3.38 wt.% for the starting commercial powder and the sintered samples respectively. Different authors have pointed out this additional amount of MgO and the effect on the superconducting properties. A residual carbon peak is observed at 26°. It is probably due to the graphite mold. However, in case of S3 the carbon used to coat the boron may also contribute.

The MgB₂ peaks correspond to a hexagonal structure with the space group P6/mmm. The lattice parameters were calculated and are reported in Table 1. They do not show any significant variation with respect to the theoretical values.

The SEM micrographs of S1, S2, and S3 are shown in Figure 2. Sample S1, which was processed directly from commercial MgB₂ powder, has more pores than S2 and S3. This is confirmed by the estimation of the packing ratio which is around 73, 75, and 84% for S1, S2, and S3, respectively. The different packing ratios could be related to the particles size shape of starting powder and the chemical reaction (Mg + 2B or Mg + MgB₄) during the processing stage. In these cases, the magnesium diffusion at low temperature is useful to improve the contact between grains. In these cases, the magnesium diffusion at low temperature is useful to improve the contact between grains and consequently, the samples density. The S2 and S3 samples are obtained after reactive sintering (called in situ process) in contrary of ex situ process for S1 where the MgB₂ was already formed. Small size spherical grains can be observed in Figure 2B, C, which correspond to the samples prepared with the powders containing nano-boron particles. The presence of spherical grains is clearly evidenced in the micrograph of the powder before processing shown in the insert of Figure 2C. In addition, in Figure 2D, TEM images of sample S3 show also small grains. The enlargement presented in the inset of Figure 2D attests of the good crystallinity of the nanoparticles in spite of the coexistence in these nano-grains of many defects, domains and strains resulting in perturbed contrasts.

Figure 3A shows the normalized magnetic moment-temperature curves of S1, S2 and S3. The measurements were made while applying a 1 mT external magnetic field. Samples S1 and S2 show a low transition width at Tc = 38.7 K, while for S3, Tc = 35 K. This low Tc value could be due to the partial substitution of boron by carbon.

The samples critical current density were estimated from their magnetization hysteresis cycles at 20 K (see the inset in Figure 3B) by applying the extended Bean critical-state relation:

\[
J_c = \frac{20DM}{a(1-(a/b))}
\]

In Eq.(1) \(\Delta M\) is the width of the magnetic hysteresis cycle measured in emu/cm² while a and b are the sample dimensions in cm (with a < b). Figure 3B shows the magnetic field dependence of \(J_c\) at 20 K for the three samples. In self-field, \(J_c\) is equal to 1.14 x 10⁴, 3.84 x 10⁵, and 4.98 x 10⁵ A/cm² for

| Table 1 | Lattice parameters and c/a ratio of the fabricated samples |
|---------|----------------------------------------------------------|
| Sample  | a (Å) | c (Å) | Δ (c/a) |
| MgB₂ (value of literature) | 0.3086 | 0.3524 | 1.141931 |
| S1      | 0.3081 | 0.3514 | 1.140499 |
| S2      | 0.3084 | 0.3520 | 1.141237 |
| S3      | 0.3074 | 0.3523 | 1.146346 |

FIGURE 1  XRD patterns of samples S1 (commercial MgB₂), S2 (Mg + B) and S3 (Mg + B/C) [Color figure can be viewed at wileyonlinelibrary.com]
S1, S2, and S3, respectively. The increasing critical current density from S1 to S2 and from S2 to S3 is linked to the increasing density of the samples. The low difference between the densities of S1 and S2 indicates the existence of a density threshold to obtain volumetric MgB2 with a large $J_c$. While $J_c$ for S2 and S3 is significantly larger than for S1, that was prepared with the MgB2 powder, it is also larger than the values reported in $^{30}$ (2.7 $\times$ 10$^5$ A/cm$^2$) and in $^{44}$ (3.8 $\times$ 10$^4$ A/cm$^2$), that were obtained with bulks fabricated by conventional sintering. It is in the range of the values reported for bulks fabricated by hot-pressing (4.8 $\times$ 10$^5$ A/cm$^2$)$^{37}$. This comparison is summarized in Table 2.

In the 2 T range, $J_c$ is around 9.2 $\times$ 10$^4$ A/cm$^2$ and 1.5 $\times$ 10$^5$ A/cm$^2$ for S2 and S3, respectively. At high field, the $J_c$ of S3 is twice that of S2. The improved results are primarily due to the good pinning of the vortices in S3, resulting from the large number of grains boundaries present in S3 as compared to S2, that is visible when comparing Figure 2B,C.

**TABLE 2** Critical current density, $J_c$, at self-field, 20 K of sample S3 from this work compared to some reported data$^{30,37,42}$

| $J_c$ (kA/cm$^2$) | 498 | 270$^{30}$ | 480$^{37}$ | 38$^{42}$ |

$J_c$ of sample S3 as a function of the applied magnetic field at various temperatures [Color figure can be viewed at wileyonlinelibrary.com]

At 4 T, the critical current densities are around 4.4 $\times$ 10$^3$ and 9.6 $\times$ 10$^3$ A/cm$^2$ for S2 and S3, respectively. The dependence of the critical current density of S3 on the applied magnetic field and the temperature is plotted in Figure 4. As expected, $J_c$ decreases if the applied field or the temperature increases.
Typical $J_c$ are reported in Table 3. At 10 K, $5.06 \times 10^5$, $3.73 \times 10^5$, and $1.56 \times 10^5$ A/cm$^2$ were measured at 1, 2, and 3 T, respectively. We also point out the $J_c$ values equal to $1.80 \times 10^5$ and $5.16 \times 10^4$ A/cm$^2$ at 25 and 30 K, respectively, demonstrating the strong flux pinning and high compaction of the S3 sample.

Figure 5 shows the flux density above the surface of S3 at 20 K, after field cooling the sample with a 45 mm diameter NdFeB permanent magnet located above the cryostat at 10 mm from the superconductor generating 0.3 T at the surface of S3. The measurements were made by scanning a Hall sensor 10 mm above the bulk surface. The trapped field distribution shows a single dome as in single domain YBaCuO bulks. This conical distribution of the magnetic field can be associated to the homogeneous transport of the current through the grain boundaries. This is in contrast to YBaCuO bulks in which the superconducting current flows across the grains boundaries with a large $J_c$ only if the sample is textured. The trapped field of 0.3 T is lower than 1.5 T reported elsewhere, with the same size. In this case, the authors used the coil with the applied external field up to 3 T for sample magnetization. In this study, we have as a magnetization source a permanent magnet with a maximum external magnetic field of 0.5 T. For the future work, we will magnetized our samples at high applied field in order to investigate the trapped field capacities.

Figure 6 shows the levitation force measured at 20 K on S1 and S3 after field cooling the samples with the 45 mm diameter magnet at a distance of 35 mm. As could be expected, the levitation force increases as the separation between the superconductors and the magnet decreases. The levitation force measured on S3 is twice as large as that measured on S1. This is attributed to the difference in $J_c$ between the two samples (see Figure 3B).

### 4 | CONCLUSION

The results reported in this contribution show that the fabrication of MgB$_2$ bulks by Spark Plasma Sintering with starting powders containing nano-particles of boron result in samples with a packing density of 84%, large enough to ensure a large critical current density, $J_c = 500$ kA/cm$^2$ at 20 K in self-field. In presence of a magnetic field, $J_c$ decreases but in the 20 K range, it keeps values large enough for most applications up to 4 T. The combination of these characteristics to the light weight and to the mechanical properties of bulk MgB$_2$ show that this material could be the best candidate for applications using liquid hydrogen as cryo-coolant, especially the future electric planes or cryo-coolers working in the 20 K range, as for example NMR portable set-ups.

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