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High field pinning centers in MgB_2 doped with 2 at. % carbon under a low and a high hot isostatic pressures have been investigated by transport measurements. The field dependence of the transport critical current density was analyzed within the different pinning mechanisms: surface pinning, point pinning, and pinning due to spatial variation in the Ginzburg-Landau parameter ($\Delta\kappa$ pinning). Research indicates that a pressure of 1 GPa allows similar pinning centers to $\Delta\kappa$ pinning centers to be obtained. This pinning is very important, because it makes it possible to increase the critical current density in high magnetic fields at 20 K and 25 K. Our results indicate that the δT_c and δl pinning mechanisms, which are due to a spatial variation in the critical temperature (T_c) and the mean free path, l, respectively, create dislocations. The high density of dislocations with inhomogeneous distribution in the structure of the superconducting material creates the δl pinning mechanism. The low density of dislocations with inhomogeneous distribution creates the δT_c pinning mechanism. Research indicates that the hot isostatic pressure process makes it possible to obtain a high dislocation density with a homogeneous distribution. This allows us to obtain the δT_c pinning mechanism in MgB_2 wires. In addition, a high pressure increases the crossover field from the single vortex to the small vortex bundle regime (B_{sb}) and improves the δT_c pinning mechanism. Our research has proved that a high pressure significantly increases the crossover field from the small bundle to the thermal regime (B_{th}), with only a modest decrease in T_c of 1.5 K, decreases the thermal fluctuations, increases the irreversibility magnetic field (B_{irr}) and the upper critical field (B_{c2}) in the temperature range from 4.2 K to 25 K, and reduces B_{irr} and B_{c2} above 25 K. *Published by AIP Publishing*. [http://dx.doi.org/10.1063/1.4962399]

I. INTRODUCTION

In a superconducting material in the mixed state, vortices appear. The Lorentz force causes motion of these vortices, which, in turn, reduce the critical current density (J_c). These vortices can be anchored by using pinning centers, leading to an increase in J_c. The pinning centers create normal conducting areas, weak superconducting areas, and inclusions. Livingston[1] and Wang *et al.*[2] identified four types of pinning centers: volume pinning centers (e.g., big voids), surface pinning centers (grain boundaries), point pinning centers (e.g., precipitates with sizes near the coherence length), and line pinning centers (dislocations). The critical current density in the superconducting ceramic materials also depends on the connections between the grains.

Currently, we have several models for analyzing the dominant pinning mechanism, e.g., Dew-Hughes,[3] identification of the δT_c and δl pinning mechanisms,[4] and Higuchi *et al.*[5] Dew-Hughes proposed several formulas for various pinning mechanisms, e.g., point ($h(1-h)^2$—e.g., precipitates and voids of about the coherence length in size and dislocations), surface ($h^{0.5}(1-h)^2$—grain boundaries), and volume ($h^0(1-h)^2$—large voids and precipitates), where h is the magnetic field normalized by the irreversibility field (B_{irr}). Research indicates that the dislocations create high field pinning centers, because they increase the critical current density in high magnetic fields.[6,7] Currently, we have two types of edge and screw dislocations. Dam *et al.* proposed that both types of dislocations anchor vortices in the same way.[8] The results reported in the literature indicate that the surface and volume pinning increase the critical current density (J_c) in low magnetic fields, while the point pinning centers increase J_c in the middle and high magnetic fields.[9]

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The δl pinning mechanism is associated with charge carrier mean free path variations, and the δT_c pinning mechanism is linked to randomly distributed spatial variations in the transition temperature.[9] Magnetic measurements reported by Motaman *et al.*[10] and Ghorbani *et al.*[9,11] indicate that the δl pinning mechanism is dominant at low temperature, and the δT_c pinning mechanism is dominant at high temperature. In addition, studies indicate that an undoped MgB_2 material has δT_c as its dominant pinning mechanism and that a doped MgB_2 material has δl as its dominant pinning mechanism.[2,12] The effectiveness of these pinning centers is the greatest for a high Ginzburg-Landau (κ) parameter. Higuchi *et al.* proposed three types of pinning mechanisms, e.g., surface pinning, point pinning, and $\Delta\kappa$ pinning.[5,13] The results in the literature suggest that the surface pinning centers increase the critical current density in the low magnetic fields. On the other hand, the point pinning centers and the $\Delta\kappa$ pinning increase J_c in the middle and high magnetic fields.

High field pinning centers (dislocations) can be created by two methods: cold treatment and hot treatment. The different types of cold treatment include cold isostatic pressure (CIP[14]), cold drawing,[1] and equal channel multi-angle pressing (ECMAP[15]). These dislocations vanish as a result of thermal processes, however. The sintering for synthesizing an *in situ* MgB_2 material also creates dislocations, e.g., the substitution of C on B sites in MgB_2[16] and the shrinkage of the MgB_2 material.[17] These processes create an inhomogeneous distribution of dislocations. Only a hot isostatic pressure (HIP) makes it possible to obtain a homogeneous distribution of dislocations.[18,19] A homogeneous distribution of dislocations is very important, because it allows us to obtain a homogeneous distribution of the high field pinning centers. This leads to a high critical current density in the high magnetic fields.

Previous studies of the δT_c and δl pinning mechanisms were performed by using magnetic measurements.[2,4,9–12] We will show results on the δT_c and δl pinning mechanisms that were obtained by the transport measurements. In addition, in this article, we analyze the pinning mechanisms using the methods of Dew-Hughes and Higuchi *et al.*. We also show the effects of high pressure on the structure of the MgB_2 material, as well as T_c, B_{irr}, the upper critical field (B_{c2}), and J_c. Moreover, we will show that the distribution of pinning centers can have a decisive influence on the J_c at 20 K and 25 K. We will show that dislocations create high field pinning centers. These pinning centers also increase J_c at a high temperature.

II. PREPARATION OF SAMPLES

The wires were made at Hyper Tech Research using a continuous tube forming and filling (CTFF) process.[20] The MgB_2 wires had 36 filaments and a Nb barrier. The filaments comprised a nano-sized B, pre-doped with 2 at. % C with an Mg to B ratio of 1:2. The wires were fabricated to a diameter of 0.83 mm, achieving a fill factor of 14%. All the wires were annealed under an isostatic pressure at the Institute of High Pressure Research in Warsaw.[21] The HIP was a two-step process: the isostatic pressure was first applied, and then the wire sample was ramped to the set annealing temperature. The HIP process was ended by decreasing the annealing temperature to room temperature before decreasing the isostatic pressure. The samples were annealed at 700 °C at pressures between 0.1 MPa and 1 GPa (Table I). The HIP was performed in a 5 N argon atmosphere in a high gas pressure chamber. The transport critical current (I_c) of the MgB_2 wires was measured by the four-probe resistive method at 10 K, 20 K, and 25 K at the Institute for Solid State and Materials Research Dresden.[22] The I_c was determined on the basis of the 1 μV/cm criterion. The critical temperature and the critical magnetic fields were measured using the four-probe resistive method on a physical properties measurement system (PPMS), operating at 100 mA and 15 Hz at the International Laboratory of HMF and LT.[23,24] T_c, B_{irr}, and B_{c2} were determined with the respective criteria of 50%, 10%, and 90% of the normal state resistance. An analysis of the microstructure was performed using a scanning electron microscope (SEM; FEI Nova Nano SEM 230) at the Institute of Structural Research and Low Temperatures in Wroclaw, PAS.

III. RESULTS

Fig. 1(a) shows the results of scanning electron microscopy (SEM) for sample A (HIP at 0.1 MPa). These results indicate that annealing under a low pressure creates large voids, large grains, lower MgB_2 material density, and an inhomogeneous distribution of voids and grains. In addition, a low pressure (with the high pressure) reduces the number of connections between the grains and creates an inhomogeneous distribution of connections between the grains (with greater and fewer number of connections at different locations). This leads to an inhomogeneous distribution of the pinning centers and may facilitate the movement of the dislocations. On the other hand, results from sample C indicate that annealing under a high pressure creates small voids, small grains, and a better MgB_2 material density, producing a homogeneous distribution of grains (Fig. 1(b)). In addition, a high pressure increases the number of connections between the grains and creates a homogeneous distribution of connections between the grains. This leads to a homogeneous distribution of the pinning centers and a better distribution of the dislocations.

The results in Fig. 2 show that a pressure of 1 GPa decreases the T_c by 1.5 K in $B = 0$ T (sample A: 34 K and sample C: 32.5 K). A longer annealing time (90 min) does not change the T_c ($B = 0$ T). In addition, our studies indicate

TABLE I. The parameters of the HIP process for the samples in this study.

Sample identifier	HIP annealing time (min)	temperature (°C)	pressure (Pa)
A	15	700	0.1 M
B	15	700	0.4 G
C	15	700	1 G
D	60	700	1 G
E	90	700	1 G

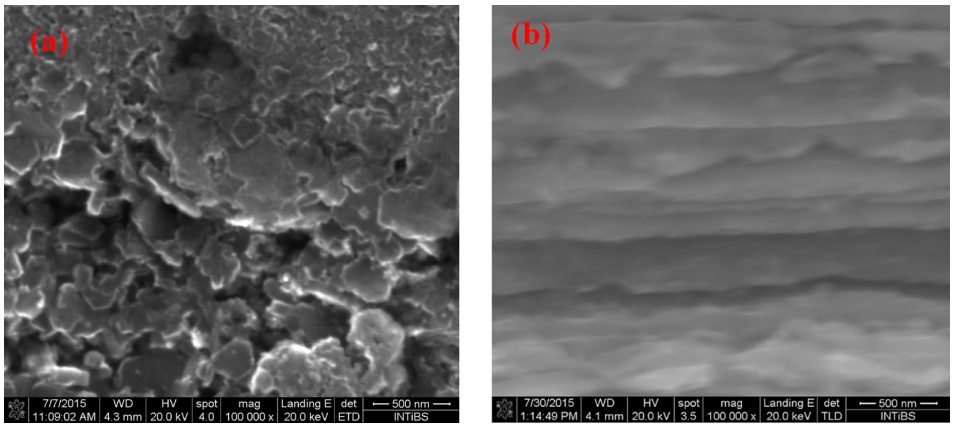

FIG. 1. The SEM images of a longitudinal section of C doped MgB_2 wire with a monel sheath and a Nb barrier: (a) Sample A: after the HIP process at 0.1 MPa and 700 °C for 15 min and (b) sample C: after HIP at 1 GPa and 700 °C for 15 min.

that a high pressure increases the T_c above 8 T, B_{irr}, and B_{c2} below 16 K, does not change the T_c in the range from 4 T to 8 T, B_{irr}, or B_{c2} in the range from 16 K to 25 K, and decreases the T_c below 4 T, B_{irr}, and B_{c2} above 25 K. These results are very important, because they indicate the temperature range in which the HIP process increases the critical parameters. The results presented by Monteverde *et al.*,[25] Lorenz *et al.*,[26] and Bordet *et al.*[27] indicated that decrease of the T_c might result from an increasing phonon frequency with an increased pressure, which broadens the density of state and lowers it on the Fermi surface. The reduction of the T_c of a wire under pressure during reaction would then be related to the loss of p_{xy} holes, the reduction of lattice parameters, and the reduction of c/a ratio (this effect can create a dislocation).[25–27] Serquis *et al.*,[18] however, have proposed that the HIP process creates strains, and this is the mechanism by which it reduces the T_c.[28] MgB_2 experiences a large shrinkage during the synthesis reactions at temperatures above 650 °C. The shrinking creates stress (dislocations). Buzea and Yamashita[29] proposed that the shrinkage of the MgB_2 crystal unit cell reduces the T_c by 1 K. Studies presented by Ghorbani *et al.*,[13] Kazakov *et al.*,[16] and Mudgel *et al.*[30] indicate that the C substitution on the B sites in the lattice creates a lattice distortion (strain) and disorder in the sigma band. It also leads to a reduction in the T_c. Our study on the undoped MgB_2 wires showed that 1 GPa pressure decreases the T_c by 1.5 K, increases the critical parameters in the range of 10 K to 27 K, and decreases the critical parameters above 27 K.[19] The similar influence of the isostatic pressure to that of C doping on the T_c may indicate that the isostatic pressure creates a lattice distortion. This is confirmed by the study of Serquis *et al.*[18] We believe that this effect decreases the T_c and the critical parameters above 25 K and increases the critical parameters below 16 K. Based

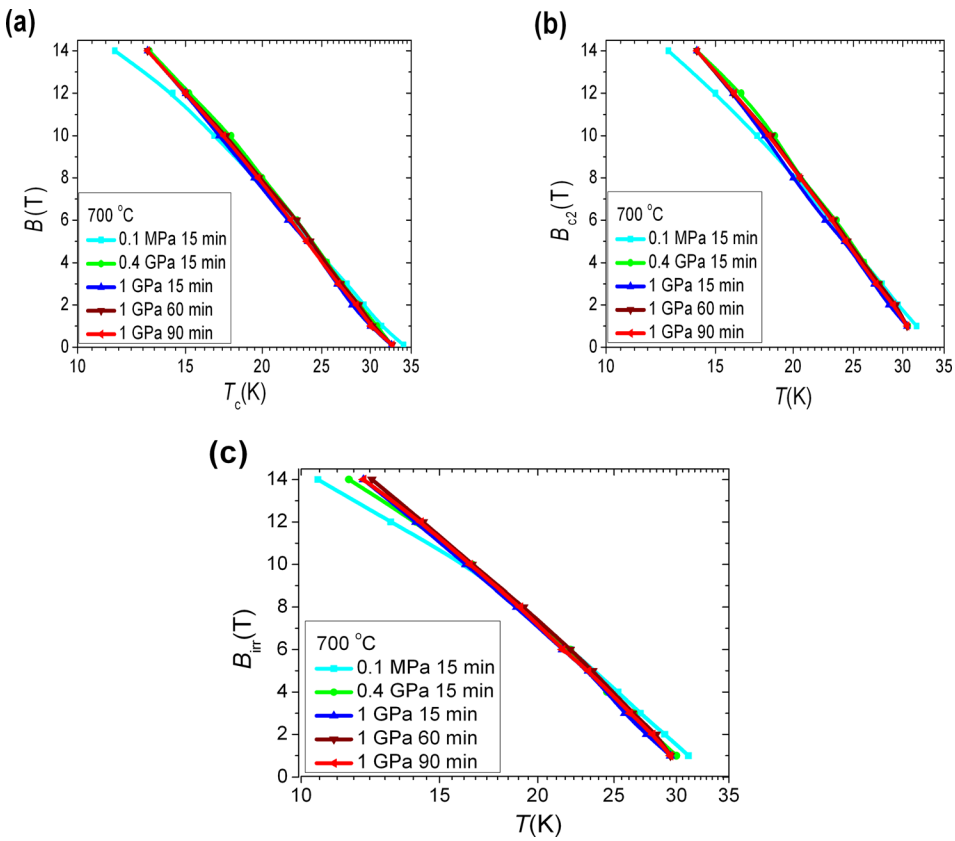

FIG. 2. The transport measurements for C doped MgB_2 wires after annealing in pressures from 0.1 MPa to 1 GPa: (a) the magnetic field dependence on the critical temperature (T_c); (b) upper critical fields (B_{c2}) dependence on the temperature; and (c) irreversibility magnetic field (B_{irr}) dependence on the temperature.

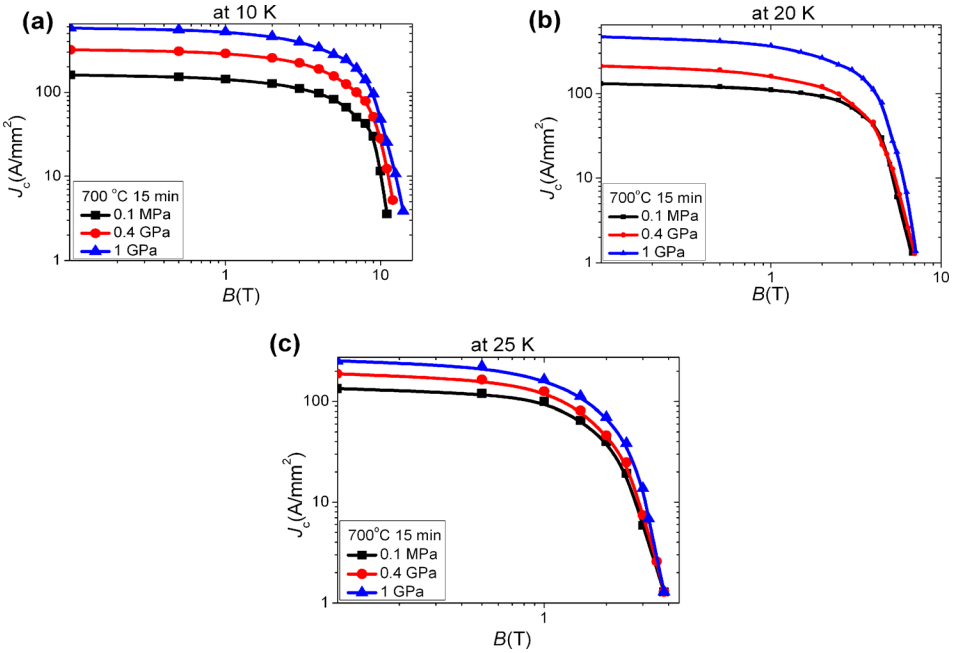

FIG. 3. Critical current density dependence on the magnetic field for samples A–C: (a) at 10 K, (b) at 20 K, and (c) at 25 K.

on the results in Fig. 2 and the results for the undoped MgB_2 wires,[19] we can deduce that a higher level of C doping and a higher pressure will cause a further reduction of critical parameters at higher temperatures (20 K–39 K). This indicates that a pressure of 1 GPa and a small amount of dopant are the optimum conditions. The shrinkage of the MgB_2 material during the solid-state reaction between Mg and B is small, about 5%, indicating that this effect does not significantly influence the T_c.[31] Monteverde *et al.*[25] proposed that the pressure decreases resistance in the normal state. The results in Fig. 1 suggest that this reduction may produce a greater number of connections between the grains.

The results in Fig. 3 show that a pressure of 0.4 GPa increases J_c, e.g., at 10 K by about 200%, at 20 K by about 10%–30% (in magnetic fields from 0 T to 2 T), and at 25 K by 15%–25% (in magnetic fields from 0 T to 1 T). Further increasing the pressure to about 0.6 GPa also increases J_c, e.g., at 10 K by about 100%, at 20 K by about 200%, and at 25 K by about 30% to 60%. An analysis of Fig. 4 allows us to determine the crossover fields from the single vortex to the small vortex bundle regime (B_{sb}) and from the small bundle regime to the thermal fluctuations regime (B_{th}).[9] Based on the transport measurements (Figs. 2 and 4), we have developed the B - T phase diagram in Fig. 5. In this diagram, the region below B_{sb} is associated with single vortex pinning, the region between B_{sb} and B_{th} small bundle pinning, the region between B_{th} and B_{irr} thermal fluctuations, the region between B_{irr} and B_{c2} vortex liquid, and the region above B_{c2} the normal state. The results in Fig. 5 for B_{th} show that a high pressure significantly reduces the thermal fluctuations and increases the region of small bundle pinning (the first such result). This result is very important, because it indicates the reason for a low J_c in the MgB_2 wires annealed at a low pressure (0.1 MPa). It is important that the HIP process can significantly reduce the thermal fluctuations above 20 K. A reduction of thermal fluctuations can cause a greater amount and a more homogeneous distribution of small bundle pinning, eliminate pure Mg (since pure magnesium has low resistivity), and lead to a greater number of connections between the grains. Our result also shows that a pressure of 1 GPa increases B_{sb}. We believe that a higher crossover field B_{sb} might indicate a greater amount of pinning centers that are more homogeneously distributed. The shape of the B_{sb} curves in Fig. 5 is similar to the curve of the δT_c pinning mechanism.[4] This indicates that a high pressure does not change the pinning mechanism.

We analyzed the pinning mechanism by using the Dew-Hughes' model, e.g., point pinning: $f(h_1) = A(h_1)^1(1 - h_1)^2$ and surface pinning: $f(h_1) = A(h_1)^{0.5}(1 - h_1)^2$, where A is a parameter and $h_1 = B/B_{irr}$. The results in Fig. 6(a) show that in the range from $h_1 = 0$ to 0.6, the point pinning mechanism is dominant, and, from 0.7 to 1, the surface pinning mechanism is dominant. This suggests that the samples have an insufficient number of high field pinning centers. Similar scaling results were obtained at 10 K and 25 K. We see that a

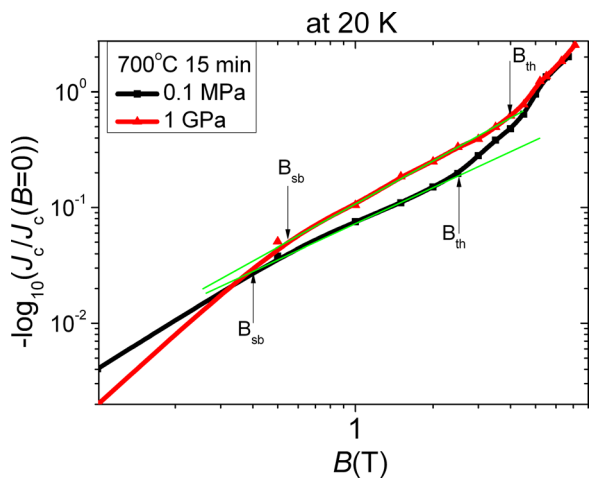

FIG. 4. A double logarithmic plot of $-\log[J_c/J_c(0)]$ as a function of B at 20 K for samples A (0.1 MPa) and C (1 GPa). The crossover fields B_{sb} and B_{th} are marked by arrows.

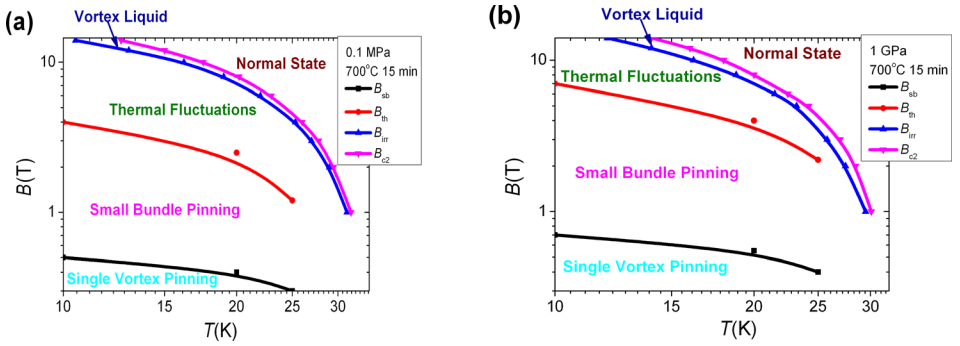

FIG. 5. A phase diagram for 2 at. % C doped MgB_2 wires. B_{sb} and B_{th} were obtained from the transport measurement data on $J_c(B)$ (see Fig. 3). $B_{irr}(T)$ and $B_{c2}(T)$ were obtained from the transport measurement data (see Fig. 2): (a) sample A and (b) sample C.

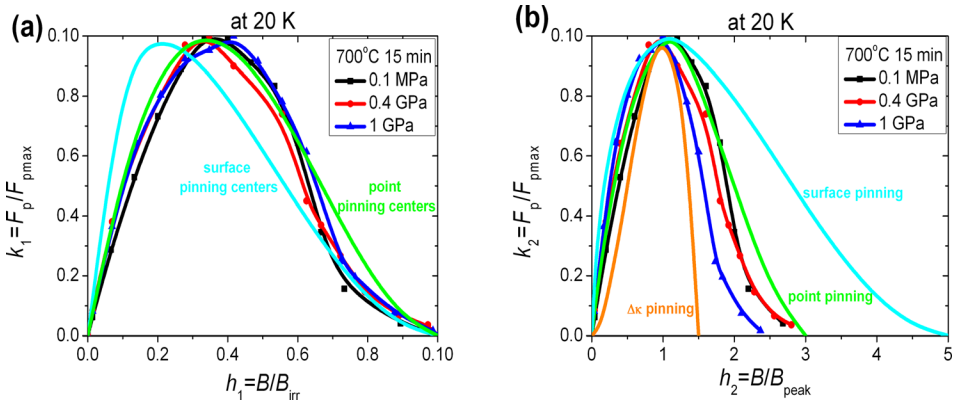

FIG. 6. (a) The reduced pinning force depending on the reduced magnetic field B/B_{irr} for samples A–C—an analysis of pinning by the Dew-Hughes' model[7] at 20 K, and (b) the reduced pinning force depending on the reduced magnetic field B/B_{peak} for samples A–C—an analysis of pinning by the model of Higuchi *et al.*[9] at 20 K.

high pressure does not change the dominant pinning mechanism. This indicates that the high pressure increases the amount of pinning centers and the number of connections between the grains (Fig. 1). This leads to an increased J_c.

We also analyzed the pinning mechanism by using the method of Higuchi *et al.*:[5] $f(h_2) = (9/4)h_2(1 - h_2/3)^2$ for normal point pinning, $f(h_2) = (25/16)(h_2)^{0.5}(1 - h_2/5)^2$ for surface pinning, and $f(h_2) = (3h_2)^2(1 - 2h_2/3)$ for $\Delta\kappa$ pinning, where $h_2 = B/B_{peak}$. Fig. 6(b) shows that the low pressure (0.1 MPa) leads to the normal point pinning in the range from 0 to 2 T. The 0.4 GPa pressure slightly shifts the curve of sample B from normal point pinning to $\Delta\kappa$ pinning in the range from 1 to 2 T. A high pressure of 1 GPa shifts the pinning mechanism to $\Delta\kappa$ pinning. This indicates that the high pressure creates the dislocations (high field pinning centers).

The results in Fig. 7 show that a high pressure increases the J_c at high temperature (10 K–26 K) in 2 T. A long annealing time slightly increases J_c. Moreover, the shape of the curves in Fig. 7 is similar to the curve for the δT_c pinning mechanism.[2] This may indicate that a high pressure does not adversely affect the δT_c pinning mechanism.

The results in Fig. 8 show that a longer annealing time increases the J_c modestly at lower temperatures but more markedly at higher temperatures: at 10 K, by about 15%–70%; at 20 K, by about 30%–90%; and at 25 K, by 100%. Moreover, a long annealing does not increase B_{sb} or B_{th} (Fig. 9) and does not change the dominant pinning mechanism in the range from 10 K to 25 K (Fig. 10). No change in the dominant pinning mechanism indicates that an annealing over a longer time creates more connections between the grains and increases the pinning center density. These factors increase J_c at a temperature range from 10 K to 25 K.

IV. DISCUSSION

Previous reports on the δT_c and the mean free path δl pinning mechanisms were based on the magnetic results (magnetic critical current density, J_{cm}).[2,4,9–12] The research presented by Shi indicated that the transport current flows mainly through the longitudinal connections.[32] In contrast, the magnetic current flows in the grains and transverse connections.[32] In our paper, we present the transport measurement results, which relate to the longitudinal connections. On the basis of the three pinning mechanisms, we will examine the impact of additives and a high isostatic pressure for the creation of the pinning centers.

Studies show that doping with C makes the δl pinning mechanism dominant.[2,10,33] The authors of these studies

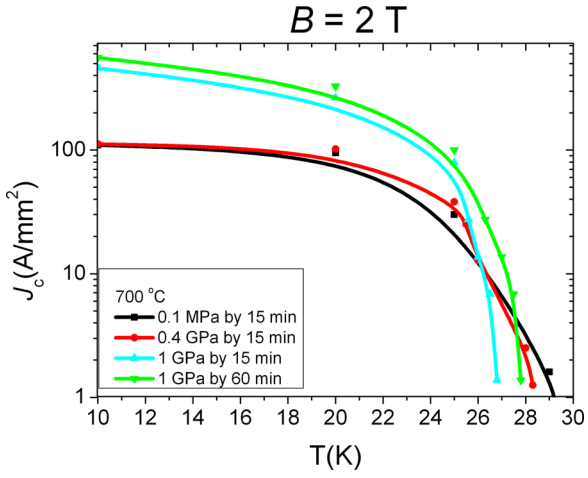

FIG. 7. The critical current density dependence on the temperature in $B = 2$ T for samples A–D.

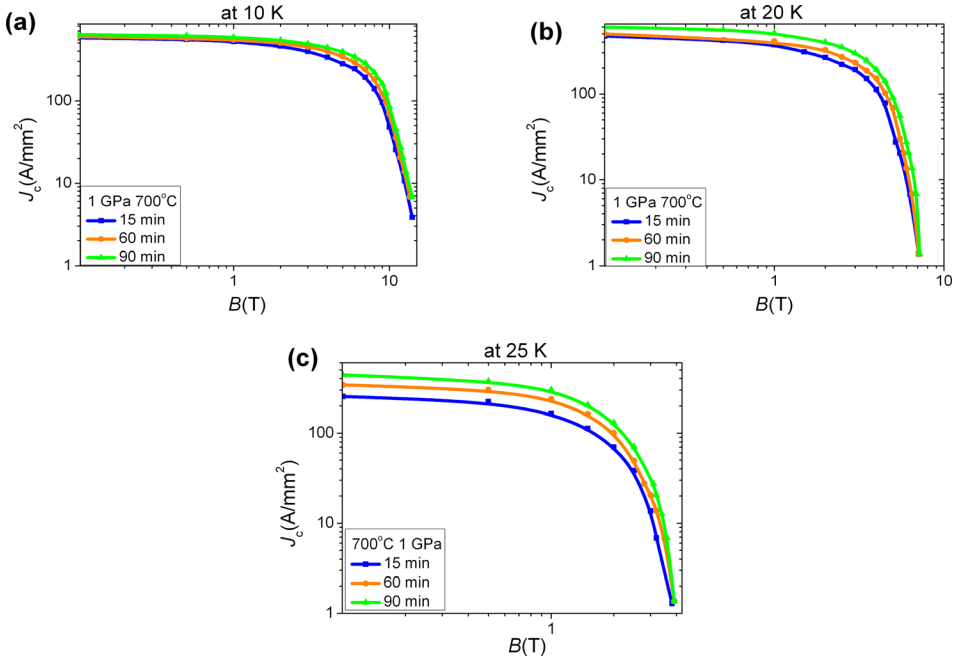

FIG. 8. The critical current density dependence on the magnetic field for samples C–E (a) at 10 K, (b) at 20 K, and (c) at 25 K.

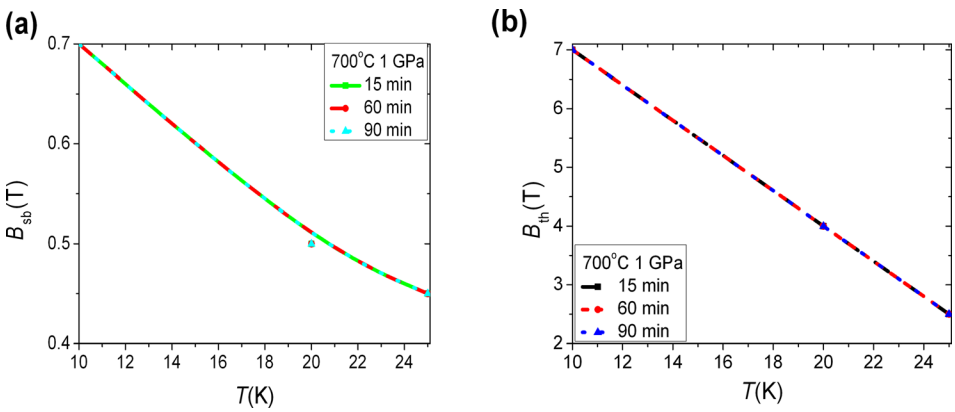

FIG. 9. (a) The crossover fields B_{sb} for samples C–E depending on the temperature and (b) the crossover fields B_{th} for samples C–E depending on temperature.

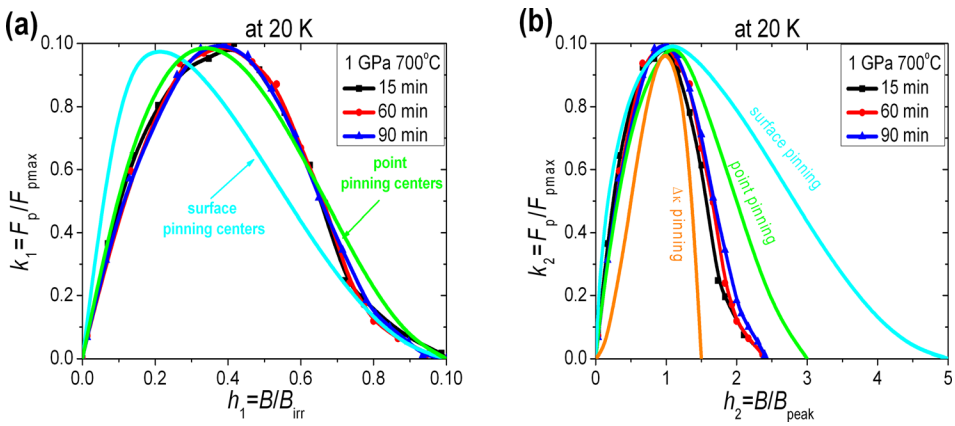

FIG. 10. (a) The reduced pinning force depending on the reduced magnetic fields B/B_{irr} for samples C–E—an analysis pinning of Dew-Hughes' model[7] at 20 K and (b) the reduced pinning force depending on the reduced magnetic fields B/B_{peak} for samples C, D, and E—an analysis of pinning by the model of Higuchi *et al.*[9] at 20 K.

have shown that C is substituted for B, creating the dislocations. This indicates that the dislocations create the δl pinning mechanism. In addition, the dopant is non-uniformly distributed in the structure of the superconducting material, leading to a non-uniform distribution of dislocations in the superconducting material. These authors showed that undoped MgB_2 material features the δT_c pinning mechanism (for annealing above 650 °C).[2,11,12,33] A heat treatment of the MgB_2 material above 650 °C (the melting point of Mg), however, causes a strong shrinkage of the MgB_2 material, even by 25%.[17] The shrinkage of the material leads to the creation of dislocations. This indicates that dislocations also create the δT_c pinning mechanism. The dislocation density created by the shrinkage is smaller, however (small reduction of T_c of about 1 K (Ref. 29)). A high dislocation density with an inhomogeneous distribution causes large fluctuations in the

mean free path. This factor at higher temperature (20 K) significantly decreases T_c, B_{irr}, B_{c2}, and J_c. A smaller dislocation density with an inhomogeneous distribution causes smaller fluctuations in the mean free path. This leads to a smaller reduction in the critical parameters at high temperatures (above 20 K) and would explain why the small amount of doping (at 2% C) in our wires does not create the δl pinning mechanism. Studies indicate that the HIP process increases the dislocation density in the MgB_2 material. This should create the δl pinning mechanism. Our research indicates, however, that we obtain the δT_c pinning mechanism. This may be because the HIP process creates a uniform distribution of dislocations in the superconducting material. A high dislocation density with a homogeneous distribution causes smaller fluctuations in the mean free path. This leads to the δT_c pinning mechanism and increases the critical parameters at high temperature.

The results presented by Embon suggest that a vortex typically interacts with small clusters of a few pinning defects separated by about the coherence length.[34] This may mean that the random distribution of pinning centers causes a strong clustering of the vortices. This leads to a reduction in the critical parameters. In contrast, a homogeneous distribution of the pinning centers reduces the clustering of pinning centers and thus of vortices, leading to an increase in the critical parameters. Moreover, the results in Ref. 34 indicate that a random configuration of the pinning centers (defects) leads to a greater thermally activated depinning of vortices, even in the case of strong pinning. Our research shows that the HIP process significantly reduces the thermal fluctuations. This might indicate that the HIP process leads to a more uniform distribution of the pinning centers.

The δT_c and δl pinning mechanisms have mainly been associated with the effects of the temperature on pinning centers. In contrast, the models of Dew-Hughes[3] and Higuchi *et al.*[5] mainly deal with the effects of the magnetic field on the pinning centers. Our results suggest that both the methods lead to similar results. The Dew-Hughes' method in Fig. 6(a) shows that we do not have pinning points above $h_1 = 0.6$ (high field pinning centers), although this method does not indicate which type of defect is missing (precipitates with a thickness similar to the coherence length or dislocations). The Higuchi model shows that we do not have enough strong high field pinning centers. This analysis strongly suggests that we have a small amount of $\Delta\kappa$ pinning centers. Our research shows, however, that a high isostatic pressure is a method that can yield a dominant $\Delta\kappa$ pinning mechanism. The results presented by Livingston indicate that the dislocations mainly create $\Delta\kappa$ pinning[1] This suggests that the HIP process increases the dislocation density. The results in Fig. 6(b) indicate that a higher dislocation density reduces the thermal fluctuations and increases B_{sb}, B_{th}, and J_c at 20 K.

V. CONCLUSIONS

From our research, a high isostatic pressure of 1 GPa creates the pinning centers similar to the $\Delta\kappa$ pinning centers. An analysis performed by using the three models of pinning mechanisms indicates that the dislocations create the $\Delta\kappa$ pinning centers. Our measurements show that a homogeneous distribution of the $\Delta\kappa$ pinning centers increases the critical current density at 20 K and 25 K, increases the crossover fields B_{sb} and B_{th}, as well as B_{irr} and B_{c2}, below 25 K, significantly reduces the thermal fluctuations, and improves the δT_c pinning mechanism. This is a very important result, because it shows that the HIP process can control the process for creating the pinning centers. By controlling the value of the pressure, we can control the sizes of grains and inclusions, the dislocation density, and the density and uniformity of the material. The HIP process is a simpler, easier method and can be used on a wider scale to obtain structure texturing of the MgB_2 material than thin film methods. Our results show that the HIP process increases J_c by about 200%.

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