A trapped magnetic field of 3 T in homogeneous, bulk MgB$_2$ superconductors fabricated by a modified precursor infiltration and growth process

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Abstract
The wetting of boron with liquid magnesium is a critical factor in the synthesis of MgB$_2$ bulk superconductors by the infiltration and growth (IG) process. Poor wetting characteristics can therefore result potentially in non-uniform infiltration, formation of defects in the final sample structure and poor structural homogeneity throughout the bulk material. Here we report the fabrication of near-net-shaped MgB$_2$ bulk superconductors by a modified precursor infiltration and growth (MPIG) technique. A homogeneous bulk microstructure has subsequently been achieved via the uniform infiltration of Mg liquid by enriching pre-reacted MgB$_2$ powder within the green precursor pellet as a wetting enhancer, leading to relatively little variation in superconducting properties across the entire bulk sample. Almost identical values of trapped magnetic field of 2.12 T have been measured at 5 K at both the top and bottom surfaces of a sample fabricated by the MPIG process, confirming the uniformity of the bulk microstructure. A maximum trapped field of 3 T has been measured at 5 K at the centre of a stack of two bulk MgB$_2$ samples fabricated using this technique. A steady rise in trapped field was observed for this material with decreasing temperature down to 5 K without the occurrence of flux avalanches and with a relatively low field decay rate (1.5%/d). These properties are attributed to the presence of a fine distribution of residual Mg within the bulk microstructure generated by the MPIG processing technique.

Keywords: MgB$_2$, infiltration and growth, trapped field

(Some figures may appear in colour only in the online journal)

Introduction
The ability of a type II bulk superconductor to trap magnetic field is a direct indication of the presence of strong flux pinning, and hence high critical current density, $J_c$, within the sample microstructure. In general, the magnitude of the field trapped by a bulk superconductor is determined by the product of the sample radius, $r$, and $J_c$, assuming that the current flows homogeneously throughout the volume of the sample. As a result, superconducting ‘permanent magnets’ have the...
potential to generate magnetic fields that are significantly higher than conventional Nd-Fe-B magnets, which are limited generally to less than 2 T. MgB$_2$ superconductors, in particular, have already been used to generate trapped fields of greater than 5 T [1, 2]. Furthermore, the relatively high $T_c$ (39 K), low raw material cost, lower density, relative ease of fabrication and ability of grain boundaries to support supercurrent make this material an exciting choice for practical applications. Techniques to fabricate bulk MgB$_2$ mainly include sintering (either in situ or ex situ) and those based on an infiltration process (such as reactive liquid infiltration (RLI) or infiltration and growth (IG)), both of which offer certain advantages. In situ sintering, for example, can be used generally to produce a well-connected MgB$_2$ phase with relative ease [3], whereas the infiltration method is useful for the fabrication of near net shaped, dense MgB$_2$ bulk materials, as reported by Giunchi et al [4, 5]. This is illustrated by the closed end cavity fabricated using an infiltration technique discussed later, as shown in figure 1, which required only minimum post-processing machining to obtain a smooth inner cavity surface. A significant increase in trapped field has been reported recently for sintered MgB$_2$ bulk samples. Notably, Fuchs et al and Yamamoto et al reported high trapped fields of 5.4 T (12 K) and 3.72 T (5 K) respectively, in nano-crystalline MgB$_2$ samples [1, 6]. In addition, Yamamoto et al reported a trapped field of 4 T at 10 K in an in situ sintered MgB$_2$ sample and Naito et al obtained 4.6 T in a Ti-doped MgB$_2$ bulk at 14 K [7, 8]. Finally, Durrell et al reported 3 T at 17.5 K in a sintered, hot-uniaxially pressed MgB$_2$ bulk sample fabricated by an ex situ technique [9]. These recent data represent a significant improvement over the previous reports of the field trapping ability of MgB$_2$ of Viznichenko et al and Murlidhar et al Naito et al Perini et al and Giunchi et al [10–14]. However these levels of field have not yet been realized in samples fabricated by an infiltration technique [13–15], although samples processed by infiltration route are denser and the local critical current density in samples fabricated by the two methods is comparable [16–20].

Several existing reports [16, 21–23] and more recent studies by the current authors [19, 20] indicate that all infiltration processes generate a considerable amount of residual Mg, up to 20%, in the fully processed sample microstructure, due primarily to 25% volume shrinkage in MgB$_2$ during the phase formation process. This makes way for the surrounding liquid Mg during the primary phase formation process, assuming that MgB$_2$ nucleates everywhere simultaneously in the precursor and that the sample does not shrink overall. Giunchi et al observed the presence of residual Mg both in the form of discrete, fine inclusions between large MgB$_2$ particles and as thread-like structures (≈20 μm long) in individual MgB$_2$ particles in RLI process [16]. This is consistent with our recent studies. In addition, we also observed a large number of continuous Mg channels extending from the surface of bulk MgB$_2$ sample to its centre, as illustrated in figure 2(a) [20]. Such samples generally exhibit very low trapped fields, despite their superior local superconducting properties, since concentric current loops that form in the superconductor are impeded by these non-superconducting Mg channels. It is apparent, therefore, that the presence of residual Mg, especially in the form of continuous channels, may have an adverse effect on the trapped field performance of MgB$_2$ bulk superconductors fabricated by an IG process.

In RLI processed samples, Naito et al suggested casing the precursor with a stainless steel to curb any expansion of sample in radial direction [23]. This approach ameliorated the problem of continuous unreacted Mg in the bulk. In this work, we aim to control the formation of residual Mg channels and to fabricate homogeneous MgB$_2$ bulk superconductors by incorporating MgB$_2$ powder as wetting enhancer in the IG process. The resulting spatial variation in superconducting properties of bulk samples fabricated by this technique and the trapped fields achieved are reported.

Initial studies on enriching B powder with MgB$_2$ prior to diffusion processing were reported by Iwayama et al [24]. This approach was effective to suppress formation of voids and cracks that formed during the process, as a result of low and inhomogeneous filling density of boron powder [24–26].

**Experiment**

**Sample preparation**

Crystalline boron (98% pure, HC Starck, <38 μm) and MgB$_2$ powders (Pavezyum, <44 μm) were mixed thoroughly in a molar ratio of 90:10. The resulting powder mixture was pressed uniaxially into two cylindrical precursor pellets of

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**Figure 1.** A MgB$_2$ cavity structure of depth 6 mm and diameter 18 mm.
Magnesium was melted in a separate process and held at 700 °C in a graphite crucible. The precursor pellet, which was contained in a porous steel vessel, was transferred slowly to the crucible containing the liquid Mg. The use of the porous steel vessel ensured that no part of precursor pellet came into contact with the walls of the crucible and that it did not float on the Mg melt, which could result potentially in a non-uniform infiltration process. Column height of Mg was maintained at 10 cm above precursor to obtain a hydrostatic pressure of approximately 1.7% that of atmospheric pressure, which was found to be necessary to achieve a uniform infiltration process. The entire assembly was then subjected to the thermal profile reported in reference [20] and held at a reaction temperature of 850 °C for 4 h. A controlled, continuous flow of gas of a mixture of N₂ + SF₆ of volume ratio 95:5 was maintained over the sample during processing to minimize oxidation of Mg. SF₆ gas is known to react with Mg to form MgF₂, which fills the pores in otherwise porous MgO film on the melt surface. This forms a continuous impervious MgO/MgF₂ layer on melt surface which protects underlying Mg from oxidation [27]. After the end of thermal cycle (at 700 °C), the steel vessel containing reacted product was taken out from the melt. The excess Mg surrounding the sample after processing was removed by grinding to recover the bulk specimen (ρ ≈ 2.45 g cm⁻³). The sample was then characterized using various physical property and compositional measurement techniques. The method of fabrication used in this study, based on a composite precursor containing 10 mol% MgB₂ powder, is referred to subsequently as the modified precursor infiltration and growth (MPIG) method, whereas IG is used to describe any method that uses a precursor pressed purely from boron powder.

**Characterization**

The microstructure of the sample fabricated by IG and MPIG process was studied by scanning electron microscopy. The magnetic moment of small specimens cut from the as-processed bulk was measured using a superconducting quantum interference device (SQUID) magnetometer. The critical current densities of these samples were calculated from measured magnetic moment hysteresis loops using the extended Bean model for a rectangular cross section in a perpendicular applied magnetic field [28].

**Physical property measurements**

Two separate trapped field measurements were made, on an individual MPIG MgB₂ bulk sample, and on a two-sample stack, respectively, using the same measurement procedure in each case. Stacked arrangement was made primarily to minimize demagnetization effect, which is more severe in case of a single bulk. The measurements involved cooling down the sample or sample arrangement to 5 K under an external magnetic field of 5 T applied perpendicular to the top surface of the sample using a Gifford–McMahon cryocooler (CRTHE05-CSFM, Iwatani Gas). The external magnetic field was then reduced very slowly to zero at a rate of 1.8 T h⁻¹ to minimize any loss of trapped flux from the sample(s), as shown in figure 3. The sample(s) was then heated slowly at 0.1 K min⁻¹ and the trapped magnetic flux density was measured at temperatures up to 40 K by two cryogenic Hall sensors (HGCT-3020, Lake Shore). The Hall sensors were placed at the centre of the top and bottom surfaces of an individual bulk sample to enable the trapped field to be measured simultaneously in the first measurement. In the second measurement, one Hall probe was positioned at the centre of the top surface of the upper sample in a two sample
stack, with the second probe used to measure the trapped field between the sample pair.

Results and discussion

Role of MgB₂ in the powder precursor

Figures 2(a) and (b) show photographs of the MgB₂ samples fabricated by IG and MPIG processes, respectively. The sample fabricated by IG exhibits a number of residual Mg channels that are not present in the sample fabricated by MPIG. As a result, the former underwent significant macroscopic expansion after processing whereas the latter retained its dimensions fairly closely. Furthermore, MPIG method has also enabled fabrication of homogeneous complex cavity structure (figure 1). The formation of continuous Mg channels in IG processing are believed to form due primarily to poor wetting between B(s) and Mg(l), which is further accentuated by volume shrinkage (by up to 25%) that occurs as a consequence of the Mg(l) + 2B(s) → MgB₂(s) reaction and hydrostatic pressure exerted by Mg(l) column on to the precursor during the infiltration process. The incorporation of MgB₂ powder enhanced the overall wettability of the precursor with Mg(l), which ensured uniform influx of Mg(l) into the pores. Moreover, the presence of MgB₂ powder in the precursor is observed to provide sufficient strength to withstand the compressive hydraulic stresses exerted by the Mg liquid. Also, presence of a wetting enhancer in the precursor pellet allows the precursor powder to be packed under higher uniaxial load, which directly benefits sintering and can potentially result high density of the processed sample.

Scanning electron microscopy

Figures 4(a) and (b) show SEM images of the polished surfaces of MgB₂ bulk samples fabricated MPIG process. Figure 4(b) shows the presence of two types of MgB₂ particles. Ones with a smooth surface and dark inner core and other that appears rough and bright. The particles with a dark-in-contrast boron rich, inner core comprise of B-rich intermediate (Mg₂B₂₅) phase [20]. These are almost certainly formed during the process, while the bright-in-contrast are pre-existing, unreacted MgB₂ particles. When a boron particle undergoes a reaction to transform into MgB₂ (i.e. 2 moles of B giving 1 mole of MgB₂), the original B particle almost doubles in volume, due to associated volume expansion (MgB₂(molar volume)/2 × B(molar volume) ≈ 2). The growing MgB₂ particles then impinge upon each other and sinter simultaneously. This is evidenced by a large contact area between MgB₂ particles in the area shown by continuous contours in figures 4(a) and (b). Similar features were also observed elsewhere in the microstructure. However, such a large contact area was not observed between newly formed MgB₂ and pre-existing powder particles (dotted contours in figure 4(b)). This could be due to the fact that newly formed MgB₂ surface is free from impurities and therefore likely to sinter readily, whereas nano-scale impurities such as MgO and BO₃ are more likely to be present on the surface of pre-existing MgB₂ particles [29].

Spatial variation in MPIG samples

The uniformity of the superconducting properties throughout the volume of the MgB₂ cylinder, which is required to generate high trapped fields, was investigated by cutting nine, relatively large (4 mm × 3 mm × 2 mm) rectangular samples from various positions within the MPIG bulk. Figure 5(a) shows the normalized magnetization of these samples as a function of temperature, measured using a SQUID magnetometer. Typical magnetization data for IG-processed samples are also plotted for purposes of comparison. The measurements were made using a small, applied external magnetic field of 2 mT and normalized to the value at 30 K, with the position of the sample in the parent bulk indicated in the inset of figure 5(a). The IG processed sample exhibits a Tc of 37.50 K, whereas the samples cut from the MPIG bulk exhibit Tc’s that vary between 37.25 and 37.40 K. Also, samples cut from rows 1 and 2 of the parent MPIG bulk exhibit very similar M–T curves with a sharp transition, whereas all the samples from row 3 (bottom surface) exhibit relatively broader transitions. XRD analysis (not shown here) on MPIG bulk shows presence of MgB₂, Mg and Mg₂B₂₅ phases with similar phase fraction on both top and bottom surfaces. Such M–T behaviour could be due possibly to the fact that slightly denser MgB₂, in comparison to boron, tends to settle preferentially towards the bottom of the precursor powder prior to palletization.

Figure 5(b) shows the dependence of external magnetic field on Jc for the MPIG specimens cut from the various sample locations. The geometrical location in the parent MPIG bulk sample appears to have little effect on Jc, which does not change appreciably across the volume of the bulk investigated. All the samples in MPIG bulk show Jc (5 K, 1 T) within the close range of ±10% of average Jc. This does not correlate directly to broad transitions observed
in some of the samples (figure 5(a)). This is because, at lower temperatures (5 K in this case), difference in ratio \( T/T_c \) is small enough in each sample to leave \( J_c \) 
\[
\alpha \left( 1 - \left( \frac{T}{T_c} \right)^2 \right)^{1.5}, \quad \alpha \text{ being } J_c \text{ extrapolated to } 0 \text{ K}
\]
almost unaltered from sample to sample [30]. MPIG and IG samples exhibit similar \( J_c(B) \) dependence up to \( \sim 3 \text{ T} \), after which MPIG samples show a strong \( J_c(B) \) behaviour (except 1–1). Magneto optical studies on such bulks fabricated from pre-added MgB\(_2\) powder by Polyanskii et al reveal that MgB\(_2\) particles in microstructure have lower \( J_c \) than surrounding matrix. Origins of low \( J_c \) are reported to be weaker connectivity and lower \( H_{c2} \) that results in poor flux pinning strength [26].

These findings are consistent with SEM observations (figure 4) and \( J_c \) data reported here, which shows reduced \( J_c \) in MPIG bulk sample compared to IG sample. This suggests that the inter-grain electrical coupling between newly formed MgB\(_2\) particles (from boron during the processing) and pre-existing MgB\(_2\) particles in the MPIG process is probably inferior compared to the coupling between two newly formed MgB\(_2\) particles. These boundaries, therefore, probably form weak links at higher fields resulting in reduced \( J_c \).

**Trapped field measurements**

The measured trapped field as a function of temperature for a single bulk and a stacked pair of MPIG processed bulk samples is shown in figures 6(a) and (b), respectively. A difference of less than 0.5% in the magnitude of trapped field recorded at the top and bottom surface of the single bulk confirms its homogeneity, given that any cracks, porosity or non-superconducting regions would impede the flow of supercurrent throughout the volume of the bulk. These measurements are consistent with narrow range of \( J_c \) (figure 5(b)) observed in the MPIG bulk. Although sample 3–3 (centre of
bottom surface, figure 5) shows broad transition and low $J_c$ amongst all samples, it is not likely to alter trapped field performance of MPIG bulk greatly. This is because the trapped field in the bulk is mainly governed by low field $J_c$ in the outer regions of the bulk, as the current loop is largest. Recently, numerical simulations, based on a novel 2D axi-symmetric $H$-formulation, have been performed for single a MPIG bulk sample using $J_c(B)$ data of single sub-specimen as an input for the model [31]. The model assumed, essentially, that the $J_c(B)$ characteristics of the small specimen extended over entire volume of the bulk sample. The results of the simulation reproduced the experimental results extremely well, confirming the homogeneity of the samples fabricated by the MPIG technique. Maximum trapped fields of 2.12 and 3 T were recorded for a single bulk and stacked pair of bulk samples, respectively. At 20 K, temperature that can easily achieved with cryogen free cost effective cryo-coolers, 1.35 and 2 T trapped field was recorded in a single bulk and centre of stack, respectively.

In comparison, for single bulk at 20 K, RLI processed sample trapped 1.1 T (55 mm dia, 15 mm thick) [13]. Yamamoto et al reported 1.9 T (30 mm dia, 10 mm thick) and Muralidhar et al reported 1.4 T (20 mm dia, 7 mm thick) in \textit{in situ} sintered samples [11, 32]. High isostatically pressured samples showed 2 T (23 mm dia, 24 mm thick) [33]. Nano-structured bulk trapped 3.2 T (20 mm dia, 8 mm thick) and 2.2 T (30 mm dia, 10 mm thick) as reported by Fuchs et al and Sugino et al [1, 6]. Similarly Naito et al reported 2.8 T (40 mm dia, 20 mm thick) in Ti-doped bulk MgB$_2$ sample [8]. Whereas trapped field at the centre of 2 sample stack is reported 2.6 T in \textit{ex situ} hot pressed, 2.9 T in \textit{in situ} sintered and 3.6 T in Ti-doped MgB$_2$ bulk samples [7–9].

The Biot–Savart law suggests that trapped field is directly proportional to $J_c$ and a dimensionless geometrical factor $K$, which depends on the radius of bulk sample;

$$B_T = \mu_0 \cdot J_c \cdot r \cdot k,$$  \hspace{1cm} (1)

where $t$ and $r$ are the thickness and radius of the sample, respectively [34]. Assuming $J_c$ to be the same for both samples, therefore, any change in trapped field can be attributed to a variation in $k$. The ratio of fields measured at the centre to the top of the two sample stack, $B_{(centre)}/B_{(top)}$, is 1.36 while predicted value (from equations (1) to (2)) is 1.56 (a difference of 15%). This deviation from the measured values is due to the fact that the Biot–Savart law assumes constant current density everywhere in the bulk sample whereas, in reality, central regions of the bulk are likely to have reduced $J_c$ due to the relatively strong $J_c$–B dependence for MgB$_2$. It is also interesting to note that the magnitude of trapped field at the top of the stack (2.2 T) is similar to that observed at the top of a single bulk, suggesting that the increase in thickness of the MgB$_2$ sample contributes relatively little to the trapped field (i.e. the sample thickness of the single bulk has already reached the trapped field saturation limit). Significantly, no flux avalanches were observed to occur in the MPIG samples down to a minimum measurement temperature of 5 K, despite a relatively high trapped field.

The trapped field in the bulk MPIG samples fabricated in the current study is likely to be limited by moderate intrinsic values of $J_c$, at least compared to recently reported values of the order of MA/cm$^2$ in bulk MgB$_2$ fabricated by other processes [1, 35], and a strong $M$–$H$ dependence. This also explains why only a 36% increase in the trapped field was observed at the centre of the two-sample stack compared to a single bulk.

**Flux creep**

The stability of trapped magnetic flux in the MgB$_2$ bulk samples was investigated further by performing additional magnetic moment measurements at 10 K following field
cooling at 5 T. Figure 7(a) shows variation of the trapped field with time for a single MgB₂ disc of diameter 32 mm. The trapped field after initial magnetization was measured to be 1.94 T, which decreased subsequently to 1.92 T (1% decay) and 1.91 T (1.5% decay), after 1 h and 1 d, respectively.

This behaviour is characteristic of logarithmic flux creep in type II superconductors (i.e. high initially and then decreasing rapidly to almost a linear variation after ~2 min) [36]. The flux creep behaviour of the sample measured in figure 7(a) can be expressed by the following equation:

\[ \frac{B(T)}{B(t(0))} = -0.004 \log(t) + 1 \quad (\text{For } t > 100 \text{ s}) \]  

The rate of loss of flux for these samples is relatively high compared to recent report by Yamamoto et al where the negative slope of \( B(T) \) (normalized) versus \( \log(t) \) plot was as low as \( 4 \times 10^{-6} \) [7].

During trapped field measurements, especially at low temperatures, dissipative flux motion can result in sudden local temperature rise. If this heat is not conducted away quickly enough, then it can potentially result in loss of trapped magnetic flux. Thus higher thermal conductivity is highly desirable in these quasi-permanent superconducting magnets [39].

Figure 7(b) describes measured thermal conductivity for MPIG bulk. Mg and sintered MgB₂ data are also plotted for a comparison. It is evident that thermal conductivity of MPIG bulk is significantly higher than a sintered bulk measured within 5–100 K. This is attributed to uniform distribution fine Mg grains in the inter-particle region (figure 7(a) (inset)) that possess orders of magnitude higher thermal conductivity than MgB₂. Detail SEM observation and EDS analysis suggests that such inter-particle regions (figure 7(a) inset) comprise of both MgB₂ and Mg grains.

As a result, it can be seen that thermal stability of samples has been increased and no flux avalanches were observed down town to 5 K and magnetized bulk showed low trapped field decay rate.

**Conclusion**

Near-net shaped MgB₂ bulk superconductors have been fabricated by a MPIG method, incorporating MgB₂ powder in the precursor as wetting enhancer to facilitate in-flux of Mg, leading to a more uniform infiltration process. Almost identical value of trapped magnetic flux was measured at the top and bottom surfaces of a single bulk sample suggest that MgB₂ bulk samples fabricated by MPIG method are homogeneous. A stack of two MgB₂ bulk samples trapped a field of 3 T at the centre of the stack at 5 K. These values of trapped field are the highest obtained for single bulk MgB₂ sample or a combination of bulk samples fabricated by an infiltration growth process reported to date. Although, A moderate \( J_c \) and a strong \( J_c(B) \) dependence limits the overall trapped field performance of MgB₂ bulk superconductors fabricated by MPIG. The introduction of additional pinning centres to improve \( J_c \) in an applied magnetic field, therefore, is necessary for further improvement in trapped field performance of MgB₂ bulk fabricated by this technique. Finally, this study has demonstrated clearly that the IG method has significant potential for fabrication high quality, quasi-permanent MgB₂ magnets for high field applications.

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