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Original Article

# Effects of Ion Irradiations on Crystal Structure and Superconducting Properties of $MgB_2$ Films

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**Abstract:** In this work, variations in the crystal structure and improvements in the superconducting properties of $MgB_2$ films were investigated. The almost pure crystal $MgB_2$ films with thickness of ca 800 nm were successfully prepared by using hybrid physical vapor deposition (HPCVD). The irradiations of Nb and Ni ions were carried out by using an accelerator. The irradiated conditions were set-up at an ion energy of 2 MeV and an ion dose of $5 \times 10^{13}$ ions/cm$^2$. Crystallinity of the pristine and ion-irradiated $MgB_2$ films was examined by using X-ray diffraction (XRD) technique. The temperature-dependent magnetization results showed the degradation of the critical temperature ($T_c$) of the ion-irradiated $MgB_2$ films. Interestingly, the flux pinning properties of both Ni- and Nb-irradiated $MgB_2$ films were found to improve compared to that of the pristine one; those were revealed by the increases in value of irreversibility field ($H_{irr}$) and the enlargements of the area enclosed by the half of hysteresis loops of the ion-irradiated $MgB_2$ films. The value of critical current density ($J_c$) deduced from the hysteresis loop of the ion-irradiated $MgB_2$ films was clearly enhanced, especially at high-field regions. The drop in $T_c$ and the increase in $J_c$ might be due to the creation of disorder defects caused by the ion tracing that happens during ion irradiations.

*Keywords:* $MgB_2$; flux pinning properties; critical current density; ion irradiation.

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## 1. Introduction

Since magnesium diboride ($MgB_2$) was discovered in 2001, it has been considered a promising superconductor for technological and industrial applications. Among the type II superconductors, $MgB_2$ has shown benefits like a high critical temperature of about 39 K, a straightforward crystal structure, a large coherence length, no weak points, low production costs, and a relatively high zero field critical current density of around $10^7$ A/cm². For power applications, high currents have been expected to flow through the MgB2 superconducting wires/cables under high applied fields. However, we have clearly observed the rapid decrease in in-field Jc, which we attribute to the so-called vortex motion [2, 3]. To prevent the vortex motions, the pinning forces-induced via artificial pinning centers-are strongly required [4, 5]. Technologically, there has been widespread use of accelerators to irradiate ions, creating artificial pinning sites in superconducting matrices. In $MgB_2$ films, non-magnetic ions have been irradiated at low energies. The creation of tiny defects, like lattice disorders and ion tracks, has been shown to improve the in-field $J_c$, irreversibility field ($H_{irr}$), and upper critical field ($H_{c2}$) effectively. However, the irradiations of $MgB_2$ films using Ni and Nb ions have not been studied in Vietnam. In this work, the effect of 2 MeV irradiations of Ni and Nb ions on the crystal structure and superconductivity of highly c-axis-oriented $MgB_2$ films will be investigated. Clear improvements in $J_c$ and Hirr were seen in the $MgB_2$ films that were irradiated with Ni and Nb ions.

## 2. Experiments

The c-axis oriented pristine $MgB_2$ films with the thickness of ~ 800 nm were grown on $Al_2O_3$ substrates by using the hybrid physical-chemical vapor deposition system (HPCVD). the films were exposed to 2 MeV $Ni^{2+}$ ions and 2 MeV $Nb^{2+}$ at room temperature with doses of 5 x $10^{13}$ ion/cm$^2$ by using the HUS-5SDH-2 tandem pelletron accelerator The X-ray diffractometer Miniflex 600 Rigaku system with Cu–Kα radiation was used to examine crystallinity of the fabricated samples. Values of $T_c$ of the $MgB_2$ films before and after ion irradiations were estimated from the magnetization versus temperature (M-T) curves, and the $J_c$ was deduced from the magnetization versus field (M-H) curves using Bean's critical state model. All M-T and M-H curves were measured by Quantum Design Physical Property Measurement System (PPMS) EverCool II systems, model 6000.

## 3. Results and Discussions

The crystallinity of the fabricated $MgB_2$ films is investigated by using XRD measurement with 2θ angle varied from 20° to 60°. XRD results given in Fig. 1(a) revealed the fact that pristine and Ni- and Nb-irradiated MgB2 films are highly c-axis oriented, which was evidenced by the dominant (*0001*) and (*0002*) peaks [8-10]. The impurity phases were clearly absent, which might indicate that the Ni and Nb ions were irradiated throughout the $MgB_2$ films' thickness. The typical (0002) peak was selected to analyze possible change in crystallinity [8, 11-13]. The value of full width at half maximum (FWHM) taken from the (0002) peak was found to increase from 0.2º (pristine) to 0.6º (Ni-irradiated) and 0.7º (Nb-irradiated). The obtained FWHM data might suggest the degradation in the crystallinity of both Ni- and Nb-irradiated $MgB_2$ films. Moreover, a slight shift of typical (*000l*) $MgB_2$ peaks to lower values of 2θ was also found in Ni- and Nb-irradiated $MgB_2$ films, predicting a systematic increase in c-axis parameters. Calculations of the c-axis parameters of the $MgB_2$ samples were carried out by using the formula [14]:

$$\frac{1}{d_{hkl}^2} = \frac{4}{3}\left(\frac{h^2 + h.k + k^2}{a^2}\right) + \frac{l^2}{c^2} \quad (1)$$

Obvious increases in c-axis parameters were found: from 3.5210 Å (pristine) to 3.5305 Å (Ni-irradiated) and 3.5258 Å (Nb-irradiated), those evidencing that the irradiations were successfully performed in $MgB_2$ films [13, 15, 16].

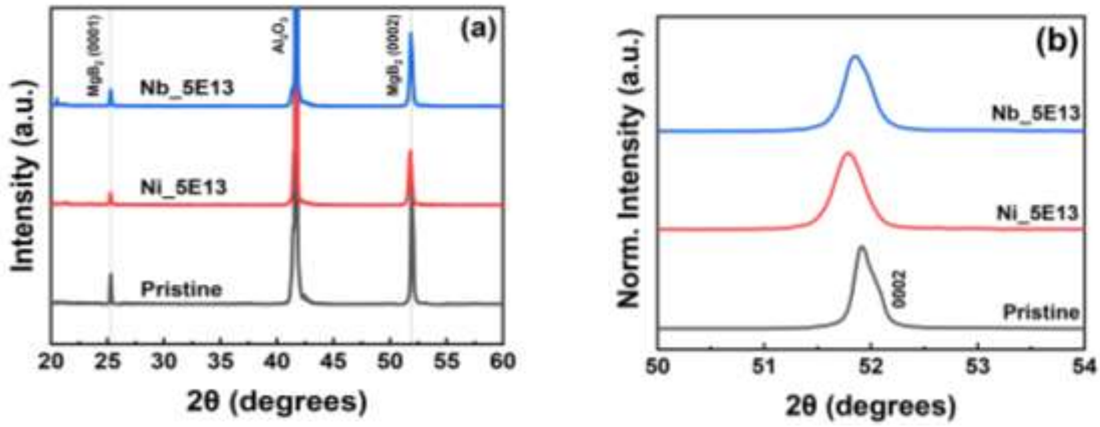


Figure 1. (a) XRD 2θ spectra of Ni and Nb ion-irradiated $MgB_2$ thin films with c-axis orientation; (b) Normalized XRD 2θ spectra of Ni and Nb ion-irradiated $MgB_2$ thin films.

There have been several ways to examine the possible degradation in microstructure of the fabricated samples. The surface SEM or AFM images have been shown to be effective in analyzing the possible change in surface morphology of the fabricated samples. The ions in the irradiation processes in our work were expected to penetrate through the film thickness, which might generate the micro-dislocation. The surface SEM or AFM images might not be needed if we expect to see the ion-tracks. The estimation of density of pinning centers by TEM were mainly carried out when the separated nano-rods or nano-columns were created by using the ion energies of ~ GeV or TeV. In our work, the ion irradiation using lower energy < 3 MeV was expected to generate the ion track as revealed in the following simulation image. So, the average size of the ion tracks was tiny leading to the accurate estimation of ion track density has been shown to be a difficult task. Hence, the application of TEM might not be useful here. The observation of vortex penetration at these pinning centers has been done by using magnetic force microscopy (MFM) [11], and will be done in our future study.

After analysing the crystal structure of the fabricated $MgB_2$ films, their superconductivity was examined. The critical temperature ($T_c$) of the fabricated $MgB_2$ films was obtained from the temperature dependent magnetization curves, those were provided in Fig. 2. Since superconductors have been proved to be perfect diamagnetic materials, the normalization M(T)/M(10K) was carried out for all results. The onset temperature ($T_{c,on}$) for the diamagnetic signal was found to decrease for both Ni- and Nb-irradiated $MgB_2$ films. Particularly, the $T_{c,on}$ of the Ni-irradiated sample was slightly lower than that of Nb-irradiated one. The result might suggest that more disorder defects were generated for Ni-irradiation cases. Values of $T_c$ of all samples were determined by taking the position of the peak in dM/dT curve in Fig. 2(b). Similar to $T_{c,on}$, value of $T_c$ was found to degrade for both Ni- and Nb-irradiated $MgB_2$ films. Estimated data were listed in Table 1.

Fig. 3 revealed the half of hysteresis loops of the $MgB_2$ films – on the positive applied fields - with different ion irradiations under the fields applied perpendicular to the film's surface were measured at 10 K and 20 K. Typical hysteresis property of type-II superconductors were clearly observed. For both

Ni- and Nb-irradiated $MgB_2$ films, the expansions of hysteresis loops towards higher applied fields were found [17, 18]. The Nb-irradiated $MgB_2$ films showed the overall enlargement of the area enclosed by the half of hysteresis loops, which might present the improvements of flux – pinning property in that sample [17-20]. Values of the irreversibility field ($H_{irr}$)– the field at which the hysteresis process started – were also listed in Table. 1. The Nb-irradiated $MgB_2$ films also had the highest enhancement of $H_{irr}$.

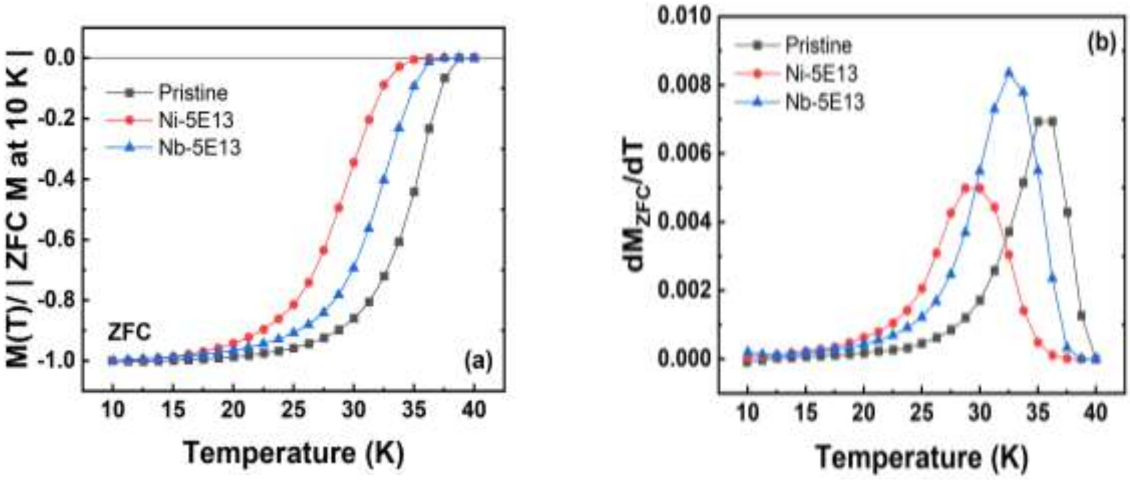


Figure 2. (a) Normalized temperature-dependent magnetizations of Pristine Ni and Nb irradiated films;
(b) First derivative of temperature-dependent magnetizations ($dM_{ZFC}/dT$) of Pristine Ni and Nb irradiated films.

Table. 1. Summary of results for MgB2-thin film samples: onset critical temperature (Tc,on), critical temperature (Tc), full width at half maximum (FWHM) of (0002) peak, c-axis lattice parameter (c-axis) and irreversible field (Hirr).

| Sample | FWHM (degrees) | c-axis (Å) | $T_{c,on}$ (K) | $T_c$ (K) | $H_{irr}$ at 10 K (T) | $H_{irr}$ at 20 K (T) |
|---|---|---|---|---|---|---|
| Pristine | 0.26 | 3.5210 | 38.75 | 36.26 | 2.18 | 1.54 |
| Ni_5E13 | 0.34 | 3.5305 | 36.26 | 30.01 | 4.45 | 2.91 |
| Nb_5E13 | 0.32 | 3.5258 | 37.50 | 32.50 | 4.92 | 3.03 |

Fig. 4 showed the change in $J_c$ of the $MgB_2$ films with different ion irradiations under the fields applied perpendicular to the film's surface at 10 K and 20 K. Values of $J_c$ were deduced from the half of hysteresis loops (on the positive field) by using the equation of the Bean's model [21]:

$$J_c = \frac{30\Delta M}{r}, \tag{2}$$

In this equation, ΔM was the difference between magnetization value between upper and lower branches of (M-H) curve at each applied field and r was taken as the average of length and width of the sample. As seen in Fig. 4 (a-b), the relatively large values of zero-field $J_c$ and the fast reduction in magnetic fields for all $MgB_2$ films revealed that the pristine and ion irradiated $MgB_2$ films were high quality. The self-field $J_c$ of both Ni- and Nb-irradiated $MgB_2$ films was slightly enhanced in comparison with that of pristine one. More interestingly, the enhancements of $J_c$ became obvious as increasing the applied fields. At 10 K, $J_c$ of the pristine film dropped to $10^4$ A/cm$^2$ while that was ~ $10^6$ A/cm$^2$ for Ni- and Nb-irradiated $MgB_2$ films. At 20 K, $J_c$ of the pristine film decreases very quickly when the magnetic field is at 1.5 T while the $J_c$ of the two samples is about $10^5$ A/cm$^2$ and begins to decrease when the magnetic field reaches a value of 2.5 T. When the effect of each irradiation was compared, it would be seen that the $J_c$ enhancement of Nb-irradiated $MgB_2$ film was relatively stronger than that of Ni-irradiated case [17, 18]. The higher applied fields were, the stronger $J_c$ enhancements generated. Nb was a superconductor with $T_c$ ~ 9.7 K. At the two measurement temperatures of 10 K and 20 K, Nb existed in normal state and it was worth saying that Nb-irradiation induced non-magnetic disorders, those effectively served as pinning centers in $MgB_2$ films. Moreover, the $J_c$ was not found to enhance for the whole range of the applied field. According to Higuchi et al., [19], the observations were attributed to the contribution of different types of pinning centers. At low-field regions, the surface pinning centers were predominantly, while the point pinning centers played a more significant role at middle and high-field regions. The exploration of the type of pinning centers is going to perform.

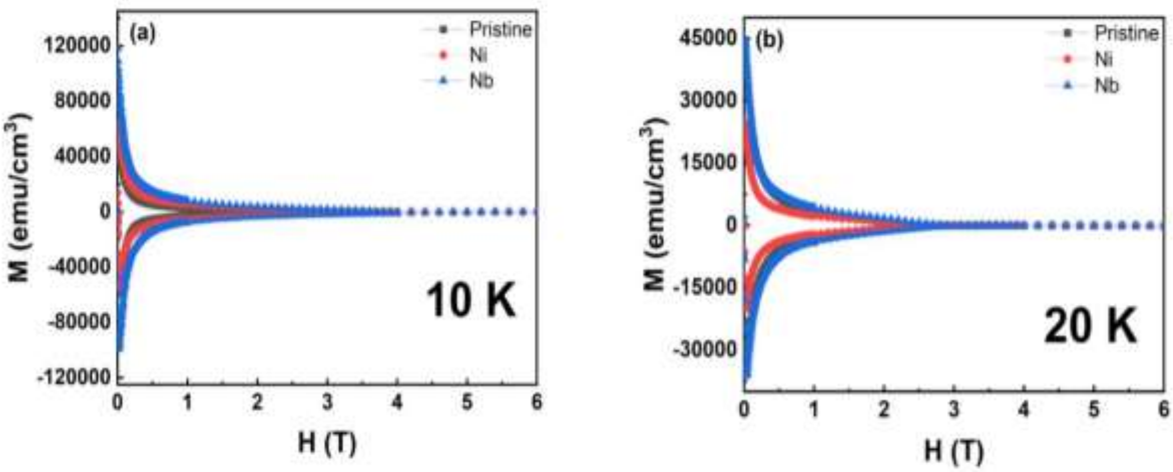


Figure 3. M-H hysteresis loop for $MgB_2$-thin film samples at (a) 10 K and (b) 20 K.

We examined the magnetic field dependency of the flux pinning force density $F_p$ in the $MgB_2$ films at 5E13 irradiation doses in order to look into how Ni and Nb-ion irradiation affected the kind of flux pinning in the $MgB_2$ films. $F_p=\mu_0 H \times J_c$ was the formula used to derive the $F_p$ values from the $J_c(H)$ data. As functions of the decreased magnetic field, which is determined by $h=H/H_{peak}$, where $H_{peak}$ is the magnetic field at which the greatest flux pinning force ($F_{p,max}$) occurred, the reduced flux pinning force densities, which is determined by $f_p=F_p/F_{p,max}$ at 10 and 20 K are shown in Figs. 5 (a) and 5 (b). The following equations explain the three types of pinning models [22]:

$$f_p(h) = \frac{25}{16}\sqrt{h}\left(1-\frac{h}{5}\right)^2 \mathit{for\ surface\ pinning}, \quad (3)$$

$$f_p(h) = \frac{9}{4}h\left(1-\frac{h}{3}\right)^2 \mathit{for\ normal\ point\ pinning}, \quad (4)$$

$$f_p(h) = 3h^2\left(1-\frac{2h}{3}\right) \mathit{for\ \Delta\kappa pinning}, \quad (5)$$

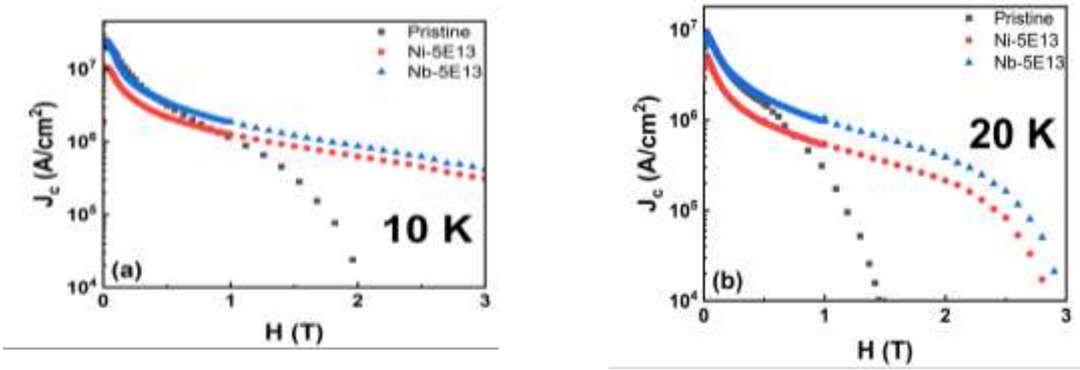


Figure 4. Magnetic field dependence of $J_c$ in Pristine and irradiated $MgB_2$ thin films at (a) 10 K and (b) 20 K.

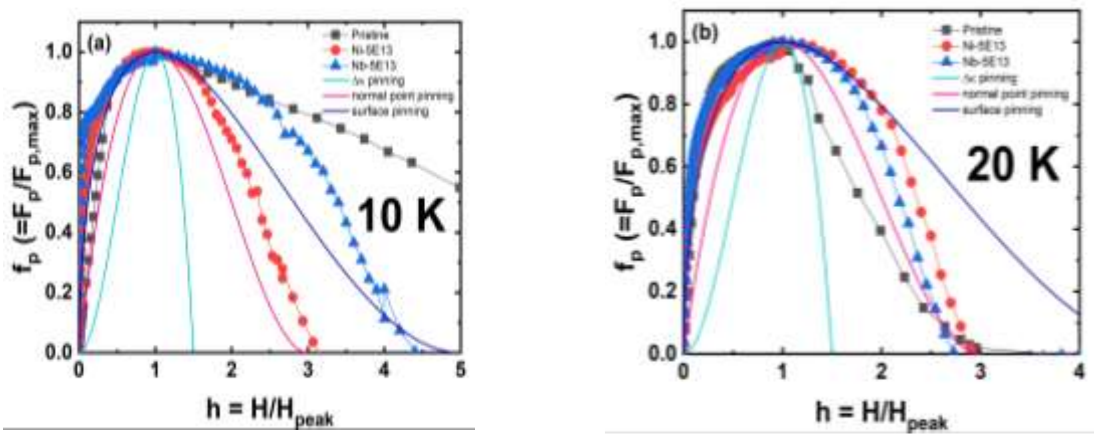


Figure 5. Normalized Fp (fp = Fp/Fp,max) at (a) 10 K and (b) 20 K as functions of reduced magnetic field (h = H/Hpeak).

The $f_p$(h) at both 10 K and 20 K deviated from the three pinning models mentioned above due to the high-quality pristine sample's lack of pinning centers and the impact of vortex-vortex interactions [23]. Whereas the doses of 5E13 with Nb irradiation followed surface pinning, the $f_p$(h) at 10 K for the doses of 5E13 with Ni irradiation followed standard point pinning. In addition, the $f_p$(h) at 20 K for the 5E13 doses under Nb and Ni radiation followed standard point pinning. The thermal fluctuations at 20 K were identified as the cause of the difference between 10 K and 20 K. For the pristine sample, the dominant pinning mechanism was identified to be normal surface pinning. For the Ni-irradiated and Nb-irradiated samples, the pinning mechanism was found to partially shift to the normal point pinning. The formation of point pinning was believed to have resulted from the Ni and Nb irradiation. Besides, other lattice distortions and vacancies might be the evenly distributed defects in the $MgB_2$ films [24, 25]. In combination with the field dependence of $J_c$, the normal point pinning was again proved to effectively work at high field regions, which might be beneficial for the in-field power application of MgB2 films. The variations in $T_{c,on}$, $T_c$, $H_{irr}$ have been proved to be strongly dependent on the existence of disorder defects in the superconductors. When more disorder defects were formed, the lower values $T_{c,on}$, $T_c$ were degraded while the values of $J_c$ and $H_{irr}$ were enhanced since the defects effectively worked as pinning centers. And the pinning centers in our works have been identified to be point pinning type, so the accurate estimation of ion track density has been shown to be a difficult task. The quantitative correlation between $T_{c,on}$, $T_c$, $H_{irr}$ versus defect density would not be easy to obtain. Another thing, the aim of the current work is just comparing the effect of irradiation using different types of ions on the flux pinning properties. The systematic study on each type of ion (such energy scan, ion dose scan) will be carried out in our next research (to be submitted for publishing).

## 5. Conclusion

In summary, we presented results in the study of effects of Ni and Nb ion irradiationson the crystal structure and superconducting properties of $MgB_2$ films. Using hybrid physical vapor deposition (HPCVD) technique, we successfully deposited nearly single-crystalline $MgB_2$ films with a thickness of approximately 800 nm. The irradiation of Nb and Ni ions was carried-out by using an accelerator with the irradiation parameters of a 2 MeV-ion energy and $5\times10^{13}$ ions/cm$^2$ ion dose. The obtained results showed that the critical temperature ($T_c$) of the Ni- and Nb-irradiated $MgB_2$ films decreased. Surprisingly, the ability of both Ni- and Nb-irradiated $MgB_2$ films to hold magnetic fields was better than that of the pristine film. Namely, the value of $H_{irr}$ was increased, and the area enclosed by the half of the hysteresis loop was enlarged. The hysteresis loop of both Ni- and Nb-irradiated MgB2 films clearly enhanced the value of $J_c$, especially at high-field regions. The drop in $T_c$ and the increase in $J_c$ may be attributed to the creation of disorder defects resulting from the ion tracing that occurs during ion irradiations.

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