

Evidence of Point Pinning Centers in Un-Doped MgB₂ Wires at 20 K after HIP Process

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Abstract

In this paper we present results of transport critical current density (J_c) at 20 K and 4.2 K, irreversible magnetic field (B_{irr}), upper critical field (B_{c2}), critical temperature (T_c), pinning force (F_p), scanning pinning force scaling results (F_p/F_{pmax} and B/B_{irr}) and electron microscope (SEM) images of un-doped MgB₂ wires of 0.63 mm diameter. All wires were annealed at pressures ranging from 0.1 MPa to 1 GPa for 15 min between 680°C to 740°C. SEM images show that 1 GPa pressure yields small grains, higher MgB₂ material density, and small voids. The results obtained by a physical properties measurement system (PPMS) show that high pressure (1 GPa) and 700°C annealing slightly decreases T_c above 27 K and increases T_c and B_{irr} below 25 K. Un-doped MgB₂ wire annealed in 1 GPa for 15 min at 680°C has a 20 K, 4.5 T J_c of 100 A/mm² in and a B_{irr} of 7 T. At 4.2 K, this wire has J_c of 100 A/mm² at 10.5 T. Scaling results show that the dominant pinning mechanism is point pinning for undoped MgB₂ wires under 1 GPa pressure and annealed at 680°C (at 20 K).

Keywords: Un-doped MgB₂ wires; Critical current density; Point pinning centers

Introduction

MgB₂ superconductors has many advantages, namely high critical temperature (39 K) [1], low resistivity, simple structure, low anisotropy and high critical field [2,3]. In addition, this superconductor can be manufactured using inexpensive components with a relatively simple powder in tube (PIT) methodology. The high critical temperature of MgB₂ enables these superconductors to operate using liquid hydrogen or cryocoolers [4]. This significantly reduces the cost of the use and application of these wires.

MgB₂ in un-doped form has several weaknesses which inhibit its use: lack of sufficient point pinning centers (normal areas of thickness similar to the length of coherence and dislocations) needed for high J_c in medium and high magnetic fields [5], and large voids with an inhomogeneous distribution and weak connections between grains [6]. Studies have indicated that the dominant pinning mechanism in un-doped MgB₂ superconductors is surface pinning [7,8], which effectively increase J_c in low magnetic fields. Studies suggest that the point pinning mechanism (small SiC particle size) can effectively increase the J_c at 20 K [9]. Livingston indicates that point pinning centers can create dislocations and voids of thickness similar to the coherence length [5]. Our previous research for doped MgB₂ wires shows that the disadvantages of un-doped MgB₂ can be eliminated by annealing a wire under isostatic pressure (HIP) [10-12]. HIP creates dislocations, eliminates voids, produces small grains and small normal area, increases connections between grains, and increases the density and homogeneity of the MgB₂ material [13,14].

We show that the HIP process produces a dominant point pinning mechanism in un-doped MgB₂ wires, leading to an increase in J_c and F_p at 20 K.

Preparation of Samples

Wires were made at Hyper Tech Research using a continuous tube forming and filling (CTFF) process [15]. The MgB₂ wires comprised

of amorphous B (99B) with a Mg to B ratio of 1.1:2, Nb barrier and eighteen filaments. The wires were fabricated to a diameter of 0.63 mm, achieving a fill factor of 15%. All wires were annealed in isostatic pressure at the Institute of High Pressure Research in Warsaw [16]. The HIP was a two-step process: isostatic pressure is first applied and then the wire sample is ramped to the set annealing temperature. The HIP process ends by decreasing the annealing temperature to room temperature before decreasing the isostatic pressure. Samples were annealed at a temperature range from 680°C to 740°C for 15 minutes at pressures between 0.1 MPa to 1 GPa (Table 1). The HIP was performed in 5N argon atmosphere in a high gas pressure chamber. The transport critical current (I_c) of the MgB₂ wires was measured with the four-probe resistive method at 4.2 K up to 150 A in a perpendicular magnetic field at the International Laboratory of High Magnetic Fields and Low Temperatures in Wrocław [17,18]. Transport critical current measurements at 20 K were conducted at the Institute for Solid State and Materials Research Dresden [19]. The I_c was determined on the basis of 1 μ V/cm criterion. The critical temperature and critical magnetic fields were measured using the four-probe resistive method using a physical properties measurement system (PPMS) for 100 mA and 15 Hz. T_c , B_{irr} and B_{c2} were determined with criterion of 50%, 10% and 90% of the normal state resistance. Analysis of the microstructure was performed using a Zeiss microscope (high resolution low-energy type) at the Institute of High Pressure Physics PAS in Warsaw and FEI

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Nova Nano SEM 230 SEM in the Institute of Structural Research and Low Temperatures in Wrocław PAS.

Research Results

EDAX studies on Figure 1a show that their wires are made from material of high purity. The small amount of oxygen formed during sample preparation for SEM analysis. In addition, research on Figure 1b show that the Nb barrier is uniformly distributed in the wire and do not have cracks. SEM images in Figure 2a, 2b and 3a show that annealing at low pressure produces numerous large voids (about 1µm) and large grains (agglomerates as large as 400 nm). Moreover, 0.1 MPa pressure decreases the MgB₂ material density and creates

Sample Identifier	HIP annealing time, temperature and pressure		
	T [°C]	t [min]	p [Pa]
A	700	15	0.1 M
B	700	15	0.4 G
C	700	15	1 G
Dss	680	15	1 G
E	725	15	1 G
F	740	15	1 G

Table 1: The parameters of HIP process for wire MgB₂

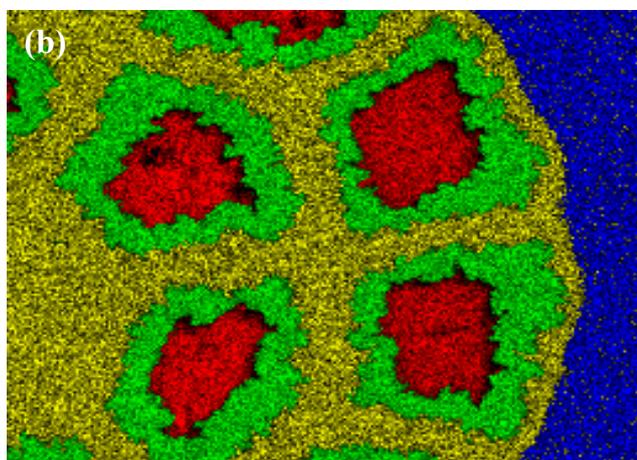
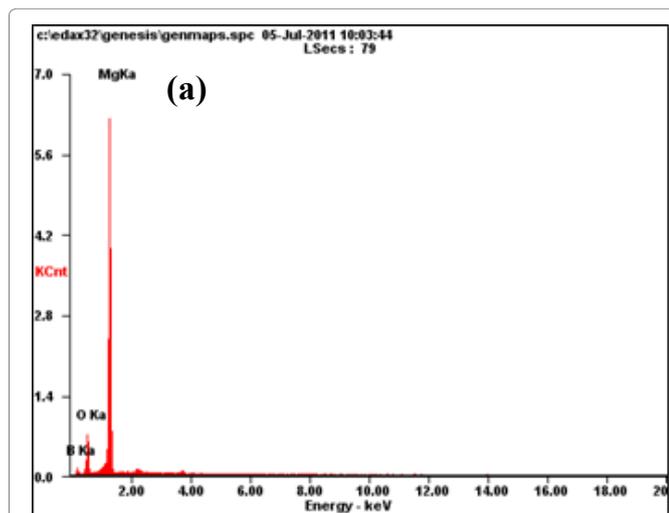


Figure 1: (a) EDAX analysis for sample C and (b) EDX maps for a cross-section undoped MgB₂ wire (sample C). The red color means magnesium, green - Nb barrier, yellow-copper, navy blue-Monel shield.

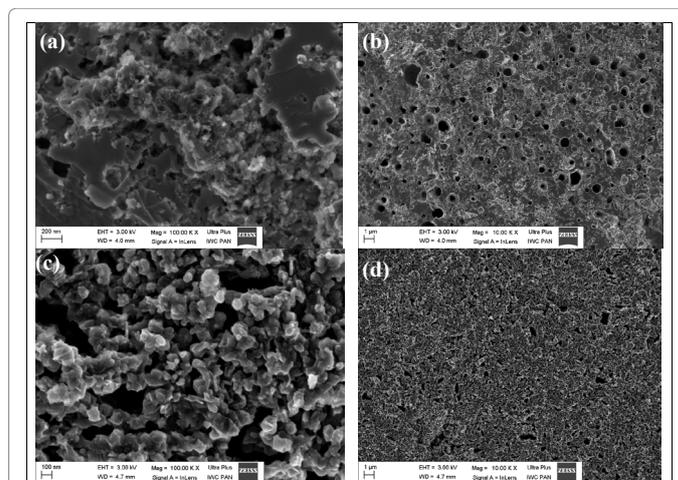


Figure 2: The SEM photographs-the cross section of undoped MgB₂ wires (a) and (b) sample A-0.1 MPa 700°C 15 min, (c) and (d) sample C- 1 GPa 700°C 15 min.

an inhomogeneous distribution of voids. This structure reduces the number of connections between the grains. On the other hand, high pressure (sample C) produces small grains (from 50 nm to 100 nm), a reduced number and size and better distribution of voids, and higher MgB₂ material density (Figure 2c, 2d and 3b). High pressure increases the number of connections between grains. The results in Figure 4a and 4b show the structure of sample D (680°C, 1 GPa). These results show that low temperature annealing produces small grains (50-100 nm), a small quantity and size of voids and higher MgB₂ material density. On the other hand, annealing at high temperature (sample F, 740°C, 1 GPa) produces larger grains (200 nm), an increased number and size of voids (1 µm) and lower MgB₂ material density, Figure 4c and 4d. These factors reduce the number of connections between grains. The formation of MgB₂ in the samples C and D was a solid state reaction of Mg and B, because small voids, small grains and high MgB₂ material density was observed. This is corroborated by [20, 21], which indicate that in 1 GPa pressure, magnesium transforms to the liquid state above a temperature of 725°C, during which the reaction of Mg is in the solid state and the shrinkage of Mg is about 5% [20]. The small amount of shrinkage yields a small amount of small voids. On the other hand, the reaction in samples A (700°C and 0.1 MPa) and F (740°C and 1 GPa) indicate that was the liquid state of Mg and solid state of B, because numerous large-sized voids and large grains (agglomerates) are observed in the SEM images. The MgB₂ reaction in liquid state of Mg and solid state of B produces a large shrinkage rate of about 25% (many voids of large size) [20-22]. Mg is in a liquid state at 0.1 MPa pressure and 650°C or at 1 GPa and 725°C [20].

Results in Figure 5a show that an increase of pressure from 0.1 MPa to 1 GPa at 700°C increases T_c between 10 K and 27 K and decreases T_c between 27 K and 35 K. In contrast, the increase of annealing temperature from 680°C to 740°C under 1 GPa, does not change T_c between 10 K and 35 K. Previous work indicates that pressure decreases the T_c of MgB₂ [23-25]. Monteverde, Lorenz and Bordet independently claimed that decrease of T_c may result from increasing phonon frequency with applied pressure, with broadens the density of state and reduces it on the Fermi surface. The decrease of T_c of a wire under pressure during reaction would then be related to the loss of p_{xy} holes, the decrease of lattice parameters and of c/a ratio (this effect can create dislocation). On the other hand, Serquis indicated that the annealing in isostatic pressure can increase the density of the MgB₂ material, which

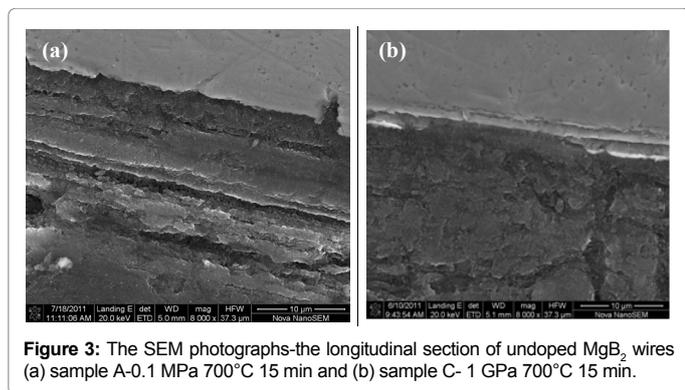


Figure 3: The SEM photographs-the longitudinal section of undoped MgB₂ wires (a) sample A-0.1 MPa 700°C 15 min and (b) sample C- 1 GPa 700°C 15 min.

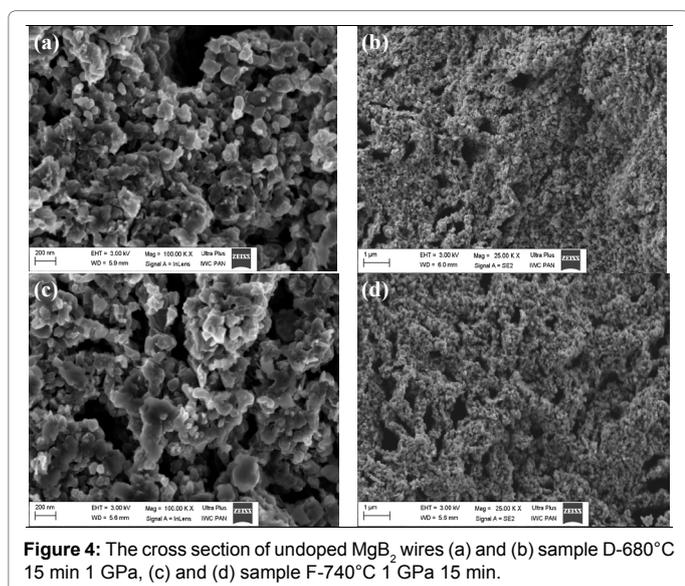


Figure 4: The cross section of undoped MgB₂ wires (a) and (b) sample D-680°C 15 min 1 GPa, (c) and (d) sample F-740°C 1 GPa 15 min.

also produces small grains, and create dislocations [13]. In another article, Serquis showed that an increase of strain (dislocation) in MgB₂ decreases T_c [26]. These results indicate that in the pressure range from 0.1 MPa to 1 GPa, the dislocations have a decisive influence on T_c . The increase of pressure increases the dislocation density, explaining why T_c is reduced above 27 K and increased below 27 K for sample C (1 GPa). Buzea suggested that the shrinkage of the MgB₂ crystal unit cell decreases T_c by 1 K [27]. Shrinkage occurs during annealing at 740°C and 1 GPa when Mg is in the liquid state of and B in solid state. Shrinkage increases the dislocation density. A small change of T_c caused by shrinkage may explain why T_c does not change with increasing annealing temperature (Figure 5b). Figure 6a shows that increase of pressure from 0.1 MPa to 1 GPa does not change B_{c2} between 10 K and 25 K, and slightly decreases B_{c2} above 25 K. By contrast, the increase of annealing temperature from 680°C to 740°C does not change the value of B_{c2} . Small changes of B_{c2} indicate that it is slightly dependent on pressure and annealing temperature. This suggests that the starting composition of the material may influence B_{c2} . The results in Figure 6a show that 1 GPa pressure can increase B_{irr} from 10 K to 27 K, and slightly reduce B_{irr} above 27 K. By contrast, an increase in annealing temperature (Figure 6b) reduces B_{irr} in between 15 K to 25 K and does not change B_{irr} from 10 K to 15 K and above 25 K. Maeda and Susner claim that the value of the B_{irr} is dependent on dislocation [28, 29]. Moreover, Serquis showed that the HIP process can increase the density of dislocations [13]. Therefore the increase of B_{irr} in this study

occurred because of increased dislocation density. Our research shows that B_{irr} at 20 K increases most significantly during the reaction at 680°C in 1GPa (solid state Mg and B).

The results in Figures 7a and 7c show that the increase of pressure from 0.1 MPa to 1 GPa significantly increases the critical current density (J_c) and pinning force (F_p) at 4.2 K by a factor of two between 2 T to 7 T and a factor of three above 7 T. Moreover, these results show that the increase of pressure does not shift the maximum pinning force at 3 T ($F_{p,max}$). At 20 K, the increase of pressure from 0.1 MPa to 1 GPa nearly doubles J_c and F_p between 2 T and 4 T but the higher pressure does not increase J_c and F_p above 4 T. Moreover, Figure 7b shows that the increase of the pressure does not shift the maximum pinning force at 2.5 T ($F_{p,max}$). The results in Figure 7a indicate that J_c at 20 K is three times lower than the J_c at 4.2 K. The results in Figure 7d show that the increase of the annealing temperature from 680°C to 700°C in 1 GPa reduces J_c at 4.2 K by almost one half between 2 T and 8 T and by one third above 8 T. At 20 K, the increase of annealing temperature (from 680°C to 700°C) causes an increase of J_c and F_p of about one third between 1.5 T to 3.5 T and a reduction of J_c and F_p of about one half above 3.5 T. Further, increasing the annealing temperature (from 700°C to 740°C) practically does not change J_c at 4.2 K. At 20 K, the increase of annealing temperature from 700°C to 725°C does not change J_c and F_p . However, the results in Figure 7d and 7e indicate that an increase of annealing temperature from 725°C to 740°C at 20 K causes a reduction of J_c by four times between 1 T and 5.3 T and a slight increase of J_c and F_p above 5.3 T. Scaling results (Figure 8a) indicate that the increase of pressure from 0.1 MP to 1 GPa at 4.2 K and 20 K does not change the dominant pinning mechanism and does not shift k_{max} ($k = F_p/F_{p,max} = 1$). However, the increase of temperature

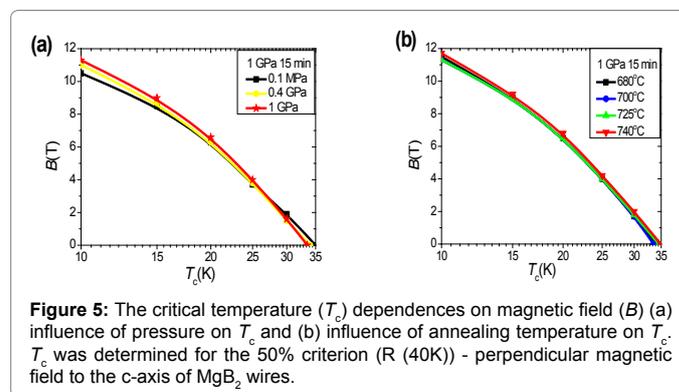


Figure 5: The critical temperature (T_c) dependences on magnetic field (B) (a) influence of pressure on T_c and (b) influence of annealing temperature on T_c . T_c was determined for the 50% criterion ($R(40K)$) - perpendicular magnetic field to the c-axis of MgB₂ wires.

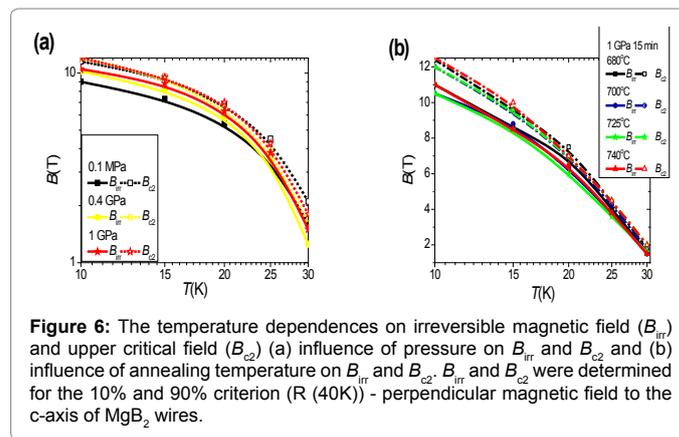


Figure 6: The temperature dependences on irreversible magnetic field (B_{irr}) and upper critical field (B_{c2}) (a) influence of pressure on B_{irr} and B_{c2} and (b) influence of annealing temperature on B_{irr} and B_{c2} . B_{irr} and B_{c2} were determined for the 10% and 90% criterion ($R(40K)$) - perpendicular magnetic field to the c-axis of MgB₂ wires.

from 4.2 K to 20 K shifts k_{\max} from h (B/B_{irr}) 0.19 to 0.31, and obtains the dominant point pinning mechanism in the range of k from $h = 0$ to $h = 0.31$. In samples A and C at 4.2 K and 20 K we see significant reduction of k above k_{\max} for $h = 0.19$ and $h = 0.31$. At 4.2 K, the increase of annealing temperature from 680°C to 700°C does not shift k_{\max} ($h = 0.2$) and significantly reduces k above k_{\max} ($h = 0.2$). At 20 K, the increase of annealing temperature from 680°C to 700°C shifts k_{\max} from $h = 0.36$ to $h = 0.31$ and slightly reduces the value of k above k_{\max} ($h = 0.36$ and $h = 0.31$). The increase of the temperature from 4.2 K to 20 K leads to a change in the dominant pinning mechanism from surface to point (Figure 8b). The results in Figure 8b show that annealing at 680°C and 740°C produces a dominant point pinning mechanism at 20 K.

Discussion

Results of SEM and scaling (no change of the dominant pinning mechanism, (Figure 8a) indicate that the increase of J_c at 4.2 K for sample C (1 GPa) is due to smaller grains, a larger number of connections between grains, greater uniformity of MgB₂ material, and an increase of the pinning force density. The increase of J_c in all magnetic fields indicates that 1 GPa increases both point and surface pinning force densities. The large increase of J_c in high magnetic fields indicates that 1 GPa produced more point pinning centers in this regime. At 4.2 K for the sample annealed in 0.1 MPa and 1 GPa (Figure 8a) the dominant pinning forces are at the grain boundary surfaces.

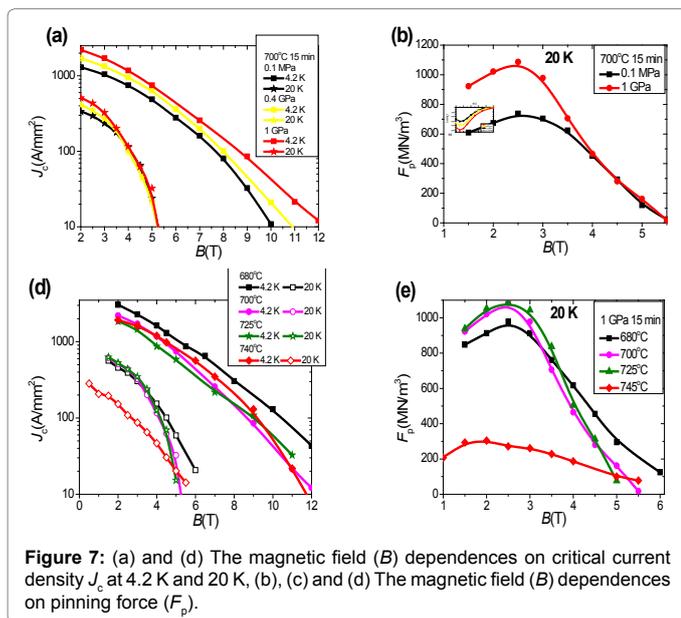


Figure 7: (a) and (d) The magnetic field (B) dependences on critical current density J_c at 4.2 K and 20 K, (b), (c) and (e) The magnetic field (B) dependences on pinning force (F_p).

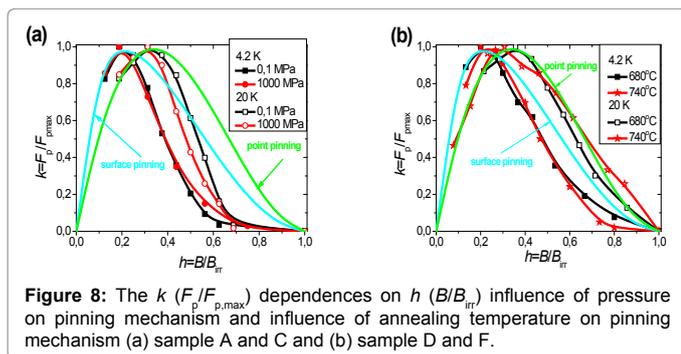


Figure 8: The k ($F_p/F_{p,\max}$) dependences on h (B/B_{irr}) influence of pressure on pinning mechanism and influence of annealing temperature on pinning mechanism (a) sample A and C and (b) sample D and F.

The observed significant decrease of J_c at 20 K as compared to 4.2 K indicates that surface pinning centers have less impact at 20 K. Moreover, the increase of temperature from 4.2 K to 20 K may also eliminate (turn off) weak connections between grains. Ghorbani suggests that pinning mechanisms are dependent on temperature and magnetic field [30]. In addition, the increase of the temperature (from 4.2 K to 20 K) increases the size of the vortices core. These results indicate that the increase of temperature and magnetic fields may vary pinning centers, e.g., from point to surface, surface to volume, and may connect several dislocations in one dislocation cluster. The increase of temperature to a small extent causes loss of dislocations in ceramic materials. Scaling in Figure 8a indicates that increase of J_c at 20 K after the HIP in 1 GPa in range of ratio h from 0 to 0.31 (0 T to 4 T) corresponds to more point pinning centers, because the curves of samples A and C coincide with the curve of the dominant point pinning mechanism. These pinning centers can create voids and dislocation cluster with a thickness similar to the coherence length. Livingston shows that dislocations create line pinning centers [5]. Dam show that the structure of the superconducting materials may be screw and edge dislocations [31]. In addition, they indicate that both types of dislocations the same anchor vortex. This indicates that the distribution of dislocations is very important. Studies indicate that dislocations create pinning centers, which enhance J_c in high magnetic fields [32, 33]. The results show that the increase of pressure from 0.1 MPa to 1 GPa causes a slight increase of J_c at 20 K above 4 T. We believe that this may be due to the linking of few dislocations (at 20 K) in regions with a thickness similar to the coherence length or slightly larger.

The HIP process had been demonstrated to increase dislocation density [13]. The annealing of MgB₂ material itself creates dislocations, e.g., the unit volume of Mg and B is reduced by 5% after the solid state reaction of Mg and B [20], and by 25% after the reaction of Mg in liquid state and B in solid-state [22]. The difference between the two mechanisms lies in the fact that HIP produces a uniform distribution of dislocations and shrinkage (reduction of the unit volume) rather causes an inhomogeneous distribution of dislocations. The inhomogeneous distribution of voids is evident in Figures 2, 3 and 4.

Studies have shown that annealing in atmospheric pressure (0.1 MPa) at 700°C (liquid state of Mg) creates many large voids and grains indicating significant shrinking in the MgB₂ material and thereby creating a lot of strain (dislocation). These dislocations feature higher density near voids, creating inhomogeneity in the MgB₂ material. These factors reduce and limit physical and mechanical parameters.

Our measurements show that the increase in annealing temperature (680°C to 740°C) for 1 GPa increases the size of grains and voids, increases dislocation density by shrinking, and decreases the number of connections between the grains (Figure 4). This leads to a reduction of J_c at 4.2 K. These results indicate that the inhomogeneous distribution of the dislocations does not increase J_c in high magnetic fields. Studies show that surface pinning centers are poor at 20 K, because increasing the temperature from 4.2 K to 20 K significantly decreases J_c (Figure 8b).

At 20 K, increasing the annealing temperature (680°C to 740°C) decreases J_c by three times. Sample D, annealed at 680°C, exhibits uniform distribution dislocations, small grains (Figure 2), a large number of connections between the grains and a very small number of small voids (Figure 2), for which the latter produces a dominant point pinning mechanism. This leads to an increase of J_c at 20 K above 4 T, achieving 100 A/mm² at 4.5 T.

Sample F, annealed at 740°C, exhibits large grains, numerous large voids (Figure 4), more dislocation (HIP and shrinking) of inhomogeneous distribution. This leads to a reduction in the amount of connections between the grains and decreases J_c at 20 K compared to sample D (680°C). The reduction of the number of connections between grains also suggests scaling because the curves of samples D (680°C) and F (740°C) are the same (Figure 8b) at 20 K.

Annealing in the temperature range from 700°C to 725°C creates pinning centers, which increases J_c at 20 K in low and middle magnetic fields. The results of scaling indicate that this increase causes point pinning centers (curves of samples D and F coincides with the curve of the dominant point pinning mechanism). These pinning centers create void and dislocation clusters with a thickness similar to coherence length.

Un-doped MgB₂ superconductors fabricated using the PIT method by other groups have a 100 A/mm² J_c at 8 T, 4.2 K and at 4 T, 20 K [34-36]. Our research shows that the HIP can increase superconductivity properties, e.g., pushing the 100 A/mm² J_c at 4.2 K to 10.5 T and at 20 K to 4.5 T.

Conclusions

Our research shows that annealing in high pressure of 1 GPa and 680°C (solid state of Mg) can significantly increase the uniformity and density of the MgB₂ material, eliminate voids and increase the number of connections between the grains. The HIP process produces a uniform distribution of the dislocations. These factors slightly decrease T_c and increase B_{irr} between 10 K and 27 K and increase J_c and F_p at 4.2 K and 20 K.

On the other hand, annealing at 740°C (1 GPa) and 700°C (0.1 MPa) (liquid state of Mg) leads to increasing the grain size, an inhomogeneous distribution of dislocations and a large amount of large voids. These factors limit intergranular connectivity and lead to a reduction of J_c and F_p at 4.2 K and at 20 K.

Our results indicate that the distribution of the dislocations can have a crucial impact on the J_c at 20 K. Reaction in the solid state of Mg and B produces a more uniform distribution of the dislocations whereas the reaction in liquid state of Mg introduces an inhomogeneous distribution of dislocations.

Studies show that the HIP process generates a dominant point pinning mechanism and more point pinning centers in un-doped MgB₂ wires. These pinning centers can create voids and dislocation clusters with a thickness similar to the dislocation and coherence length. Our studies indicate that the dominant pinning mechanism can change depending on the temperature and magnetic field. Since surface pinning centers are not effective at 20 K, increasing the operating temperature from 4.2 K to 20 K significantly reduces J_c and F_p . The HIP process produces high J_c at 4.2 K (10.5 T-100 A/mm²) and 20 K (4.5 T-100 A/mm²).

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