

Effect of laser irradiation on superconducting properties of bulk MgB_2 sintered at different temperatures

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Abstract The surfaces of MgB_2 bulk samples produced by a pellet/closed tube method at two different sintering temperatures of 650 and 850 °C, after hot pressing at 200 °C, were irradiated with the same irradiation dose by using an Nd:YVO₄ laser in order to study the possible potentiality of laser irradiation to improve pinning performances and critical current density of MgB_2 superconductor. The measurements showed that the magnetic field dependence of the critical current density values of irradiated sample sintered at 650 °C slightly increased with a narrowing in superconducting transition region as compared to the reference sample sintered at same temperature. However, irradiated sample sintered at 850 °C showed a decrease in pinning performance and similar critical temperature values as compared to the corresponding reference sample. From these results it can be said that the same laser irradiation dose affects superconducting properties of bulk MgB_2 in different ways depending on sintering temperature of the superconductor.

1 Introduction

Since the discovery of superconductivity in MgB_2 [1], with the highest transition temperature (T_c) of 39 K among metallic superconductors, extensive studies have been carried out not only because of its special physical properties such as high critical current density (J_c) and the absence of weak-links [2, 3], but also because it is a good candidate for several technical applications in superconducting magnets [4], contactless magnetic bearings [5] and superconducting fault current limiters [6], with a relatively low cost in comparison with other superconducting materials.

It is well known that the supercurrent flow in MgB_2 is unhindered by grain boundaries as opposed to high T_c superconductors (HTSCs). However, in a clean MgB_2 superconductor, the critical current density is strongly suppressed [7] when a magnetic field is applied because of lack of natural defects that can act as pinning centres in the MgB_2 structure. For high field applications, it is crucial to create pinning centres in this material. Therefore, in the last decade, there have been significant efforts to enhance the high field performance of J_c by introducing effective pinning centres into MgB_2 using several methods such as nano C [8] and nano SiC doping [9, 10], proton irradiation [11], hot isostatic press (HIP) [12], gamma irradiation [13] and heavy-ion irradiation [14] etc.

One of the promising approaches to enhance the pinning properties of MgB_2 is the introduction of defects and atomic disorder by means of particle irradiation. The physical mechanism of irradiation is very simple. Particles with high energy pass through the sample and displace atoms from their equilibrium lattice positions, which create vacancies and interstitial positions in the lattice [15]. The study of irradiating MgB_2 bulk samples using gamma-rays, studied by Obaidat et al. [13], showed an enhancement in

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J_c because of the migration of MgO inside the MgB_2 grains which improves the grain connectivity and causes oxygen related defects in the grains and also oxygen segregation in the grain boundaries where it increases the vortex flux pinning. Also, the study reported by Verdin et al. [15] showed that the J_c values of MgB_2 bulk samples at low temperatures could be enhanced by using electron, proton and gamma irradiations. Especially, the gamma-irradiated sample presented the best J_c character compared with the other irradiated samples due to the higher number of lattice defects created by the gamma irradiation.

Although the influence of laser irradiation on the superconducting properties of HTSCs such as $\text{YBa}_x\text{Sr}_{2-x}\text{Cu}_3\text{O}_{7-x}$ [16], Y-123, Bi-2212 and Bi-2223 [17], Bi(Pb) system (2223 composition) [18] and Bi-2212 [19] has frequently been studied by several groups, there isn't any study on bulk MgB_2 -laser beam interaction in the literature. In the case of HTSCs, laser has been used to induce a textured microstructure by controlling the solidification process after laser induced melting. Due to the incongruent melting behaviour, an adequate thermal annealing is usually required to recover the superconducting phase. The final textured superconducting samples show high critical current values. Okutomi et al. [16] reported that the ceramic superconductor $\text{YBa}_x\text{Sr}_{2-x}\text{Cu}_3\text{O}_{7-y}$ was successfully recrystallized by CO_2 laser irradiation and the J_c value increased by 25 times after the irradiation. Also, Luticiv et al. [17] reported an increment in J_c of HTS ceramic superconductors by 2.5–3 times after the recrystallization by laser irradiation and the additional thermal annealing in O_2 for restoration of superconducting properties after laser process.

It is necessary to understand how the laser irradiation can modify the superconducting properties of MgB_2 . Therefore, the goal of this study is focused on investigating the effect of near-infrared continuous laser irradiation on bulk MgB_2 samples sintered at two different temperatures of 650 and 850 °C. These two sintering temperature values were selected because of the fact that 650 °C is the melting point of magnesium (Mg) and these two values are the general sintering temperatures for MgB_2 , as reported in literature.

2 Experimental details

In this study, disk-shaped polycrystalline MgB_2 superconductors were prepared from commercially available high purity powders of Mg (−325 mesh, 99.8%, Alfa Aesar) and amorphous nano B (>98%, Pavezyum) by using a pellet/closed tube method after hot pressing at 200 °C. The two MgB_2 pellets used in this study were produced with the same production conditions described in our previous

studies [20, 21], only the sintering temperature was modified. One of the pellets was sintered at 650 °C and the other one at 850 °C for 3 h. The obtained pellets with 18.5 mm in diameter and 2.5 mm in height were cut into two pieces. The two pieces, cut from the pellet sintered at 650 °C, were named as 650R and 650L and the other two pieces obtained from the pellet sintered at 850 °C, were named as 850R and 850L.

Samples 650L and 850L were irradiated by using an Nd:YVO₄ laser (Powerline E, RoFin) emitting at a wavelength of 1064 nm in combination with a vacuum chamber coupled to an automatized positioning system to perform the laser scanning treatment of the samples. The two untreated samples (650R and 850R) were considered as the corresponding reference samples. Figure 1 shows an image of the experimental setup employed. The laser apparatus, which allows scanning the surface of the samples within the xy-plane, is fitted with a galvanometer beam steering system and a flat-field lens with a 160 mm focus. In order to apply the laser treatment, 650L and 850L samples were placed into the vacuum chamber. At a high vacuum conditions (around 10^{-6} mbar), a laser scanning process was applied to the samples moving with a speed of 0.08 mm s^{-1} in $\pm x$ direction. The laser beam was set to scan the surfaces of the samples on the $\pm y$ direction with an irradiance of $8.63 \times 10^6 \text{ W cm}^{-2}$ in continuous mode. The laser irradiation scanned the surface of the samples at 500 mm s^{-1} during the motion in $\pm y$ direction.

The phase compositions of all samples were examined by using X-ray diffraction (XRD) with a Rigaku D/Max-IIIC X-ray diffractometer. The X-rays were generated at 40 kV and 30 mA. Measurements were performed at 2θ between 20° and 80° with $\text{step}=0.03^\circ$ and $t=2 \text{ step/m}$. Also, the modifications of surface morphologies of the

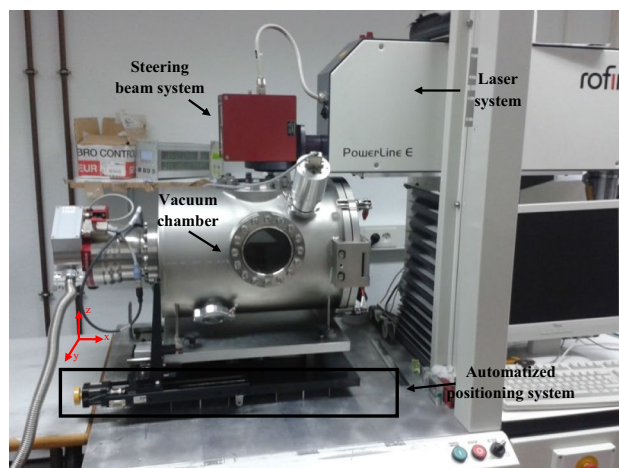


Fig. 1 The experimental setup used for the laser irradiation in this study

irradiated samples were analysed by using a field emission scanning electron microscope (FESEM, Carl Zeiss MERLIN™).

In order to analyse the superconducting properties, the studied samples were cut into rectangular shapes. The superconducting transition temperatures (T_c^{onset} and T_c^{offset}) were determined according to the Zero Field Cooled DC magnetic susceptibility measurements ($H=100$ Oe) at temperatures between 5 and 50 K by using a Quantum Design Physical Properties Measurement System (PPMS). The magnetic hysteresis loops $M(H)$ of the samples were obtained by using a Vibrating Sample Magnetometer (VSM, Quantum Design PPMS) attached on the PPMS, equipped with ± 7 T magnetic fields and a sweep rate of 100 Oe s^{-1} at three different temperatures ($T=5, 10$ and 15 K). The $M(H)$ loops were measured while the magnetic field was applied perpendicular to the pressed surface (the pressed surface was normal to the beam direction). The magnetic field dependence of critical current density ($J_c(B)$, $B=\mu_0 H$) of the studied samples was calculated by using the equation $J_c = 20\Delta M/[a(1-a/3b)]$ based on Bean's critical state model [22]. In this relationship, ΔM is the difference between the upper and lower branches of $M(H)$ curve (in emu) divided by the volume of the sample (in cm^3), $a \times b$ is the surface area of the sample which is perpendicular to the

direction of the applied magnetic field with $b > a$, a and b are in cm, while J_c is in A cm^{-2} . Also, in order to obtain an explicit insight into the pinning mechanism of the samples, the magnetic field dependence of volume pinning force, defined as $F_p = J_c \times B (B = \mu_0 H)$ by using the $M(H)$ curves of the samples, was analysed.

3 Results and discussions

The phase compositions of the samples studied in this work are shown in Fig. 2. The X-ray patterns of 650R and 650L samples (Fig. 2a) show almost the same phase formation [21]. The presence of peaks associated with the crystalline phases of unreacted Mg indicates that the sintering temperature and/or sintering time are not enough for the complete formation of MgB_2 phase. The presence of MgO phase is possibly due to the entrapped air in the powder mixture before end-sealing of the chrome tube. The lack of alignment between the MgB_2 grains and the presence of impurities (unreacted residual Mg and MgO) [21] are also seen from Fig. 2a. In opposite, the X-ray patterns of samples 850R and 850L show that the major phase is MgB_2 with a little amount of MgO phase (see Fig. 2b). The MgB_2 peaks of 850R and 850L samples become sharper and

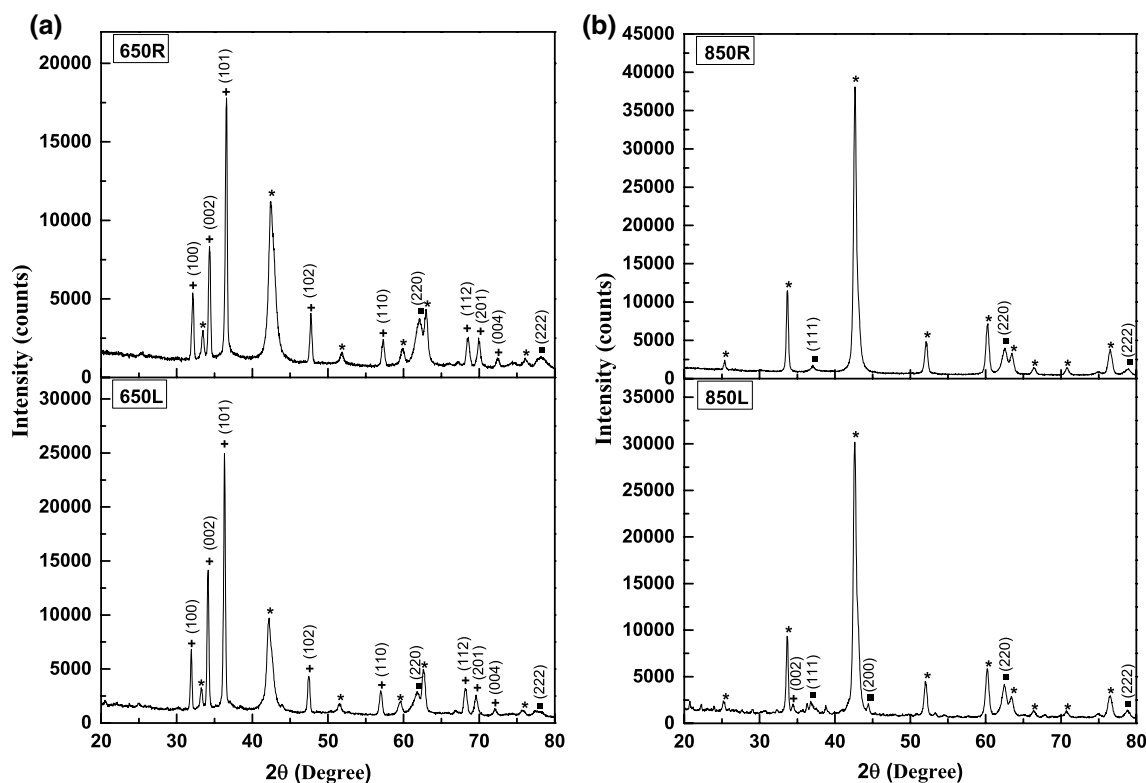


Fig. 2 XRD diffraction patterns of **a** 650L and 650R samples and **b** 850L and 850R samples. MgB_2 , Mg and MgO phases are shown with the symbols of asterisk, plus and square, respectively

higher as the sintering temperature increases from 650 to 850 °C, which indicates an increase in phase purity and/or crystallinity. Also, (200) MgO phase and (002) Mg phase are formed in 850L sample while the peak intensity of Mg phase increases in sample 650L after the irradiation. However, the X-ray peaks associated to MgB_4 or other higher borides are not observed in the studied samples, confirming that there was not any evaporation of Mg during heat treatment and the laser irradiation process. The significant difference observed between the XRD spectra of the irradiated and the reference samples, is the decrease of the volume fraction of MgB_2 superconducting phase after the laser irradiation process. These results indicate that the MgB_2 phase is preserved in the partially melted samples during the irradiation [21] and the laser irradiation damages the superconducting phase only on the surface of the irradiated samples.

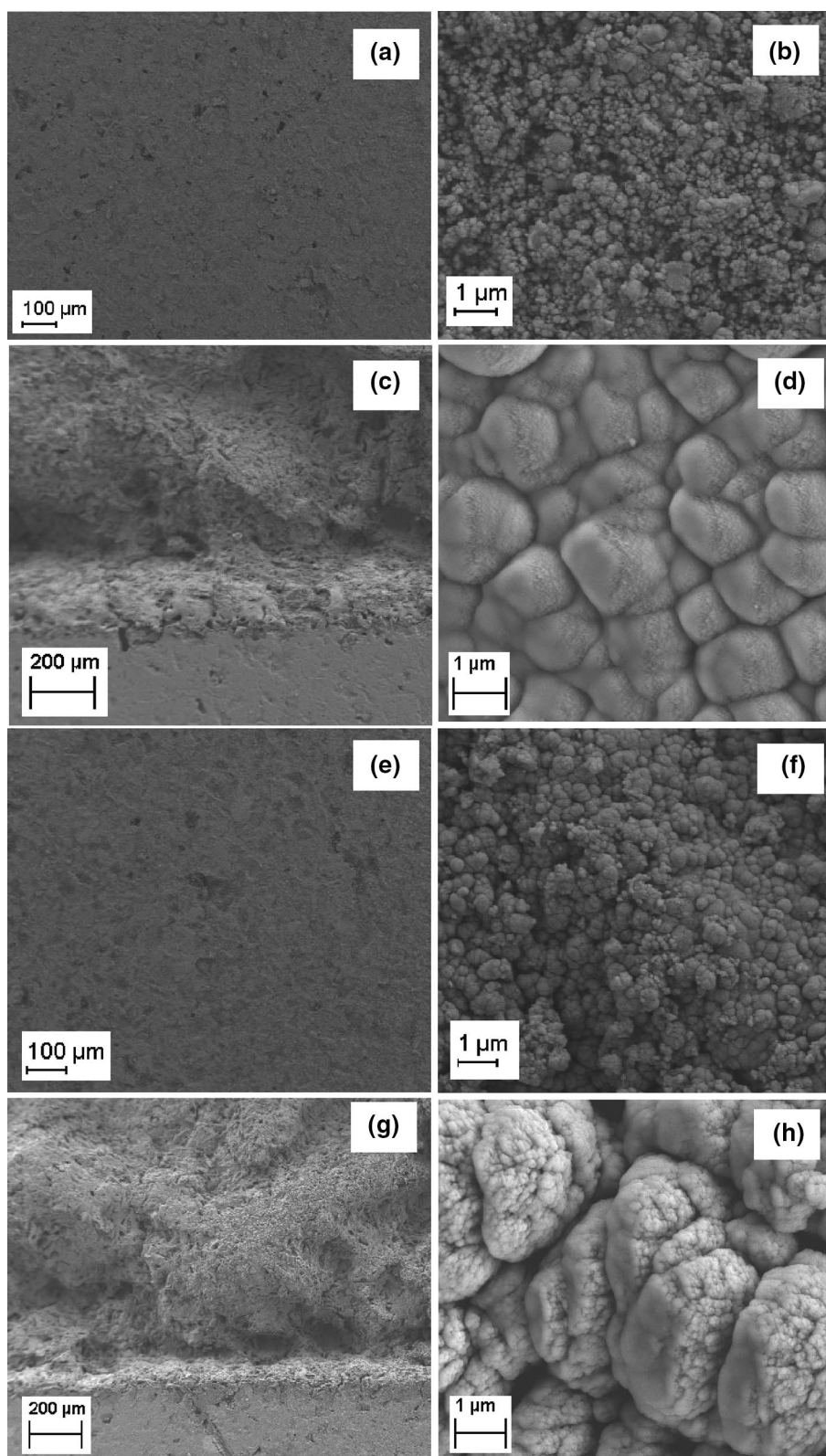
Figure 3 shows the morphological changes induced by laser irradiation on the surfaces of the irradiated samples. Figure 3a–d show the FESEM images of samples 650R and 650L, respectively. Also, Fig. 3e–h show the FESEM images of samples 850R and 850L, respectively. Comparing Fig. 3a, c for samples 650R and 650L, and also comparing Fig. 3e, g for samples 850R and 850L, respectively, it can be said that the laser irradiation promotes a melting and a subsequent ejection process on the surface of the irradiated samples [21] and causes a structural deformation because of the energy transferred to the surfaces of the samples during irradiation. In addition, comparing Fig. 3b, d for samples 650R and 650L, and also comparing Fig. 3f, h for samples 850R and 850L, respectively, it can be said that the grain size changes after the irradiation. The grains of the irradiated samples are significantly grown, as shown in Fig. 3d, h for samples 650L and 850L, respectively, due to the laser-induced sintering. The average grain size of 650R is under 0.1 μm (see Fig. 3b), and for sample 650L it is about 0.2–0.8 μm (see Fig. 3d) [21]. For 850R and 850L samples the average grain sizes are about 0.2 μm (see Fig. 3f) and 1–1.5 μm (see Fig. 3h), respectively. The grain growth larger than 20 μm was reported by Okutomi et al. for $\text{YBa}_x\text{Sr}_{2-x}\text{Cu}_3\text{O}_{7-y}$ ceramic oxide superconductor irradiated by CO_2 laser [16]. The modified surface of 650L sample has dense, compacted grains, uniform grain size distribution and better crystallinity, since many pores exist in the corresponding reference sample 650R [21]. It shows that the grain growth improves the interconnectivity among the superconducting grains [23]. This can be also attributed to the physical densification of 650L sample. On the other hand, for sample 850L the grains are grown and separated from each other after the irradiation. The breaking into parts and the formation of well-defined grains show that the material heats up and cools down without the bulk material being involved [24].

It implies that the laser irradiation dose is not sufficient to transfer the heat induced by irradiation to the all volume of the bulk sample. So, it provokes the formation of porosity on the surface of 850L sample (see Fig. 3h). Sekkina et al. also observed a morphological change on the surface of the $\text{Bi}_{0.8}\text{Nd}_{0.1}\text{Tb}_{0.1}\text{PbSr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ superconductor irradiated by Nd-laser. The surface of the sample began to have micro cracks in the direction of laser scan after the irradiation with the irradiation dose of 10 W cm^{-2} . But the morphology of the sample surface was completely changed as a result of melting and recrystallization. Then it began to coagulate reducing the number of cracks after the irradiation dose of 20 W cm^{-2} [25]. Moreover, it can be seen from FESEM images that the grain growth shows a preferred orientation in the scanning direction of the laser for both irradiated samples. This orientation to the direction of beam scan on the laser irradiated area was also found by Hotta et al. for Pb doped Bi-2223 system irradiated by CO_2 laser [18].

Figure 4 a and b illustrate the DC magnetic susceptibility versus temperature curves ($M/M(5\text{ K}) - T$), which are normalized to the value at 5 K. In order to illustrate exactly the critical temperatures of the studied samples, the insets in Fig. 4a, b were plotted in a temperature region around the transition. The superconducting transition temperature values (T_c^{onset} and T_c^{offset}) of the samples were defined by using a 10% screening and a 90% screening from the normalized magnetic susceptibility curves, respectively. Also, the transition regions (ΔT_c , $\Delta T_c = T_c^{\text{onset}} - T_c^{\text{offset}}$) were defined from the difference between the values of T_c^{onset} and T_c^{offset} . Table 1 shows the critical temperature values of all the samples. The sharpest transitions to the superconductivity were observed for the samples sintered at 850 °C. Furthermore, in the case of 650R and 650L samples, the T_c^{onset} value slightly increases and the ΔT_c value decreases about 1 K after the irradiation. This narrowing of the superconducting transition implies that the additional heat transferred during the irradiation leads to enhance the bulk property of 650L sample via improving the interconnectivity among the superconducting grains and reducing the porosity, as consistent with the FESEM image (see Fig. 3d). Also, the T_c^{offset} value of 650L sample is higher about 1 K than that of the 650R sample, implying an enhancement of inter granular connectivity after the irradiation because of the fact that the T_c^{offset} is in accordance with the features of inter granular component of a superconductor [26]. In opposite, the T_c^{onset} , T_c^{offset} and ΔT_c values remain unchanged for 850R and 850L samples, implying that the stoichiometry of 850L sample was not changed after the irradiation and the irradiation dose was not sufficient to affect the all volume of this sample.

Figures 5 and 6 show the magnetic field dependence of critical current densities $J_c(B)$ of the reference and the

Fig. 3 FESEM images of the modified surfaces of the samples **a, b** 650R, **c, d** 650L and the samples **e, f** 850R, **g, h** 850L at different magnifications



irradiated samples at three different temperatures. The J_c values were calculated from the width of magnetic hysteresis loops shown as inset figures in Figs. 5 and 6, on the

basis of the Bean's critical state model [22]. The J_c values shown in Table 1 for the magnetic field of 3 T and at the temperatures of 5, 10 and 15 K are higher for the samples

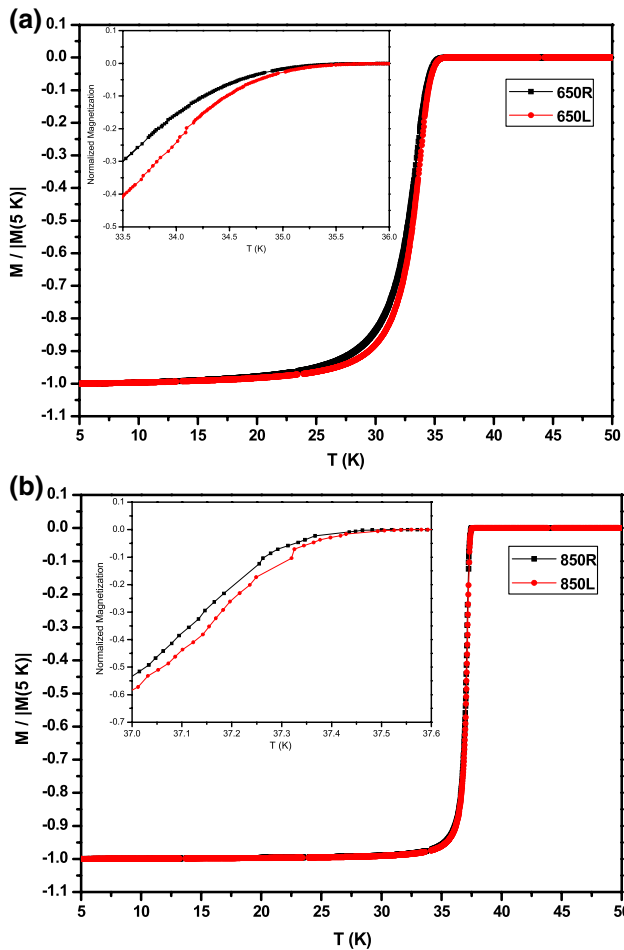


Fig. 4 Normalized DC magnetic susceptibility versus temperature curves ($M/M(5\text{ K}) - T$) at 100 Oe for **a** 650R and 650L samples and **b** 850R and 850L samples. The *inset* figures show the normalized curves at the temperatures between 33.5 and 36 K, and also between 37 and 37.6 K for the samples sintered at 650 and 850 °C, respectively

sintered at 850 °C ($\sim 10^4\text{ A cm}^{-2}$ at 3 T) as compared to the other two samples sintered at 650 °C ($\sim 10^3\text{ A cm}^{-2}$ at 3 T) (see Table 1), because of the increase in volume fraction of superconducting phase depending on the initial sintering temperature, as shown in Fig. 2b. It is known that the magnetization current results from the flux within the grains for sintered samples in the superconducting mixed state of a type-II superconductor and also the critical current density obtained from the magnetic hysteresis loop determines the critical properties of the individual grains, i.e., the intra granular current [27]. It can be seen from Fig. 5 that the 650L sample shows higher J_c in an external field at all measurement temperatures, especially at 15 K, as compared with the 650R sample. The J_c values were calculated as 2.96×10^3 and $4.10 \times 10^3\text{ A cm}^{-2}$ at 5 K and 3 T for the samples 650R and 650L, respectively. The enhancement of J_c values at high magnetic fields for 650L sample

is maybe due to the creation of additional defects induced by laser irradiation, which act as pinning centres [23]. The J_c enhancement indicates that the laser irradiation enhances the flux pinning capability and the pinning force of 650L sample. On the other hand, it can be seen from the Fig. 6 at low magnetic fields and low temperatures ($T < 15\text{ K}$) many flux jumps and sudden magnetization changes are observed for samples 850R and 850L because of extremely high critical current density [28, 29]. In addition, a small amount of heat caused by flux movement degrades the superconducting property of these samples seriously because of the very low specific heat at 5 K [30]. It is seen from the Table 1 and Fig. 6, the J_c values of 850L sample at all measurement temperatures are smaller than that of 850R sample. Comparing the J_c values of these two samples one can say that the laser irradiation causes an appreciable decrease in magnetic J_c for sample 850L. The reason is the decrement of the dense structure because of the formation of porosity and so, the deterioration of bulk pinning properties by the laser irradiation.

In order to obtain an explicit insight into the pinning mechanism of the samples, the magnetic field dependence of volume pinning force, defined as $F_p = J_c \times B (B = \mu_0 H)$ by using the Figs. 5 and 6, was analysed. Figure 7 shows the magnetic field dependence of volume pinning force $F_p(B)$ of the reference and the irradiated samples. It is known that in conventional type-II superconductors in magnetic fields above the lower critical field (B_{c1}), magnetic flux can coexist with the superconducting state by allowing flux penetration into the superconductor as quantized vortices. When currents flow in superconductors, the Lorentz force acts on the vortices perpendicularly to them. They will start to move when the currents reach the critical current density, that's why at this point the Lorentz force overcomes the pinning force [31]. Compatible with this typical behaviour, F_p curves show a linear nature at lower fields up to a maximum value that the pinning force is equal to Lorentz force, after which the curves exhibit a quadratic behaviour at higher fields because of the Lorentz force surmounts the pinning force, as shown in Fig. 7a, b. Also, it can be seen from Fig. 7 that the peak position of the maximum F_p is temperature dependent. The peak positions move to lower fields with increasing temperature for all the samples. This shifting can be clearly seen in Fig. 7b for samples 850R and 850L. For example, the maximum F_p values of 850R sample are $19.11 \times 10^8\text{ Nm}^{-3}$ at 2.17 T and $16.01 \times 10^8\text{ Nm}^{-3}$ at 1.26 T at 5 K and 15 K, respectively. In addition, the maximum F_p values for 850R sample at 5, 10 and 15 K are higher than the values of 850L sample and the samples produced at 650 °C, as seen in the Table 1, implying that the sintering temperature of 850 °C is enough to complete the formation of MgB_2 and to improve the connectivity in contrast with 650 °C. In opposite, the maximum F_p values for

Table 1 T_c^{onset} , T_c^{offset} , ΔT_c , J_c and $F_{p,max}$ values of the samples studied in this work

	650R	650L	850R	850L
T_c^{onset} (K)	34.3	34.5	37.3	37.3
T_c^{offset} (K)	28.4	29.5	36.1	36.2
ΔT_c (K)	5.9	5.0	1.2	1.1
J_c (5 K, 3 T) (A cm ⁻²)	2.96×10^3	4.10×10^3	5.38×10^4	3.66×10^4
J_c (10 K, 3 T) (A cm ⁻²)	2.15×10^3	3.03×10^3	4.42×10^4	3.03×10^4
J_c (15 K, 3 T) (A cm ⁻²)	5.74×10^2	1.67×10^3	2.98×10^4	2.14×10^4
$F_{p,max}$ at 5 K (N m ⁻³)	1.24×10^8	1.78×10^8	19.11×10^8	14.22×10^8
$F_{p,max}$ at 10 K (N m ⁻³)	0.96×10^8	1.38×10^8	18.34×10^8	13.08×10^8
$F_{p,max}$ at 15 K (N m ⁻³)	0.33×10^8	0.96×10^8	16.01×10^8	11.69×10^8

sample 650L at all measurement temperatures are higher than the values of sample 650R (see Table 1), indicating that the laser irradiation enhances the volume pinning force and flux pinning capability of the sample produced at 650 °C. However, the pinning performance of 850L sample is degraded by the laser irradiation as compared with 850R sample due to the decrement in the value of pinning centres per unit volume as a consequence of the decreased total surface area of the powder grain results from the formation of porosity after the irradiation, as shown in Fig. 3h. It is well known that the high occupation fraction of pores in MgB₂ bulk samples decreases the density and the effective cross-section area for carrying current [32]. So, a decrease in flux pinning ability and critical current density is observed.

4 Conclusions

This work presents the effects of continuous laser irradiation on the structural and magnetic properties of bulk MgB₂ samples sintered at two different temperatures of 650 and 850 °C. It was found that the phase composition is not changed by the laser treatment. It implies that the MgB₂ phase is preserved in the partially melted surfaces during the irradiation. The grains of the irradiated samples were significantly grown due to the laser induced sintering. This growing results in an enhancement of interconnectivity among the superconducting grains for the irradiated sample sintered at 650 °C and, on the contrary, a formation of porosity because of the heating up and cooling down without the bulk material being involved for the irradiated sample sintered at 850 °C. The values of T_c^{offset} and ΔT_c were increased about 1 K

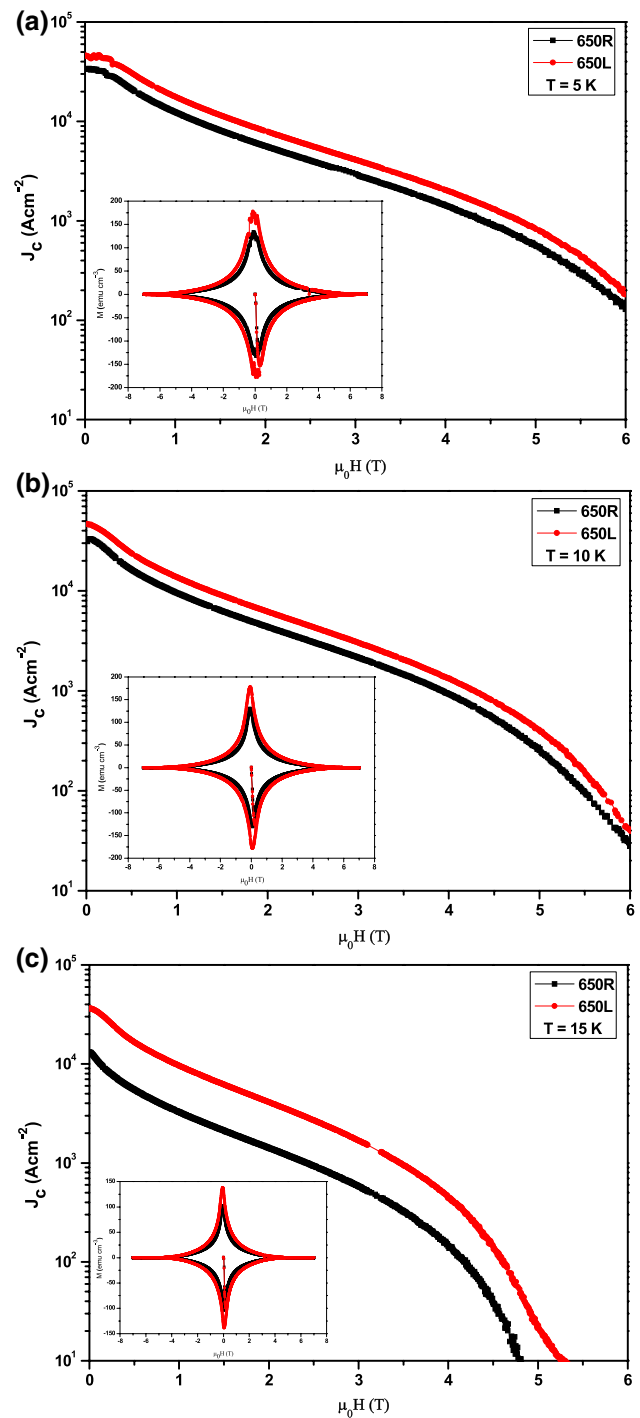


Fig. 5 Magnetic field dependence of critical current density $J_c(B)$ curves of samples 650R and 650L at **a** 5 K, **b** 10 K and **c** 15 K. The inset figures show the magnetization versus magnetic field curves at different temperatures for corresponding samples

for the irradiated sample sintered at 650 °C. It implies the improvement of interconnectivity between the superconducting grains while no change was observed for the other irradiated sample. The critical current density (J_c)

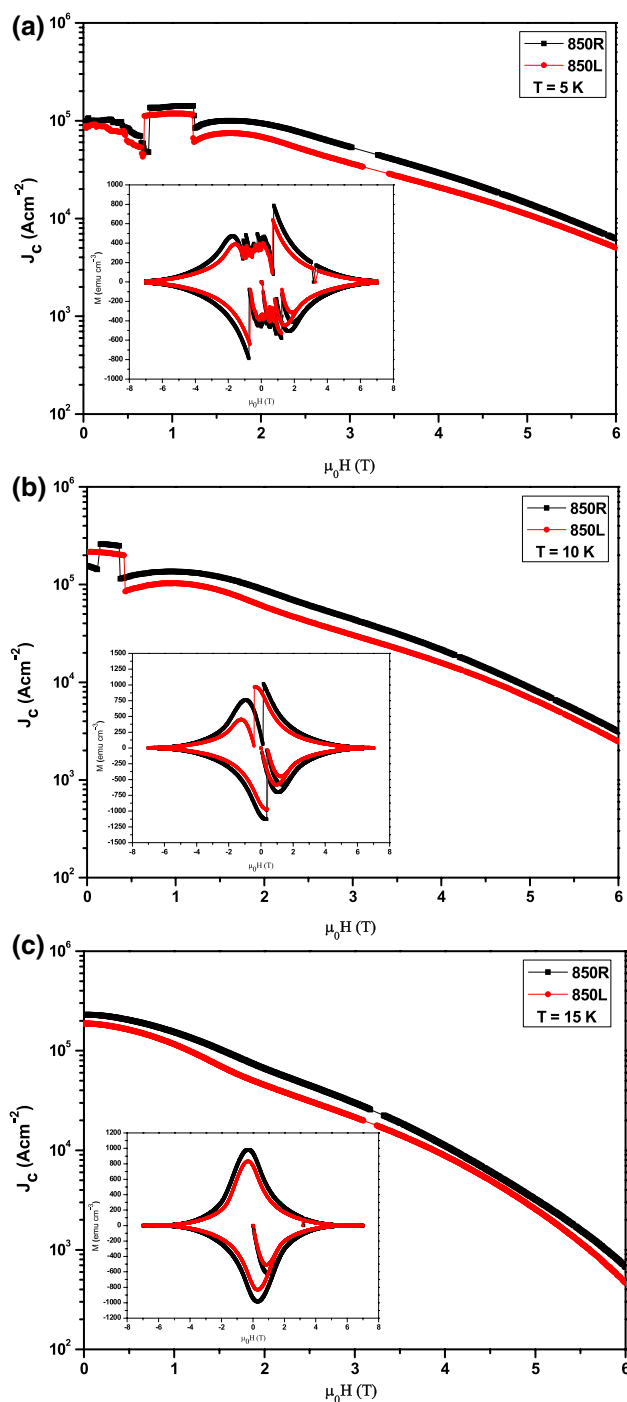


Fig. 6 Magnetic field dependence of critical current density $J_c(B)$ curves of samples 850R and 850L at **a** 5 K, **b** 10 K and **c** 15 K. The inset figures show the magnetization versus magnetic field curves at different temperatures for corresponding samples

and volume pinning force (F_p) were increased for the irradiated sample sintered at 650 °C while an opposite change was observed for the other irradiated sample. On the basis of these results, it can be finally said that the laser irradiation affects the superconducting properties of

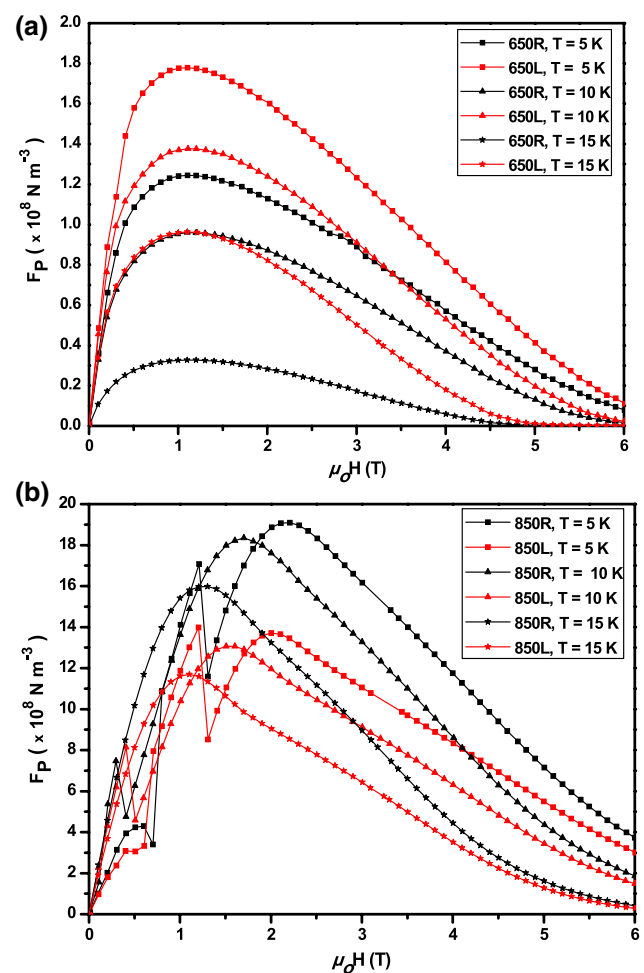


Fig. 7 Volume pinning force as a function of applied magnetic field $F_p(B)$ at 5, 10 and 15 K for samples **a** 650R and 650L and **b** 850R and 850L

bulk MgB_2 depending on the sintering temperature and the initial structural properties of it.

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