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High Magnetic Field Generated by Bulk MgB_2 Prepared by Spark Plasma Sintering

Kévin Berger, Michael Rudolf Koblichka, Bruno Douine,
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Abstract—From the applications point of view, the advantage of low density given by MgB_2 material must be taken into consideration and, the generation of strong magnetic flux densities using MgB_2 should be investigated. In this contribution, we have studied the magnetic properties of samples processed by a fast Spark Plasma Sintering machine that is able to produce dense and high quality MgB_2 samples. Experiments were carried out both on small pieces of the samples and on large-sized samples performing trapped field measurements in a Field-Cooling process. For temperatures between 10 K to 30 K, the results show a strong dependence of the magnetic behavior of the large-sized samples to the applied magnetic field sweep rate, while nothing particular appeared on the small samples. As the magnetic flux density produced by MgB_2 bulks is directly linked to the potential of the applications, we report the field produced at the surface of one single MgB_2 sample of 30 mm diameter and the field produced inside a stack of two MgB_2 samples of 20 mm in diameter. A generation of magnetic flux density (magnetic polarization $\mu_0 M$) up to 4.80 T @ 10 K and 3.92 T @ 20 K inside the stack of the two MgB_2 samples was observed under negative supporting field of -1.47 T and -1.95 T, respectively. According to these values, to their very low density and to their ease of manufacturing, MgB_2 bulks are promising materials for the applications of superconductors.

Index Terms—Bulk MgB_2 , Field-cooling process, Flux jumps, Strong magnetic field, Trapped magnetic field.

I. INTRODUCTION

IN the field of High Temperature Superconductors (HTS), some applications requires the fabrication of large, bulk samples in order to employ them for levitation devices, fault current limiters, non-contact bearings for liquid pumping, magnetic shielding screens, and motors. MgB_2 material

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represents currently the metallic superconductor with the highest transition temperature [1]. A very promising feature of this material is that one can achieve a high critical current density in polycrystalline samples without aligning the crystal orientation with a substrate, which enables a much cheaper production technology [2]. Besides, different methods can be used for densifying the material such as Spark Plasma Sintering (SPS) process [3]. Using this process, MgB_2 bulk samples with 99% of relative density have been obtained [4]. Theoretically, bulk MgB_2 samples have very low density of 2.63 g/cm^3 , compared to (RE)BaCuO samples which are around 6.36 g/cm^3 , and NdFeB permanent magnets at around 7.60 g/cm^3 . Practically, this ratio is maintained higher than 2–3. From the applications point of view, this advantage must be taken into consideration and, the generation of strong magnetic flux densities using MgB_2 should be investigated, even though MgB_2 requires a lower operation temperature. Nowadays, with developed cryocooling systems, this is not a large obstacle anymore.

In this contribution, we studied the magnetic properties of samples with a density of 2.61 g/cm^3 , processed at CRISMAT by a fast SPS machine that is able to produce dense and high quality MgB_2 samples. The process, the structural properties and the superconducting transition characteristics of the obtained bulks were reported previously [4]. Experiments were carried out both on small pieces of the samples and on large-sized samples performing trapped field measurements in a Field Cooling (FC) process.

II. SAMPLES CHARACTERIZATION

Magnetic characterization of a small sample piece of $1.377 \text{ mm} \times 1.473 \text{ mm} \times 0.638 \text{ mm}$ was carried out at IJL Nancy using a Magnetic Property Measurement System (MPMS@3) from Quantum Design.

The DC magnetic moment m was measured at 10 K, 20 K and 30 K, for an applied field parallel to the longest dimension of the sample. Half of the Major Hysteresis Loops (MHL) of the magnetic moment m are shown in Fig. 1. The field was cycled between $+5$ T and -5 T with various sweep rates: 1 mT/s, 4 mT/s and 20 mT/s. In this range, with these small samples, no phenomena of flux jumps and no influence of the sweep rate were observed.

A. Determination of $J_c(B)$

The critical current density J_c is calculated from the Major Hysteresis Loop (MHL) for which the sample is in the critical

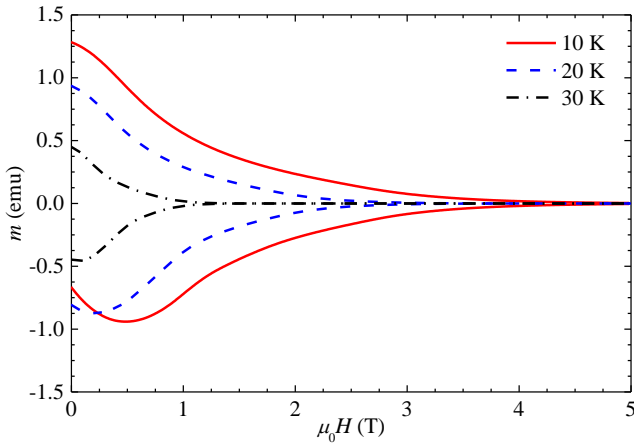


Fig. 1. Half of the Major Hysteresis Loops of the magnetic moment m of a small MgB_2 sample measured at 10 K, 20 K and 30 K, for an applied field cycled between +5 T and -5 T.

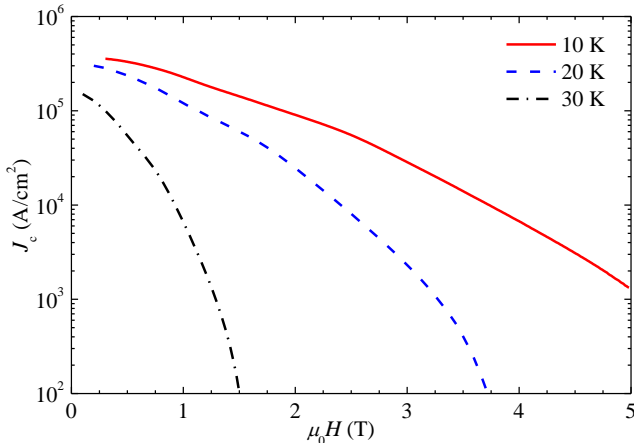


Fig. 2. Field dependence of the critical current density calculated at 10 K, 20 K and 30 K from the MHL in Fig. 1.

state. In the case of an infinitely long sample with a rectangular cross-section ($2a \times 2b$, with $a < b$), the critical current is given by

$$J_c(B) = \Delta M (a(1 - a/3b))^{-1} \quad (1)$$

where ΔM is the width of the curve [5], [6].

It can be noticed that due to some uncertainty in the size measurements of the sample and its volume estimation, the value of the critical current density may be overestimated by 5%. Nevertheless, the critical current densities at 10 K and 20 K exhibit values higher than 10^5 A/cm^2 for 1 T of applied field, what is a relatively good result without doping [7].

B. Irreversibility field H_{irr}

The irreversibility field was determined at the point where J_c meets the criterion of 100 A/cm^2 . This irreversibility field is greater than 5 T at 10 K, and equal to 3.71 T and 1.50 T, respectively at 20 K and 30 K.

III. EXPERIMENTAL SETUP

A. Apparatus

The cryostat employed for the trapped field measurements in our case is an Oxford Instruments 5 T low-loss cryostat with a room-temperature bore. On removing the secondary cryostat for sample cooling used for low-temperature STM

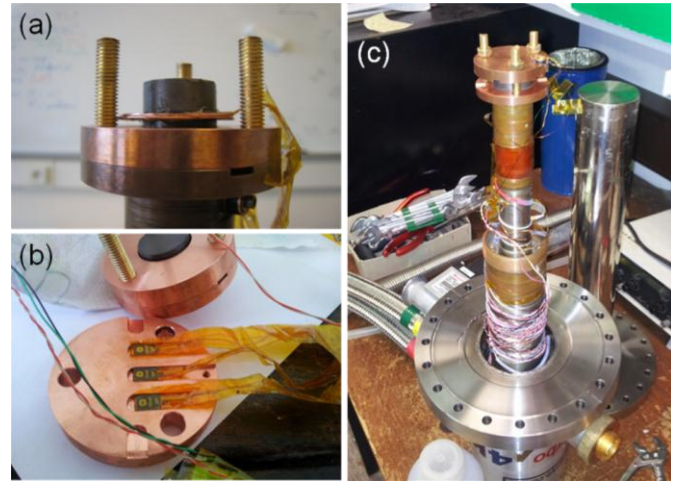


Fig. 3. (a) Head of the cryocooler with two bulk MgB_2 samples in place. (b) Copper top-plate with slits for three Hall probes spaced by 1 cm. (c) The entire cryocooler system with a mounted sample.

and MFM work, the room-temperature bore of 7.5 cm diameter is fully exposed. Fitting a cryocooler under it enables trapped-field experiments to be performed in magnetic fields up to $\pm 5 \text{ T}$ perpendicular to the sample surface. For our experiments, we have employed the ARS-4K-cryocooler system, which can reach temperatures down to 4.2 K but the lowest temperature in our experiments is roughly 9 K.

A bi-directional power supply for the superconducting coil (Oxford Instruments IPS 120-10) enables a continuous field sweep through 0 T together with a controlled magnetic field-sweep rate ranging up to 40 mT/s (2.4 T/min).

Up to 3 Hall probes may be employed to measure the fields at the sample surface. We have used high linearity Hall probes HHP-NP from Arepoc which have a sensibility higher than 70 mV/T. The overall dimensions of the Hall probe are $7 \text{ mm} \times 5 \text{ mm} \times 1 \text{ mm}$ and the active area is approximately 0.35 mm far from the sensor surface. The calibrations of these sensors were performed at each mounting of a sample.

Fig. 3 (a) presents the sample stage of the cryocooler with two bulk MgB_2 samples in place. There are copper discs between the 2 samples in order to connect them thermally despite the presence of the Hall probes. In Fig. 3 (b), a copper ring is fitted around the sample for better temperature exchange during the cooling, and another copper plate is then fixed above the sample containing three slits for the Hall probes. Finally, Fig. 3(c) shows the entire cryocooler head with a sample and the Hall probes in place. In this way, samples up to 4 cm in diameter and 3 cm in length can be cooled and submitted to a high magnetic field.

B. Large-sized MgB_2 samples

Three large-sized MgB_2 samples have been used in our trapped field measurements. The height h of each of them is 9.7 mm and they have been made using a SPS process with a heat treatment at $1200 \text{ }^\circ\text{C}$ under 50 MPa pressure.

One sample, called hereafter $d30$, with a diameter d of 30 mm, has been reacted during 15 min. The two others samples, named $d20$, with a diameter of 20 mm have been reacted during 10 min each.

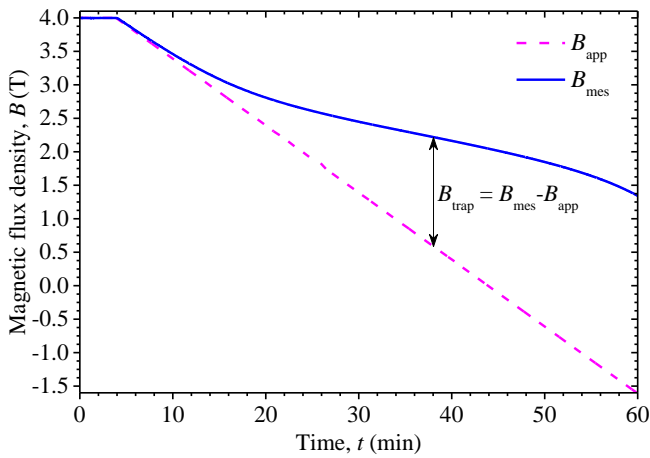


Fig. 4. Illustration of a Field Cooling (FC) process at 4 T and 20 K on the sample *d30*. The difference between the measured field B_{mes} and the applied field B_{app} is defined as the trapped magnetic field B_{trap} .

IV. TRAPPED FIELD MEASUREMENTS

In the following, we present the trapped field measurements on the large-sized bulk MgB_2 samples.

A. Field-cooling process

To activate/energize the MgB_2 pellets, we employ the Field-Cooling (FC) process. For this purpose, we tried also the use of different field sweep rates in order to see the dependence on potential flux jumps of this sweep rate. An example of FC process on the sample *d30* is illustrated in Fig. 4. Here, a magnetic field of 4 T is applied to the sample which is then cooled down below its critical temperature. The applied magnetic field is then decreased with a fixed sweep rate. The difference between the measured field B_{mes} and the applied field B_{app} is defined as the trapped magnetic field B_{trap} , this is the reaction of the sample. This definition of B_{trap} also corresponds to the magnetic polarization of the HTS sample $\mu_0 M$. The remanent trapped magnetic field B_{rem} is defined when the applied field is zero. The definition of the trapped magnetic field B_{trap} is essential, since only the remanent trapped magnetic field is usually reported. However for practical applications as in [8]–[10], higher values of B_{trap} are of great importance. Indeed, for some kinds of high power density superconducting motors, the output power is directly dependent on the magnetic flux density that can produce HTS bulks. For example, if some HTS bulks, cooled under a field of 3 T and then submitted to a negative field of -3 T, can maintain the initial value of 3 T, this means that they can generate 6 T thanks to the current density induced inside.. This is relevant value for such applications.

B. One single MgB_2 sample

In this section, we report the performances in terms of trapped magnetic field of one single MgB_2 sample.

Fig. 5 shows the magnetic field trapped at the center of the surface of the sample *d30* at 10 K and 20 K. At 20 K, there are less flux jumps when the field sweep rate is 0.10 T/min instead of 0.50 T/min. This sweep rate influence on the flux jumps has also been observed in [11]. The remanent magnetic

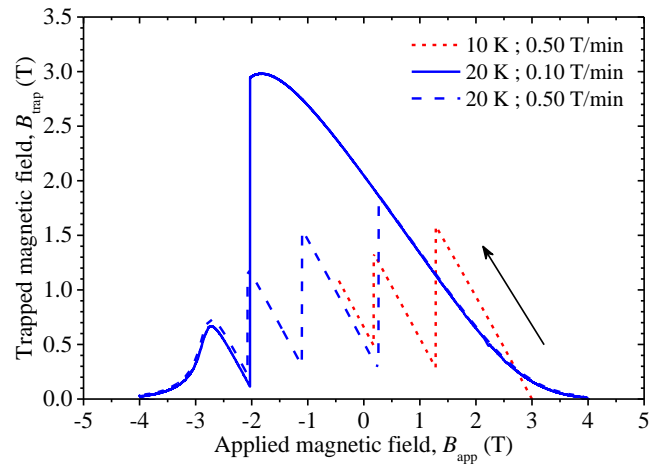


Fig. 5. Magnetic field trapped at the center of the surface of the sample *d30* at 10 K and 20 K after field cooling at 3 T or 4 T. At 20 K, there are less flux jumps when the field sweep rate is 0.10 T/min instead of 0.50 T/min.

field is equal to 2.05 T at 20 K and the highest value of trapped/produced magnetic field by this sample is around 2.98 T at 20 K. Similar values around 2 T have been recently summarized for the same sample size and at the same temperature in Table 1 of [12].

Given that many flux jumps occurred at 10 K, even at low sweep rate, it was not possible to perform high trapped field at this temperature. More details concerning the flux jumps in MgB_2 can be found in [13].

C. Two MgB_2 samples stacked together

In this section, we report the performances in terms of trapped magnetic field of two MgB_2 samples stacked together. Because we did not have two samples of 30 mm of diameter, we used samples of same properties but with a diameter of 20 mm, that is the two *d20* samples. The two *d20* pellets are separated by an air gap of the size of the Hall probe, i.e. 1 mm. By neglecting this air gap, it can be considered that the stack forms a single MgB_2 pellet of 20 mm of diameter and roughly 20 mm height, that will be later called *d20x2*.

Fig. 6 shows the trapped magnetic field results on a stack of two MgB_2 samples *d20x2* at 10 K, 20 K and 25 K (a) at the center of the surface of the stack, and (b) at the center between the two samples. As expected, the values shown in (b) are higher than the one in (a), but the shape of the curves is the same. Some flux jumps appears at 20 K with 0.50 T/min and 10 K with 0.05 T/min. Unfortunately, it was not possible to repeat measurements at 10 K with lower sweep rates in order to remove the flux jump.

Fig. 7 shows a bar graph synthetising the results of maximum trapped magnetic field using *d20x2*. The red bars (left) correspond to the measures at the center of the **top** surface of the *d20x2* assembly, whereas the black ones (right) correspond to the measures **inside** *d20x2*, in the air gap. The corresponding values are directly written above the bars. The hashed part of the bars represents the remanent trapped magnetic field B_{rem} , when the applied field is zero, whereas the full bars are associated to the maximum trapped field B_{trap} achieved in our experiments. Similar results on others samples have been reported in [14], [15]. From Fig. 1 of [14], we can

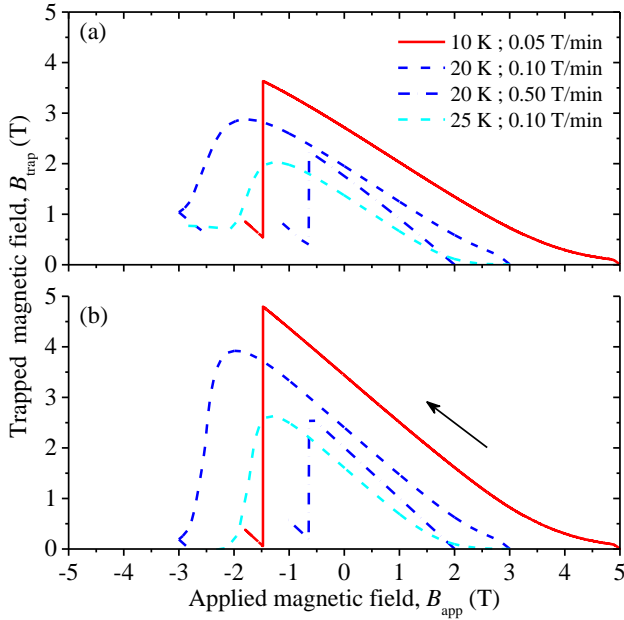


Fig. 6. Trapped magnetic field results on a stack of two MgB₂ samples *d20x2* at 10 K, 20 K and 25 K (a) at the center of the surface of the stack, (b) at the center between the two samples.

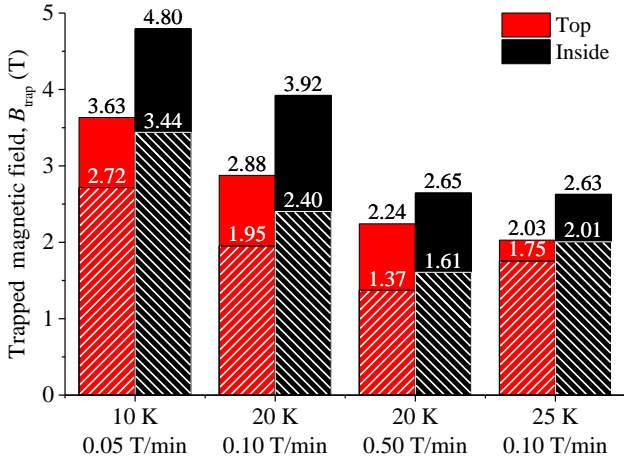


Fig. 7. Bar graph synthesizing the results of maximum trapped magnetic field using *d20x2*. The hashed part of the bars represents the remanent trapped magnetic field B_{rem} , whereas the full bars are associated to the maximum trapped field B_{trap} .

deduce the maximal trapped magnetic field B_{trap} inside the bulk disk pair: 6.25 T, 4.66 T, and 2.21 T, at 10 K, 20 K and 30 K, and under negative supporting field of -3 T, -2.23 T and -1 T, respectively. A value of B_{rem} equal to 2.60 T at 20 K has also been measured inside a disk pair in [15].

V. RESULTS AND DISCUSSION

It is obvious that the remanent magnetic field anywhere in the sample cannot be higher than the irreversibility field reported in Section II.B. The irreversibility field values of our samples prepared by SPS are clearly lower than those reported in [12] for samples prepared by hot-pressing of ball-milled precursor powder, where the irreversibility fields are about 7 T at 20 K and 2 T at 30 K.

According to [16]–[18], the external magnetic field necessary for full flux penetration at the center of the sample

TABLE I
COMPARISON OF THEORETICAL AND EXPERIMENTAL RATIO B_p / B_p^s

Ratio B_p / B_p^s	10 K		20 K		25 K	
	0.05 T/min	0.10 T/min	0.10 T/min	0.50 T/min	0.10 T/min	0.10 T/min
Theoretical	1.82	1.82	1.82	1.82	1.82	1.82
With B_{trap}^{max}	1.32	1.36	1.18	1.18	1.30	1.30
With B_{rem}	1.26	1.23	1.18	1.18	1.15	1.15

can be written as

$$B_p = \mu_0 J_c^{bulk} R f(k_d), \text{ with } f(k_d) = k_d \ln \left(\frac{1 + \sqrt{1 + k_d^2}}{k_d} \right) \quad (2)$$

with J_c^{bulk} as the field-independent bulk critical current density, R the radius, d the diameter and h the height of the sample, and $k_d = h/d$. Since B_p corresponds to the full penetration field, it gives an idea of the achievable trapped magnetic flux density at the center of the sample. However, (2) is based on the hypothesis of a constant critical current density and does not take into account the $J_c(B)$ law of the material. Using the same hypotheses, one can also express the corresponding field at the center of the surface of the sample, see (12) in [18]:

$$B_p^s = \frac{\mu_0 J_c^{bulk} R}{2} f(k_R), \text{ with } k_R = 2k_d = h/R \quad (3)$$

In that case, the ratio B_p / B_p^s is only driven by geometric parameters. For the samples studied here, this ratio is equal to 1.51, 1.63 and 1.82 for *d30*, *d20* and *d20x2*, respectively. As $\lim_{k \rightarrow \infty} f(k) = 1$, a ‘half’ infinite long cylinder would correspond to a ratio B_p / B_p^s of 2. However, extrapolating the value of the trapped magnetic field at the center of the sample, as some authors do, by multiplying the value of the trapped magnetic field at the surface with the theoretical ratio B_p / B_p^s is not sufficient, and leads to overestimated values. The comparison is made in Table I for *d20x2*. Given the fact that the only relevant value for the practical applications as permanent magnet is the trapped magnetic field at the surface of the sample, the value of the trapped field inside the sample is usually reported for comparison reasons or in order to impress.

From (2) and (3), one can say that the trapped magnetic field, for a fixed height of the sample, can be increased simply by increasing the diameter of the sample. Theoretically, choosing *d30* instead of *d20* leads to 26% and 35% of increase for B_p and B_p^s , respectively. In the same way, choosing *d20x2* instead of *d20* leads to 23% and 10% of increase for B_p and B_p^s , respectively. Therefore, the increase of the trapped magnetic field at the surface of the sample is more significant by increasing the diameter from 20 mm to 30 mm that by doubling the height of *d20*. Finally, choosing *d30* instead of *d20x2* leads to 2% and 23% of increase for B_p and B_p^s , respectively. The same tendency is reported experimentally at 20 K, the increase of the maximal trapped field from *d30* to *d20x2* is about 3%.

From (3), the field-independent bulk critical current density J_c^{bulk} can be calculated. For *d20x2* at 20 K, with 2.88 T of maximum trapped magnetic field at the surface of the sample,

J_c^{bulk} is estimated around 48 kA/cm². This value corresponds to the $J_c(B)$ curve shown in Fig. 2 at 20 K and for $B = 1.6$ T. Therefore, the mean value of trapped magnetic field in the sample volume is approximately equal to this value of 1.6 T.

The values obtained here of the remanent trapped magnetic field measured at the center of the surface of the sample are a little bit lower than highest reported up to now in [12], i.e. 4.6 T, 3.3 T and 2 T, at 15 K, 20 K and 25 K, respectively. However, the obtained values of the maximal trapped magnetic field for $d20 \times 2$ of 3.63 T at 10 K, 2.88 T at 20 K (2.98 T for $d30$) and 2.03 T at 25 K are really interesting for the intended applications.

VI. CONCLUSION

The trapped magnetic field, not only at the surface of a sample produced by SPS, but also inside a stack of two MgB₂ samples, has been deeply investigated. The useful values of trapped magnetic field obtained at 20 K at the surface of the samples are really interesting for the intended applications. According to these values, to their very low density compared to other materials and to their ease of manufacturing, MgB₂ bulks are promising materials for the applications of superconductors.

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