

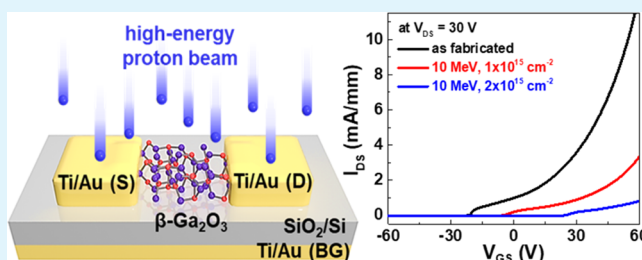
# Influence of High-Energy Proton Irradiation on $\beta$ -Ga<sub>2</sub>O<sub>3</sub> Nanobelt Field-Effect Transistors

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## S Supporting Information

**ABSTRACT:** The robust radiation resistance of wide-band gap materials is advantageous for space applications, where the high-energy particle irradiation deteriorates the performance of electronic devices. We report on the effects of proton irradiation of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelts, whose energy band gap is  $\sim 4.85$  eV at room temperature. Back-gated field-effect transistor (FET) based on exfoliated quasi-two-dimensional  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelts were exposed to a 10 MeV proton beam. The proton-dose- and time-dependent characteristics of the radiation-damaged FETs were systematically analyzed. A 73% decrease in the field-effect mobility and a positive shift of the threshold voltage were observed after proton irradiation at a fluence of  $2 \times 10^{15}$  cm<sup>-2</sup>. Greater radiation-induced degradation occurs in the conductive channel of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt than at the contact between the metal and  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>. The on/off ratio of the exfoliated  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> FETs was maintained even after proton doses up to  $2 \times 10^{15}$  cm<sup>-2</sup>. The radiation-induced damage in the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>-based FETs was significantly recovered after rapid thermal annealing at 500 °C. The outstanding radiation durability of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> renders it a promising building block for space applications.

**KEYWORDS:** gallium oxide, wide-band gap semiconductors, proton irradiation, two-dimensional materials, thermal annealing



## INTRODUCTION

Radiation hardness of semiconductor devices is indispensable for space missions because these devices are continuously exposed to fluxes of various energetic particles, including high-energy protons, electrons, neutrons, and heavy ions in space.<sup>1,2</sup> The electronics in spacecraft performing tasks in earth's orbit must consider three major sources of radiation: (i) galactic cosmic rays, (ii) energetic particles trapped in the van Allen belts, and (iii) solar particle events (SPEs). Among these, SPEs contain high-energy protons ( $>10$  MeV) at a typical fluence of  $10^{10}$  cm<sup>-2</sup>, and the occurrence of 1–2 SPEs every solar cycle is predicted.<sup>3,4</sup> Because protons constitute the primary component of the three major radiation sources, we investigated the influence of high-energy proton irradiation on  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>, which is an ultrawide-band gap semiconductor material.

Wide-band gap semiconductors are typically applicable in radiation-robust devices because of their high bond strengths.<sup>5–9</sup> The unique properties of wide-band gap materials, including their high-temperature capability and strong atomic bonding energy, enable them to operate under harsh conditions.<sup>10–13</sup>  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>, an emerging semiconductor material, has attracted great attention due to its extraordinary characteristics, such as an ultrawide energy band gap of  $\sim 4.85$  eV, a large breakdown electric field of  $\sim 8$  MV/cm, and a high saturation

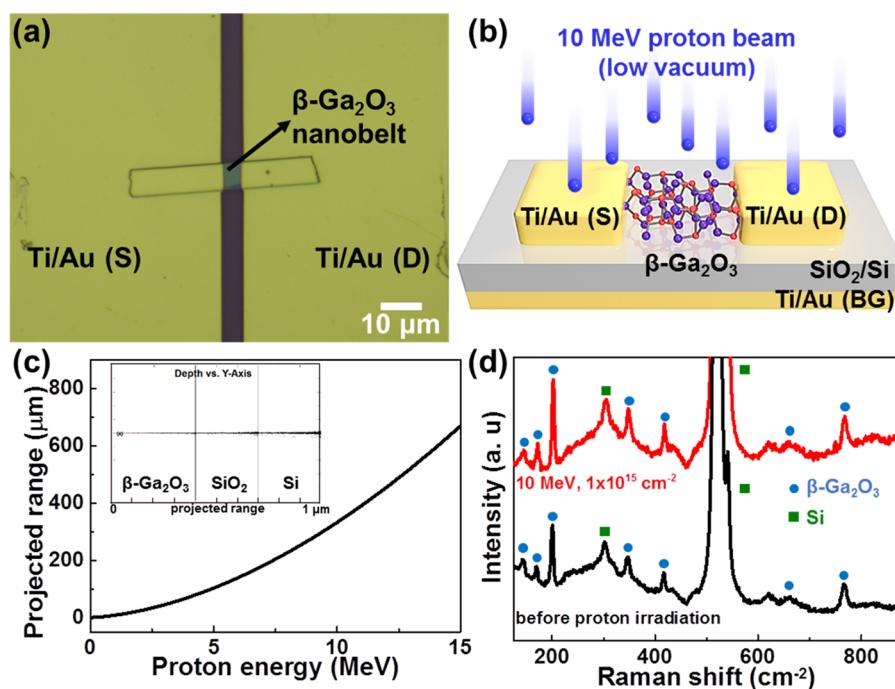
electron velocity of  $2 \times 10^7$  cm/s.<sup>14,15</sup> These desirable properties have allowed the application of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> in solar-blind photodetectors, high-breakdown Schottky diodes, and high-power field-effect transistors (FETs).<sup>16–18</sup>

Furthermore,  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> is currently receiving tremendous attention in the field of nanoelectronics because it can be mechanically cleaved from a freestanding substrate, although it is not a van der Waals material. Back-gated FETs, depletion/enhancement mode FETs, solar-blind photodetectors, and high-temperature FETs have been fabricated using mechanically exfoliated  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>.<sup>19–22</sup> However, radiation effects on nanoscale  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> have not been investigated despite its higher formation energy of vacancy defect (for Ga vacancy in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>, it is 53.3 eV) compared to that of other wide-band gap materials (7.02 eV for GaN (Ga vacancy) and 3.3 eV for 3C-SiC (Si vacancy)).<sup>23–25</sup> Only few studies have been reported on the radiation tolerance of bulk and thin-film  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>. The effects of 1.5 MeV electron irradiation on bulk  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> vertical rectifiers and 5 MeV proton irradiation damage on  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>-based photodetectors have been reported.<sup>26,27</sup> These  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>-

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**Figure 1.** (a) Optical microscopy image of a  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FET, (b) schematic of the proton irradiation on a  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FET, (c) projected range of the proton on bulk  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> estimated by Stopping and Range of Ions in Matter (SRIM) simulation (inset shows the penetration depth profile of 10 MeV protons on  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub>/Si (thickness of 400/300/300 nm)), (d) Raman spectra of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt before and after proton irradiation (10 MeV,  $1 \times 10^{15}$  cm<sup>-2</sup>).

based devices display robust radiation hardness, which is comparable to that of GaN. In this article, we report on the effects of 10 MeV proton irradiation at various doses and subsequent thermal annealing on  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt-based FETs. The electrical and structural properties of these quasi-two-dimensional (2D)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelts were analyzed before and after proton irradiation and subsequent thermal annealing.

## EXPERIMENTAL DETAILS

**Fabrication of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> Nanobelt FETs.** Quasi-2D  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelts were separated from an unintentionally n-doped single crystalline  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> substrate (Tamura Corporation) by a mechanical exfoliation method, where the facile cleavage planes are (100) and (001). The nanobelts were transferred onto a SiO<sub>2</sub>/p<sup>++</sup> Si (300 nm/525  $\mu$ m) substrate; a back-gate electrode (Ti/Au (20/80 nm)) was predeposited on the backside of the substrate using an electron-beam evaporator. Source and drain electrodes (Ti/Au (50/100 nm)) were defined by standard photolithography. The optical microscopy image of the fabricated  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt-based FET is shown in Figure 1a.

**Characterization.** The thickness of the quasi-2D  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt was measured by atomic force microscopy (Innova, Bruker). The effects of proton irradiation on the electrical and optical properties of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt were investigated using a semiconductor parameter analyzer (4155C, Agilent) connected to a probe station and micro-Raman spectrometry with a diode-pumped solid-state 532 nm laser (Omicron-Laserage) under back-scattering geometry, respectively. Rapid thermal annealing (RTA, MILA-3000, ULVAC-RIKO) was performed at 400 and 500  $^{\circ}$ C for 30 s under N<sub>2</sub> atmosphere to eliminate the damage induced by the proton irradiation.

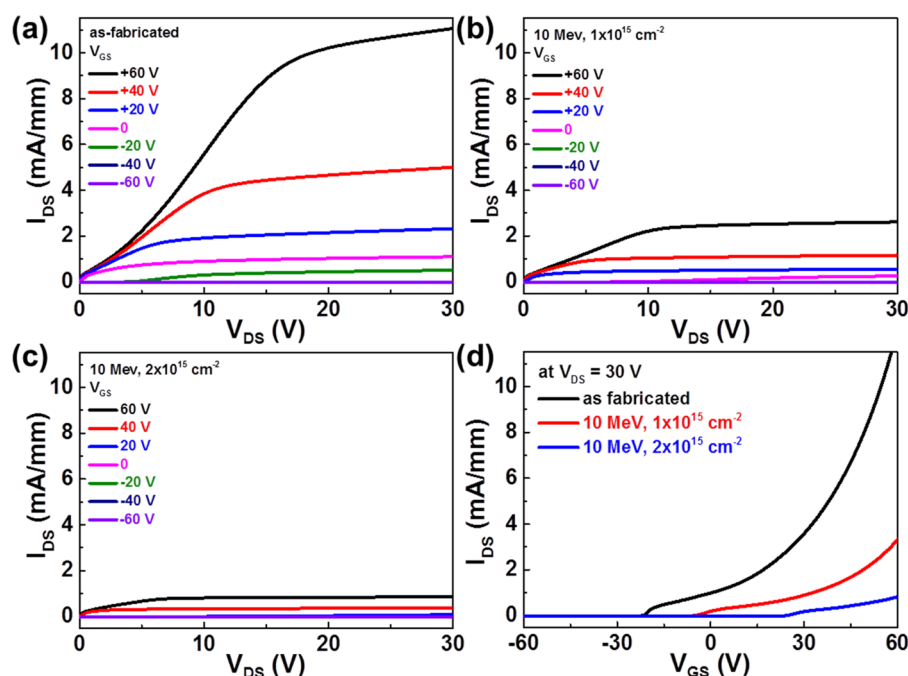
**Proton Irradiation.** Figure 1b shows a schematic of the quasi-2D  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt-based FET, which was subjected to high-energy proton fluxes. The 10 MeV proton beam was generated using the MC-50 Cyclotron at the Korea Institute of Radiological and Medical Science. The proton beam was injected into a low-vacuum chamber, in which the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>-based devices were loaded, facing the proton beam. The average beam current, measured by a Faraday cup, was 100 nA during the proton irradiation process. Proton fluence, which were

controlled by the duration of proton irradiation, were up to  $2 \times 10^{15}$  cm<sup>-2</sup>. The duration of proton irradiation was 1018 and 2036 s, which corresponds to the fluence of  $1 \times 10^{15}$  and  $2 \times 10^{15}$  cm<sup>-2</sup>, respectively. The proton beam uniformity was characterized by using a radiation sensitive film (Gafchromic MD-V2). The proton beam was uniformly emitted from MC-50 Cyclotron over an area of 1 cm  $\times$  1 cm. The electrical and optical characterizations were performed 3 days after the proton irradiation because of safety regulations. The projected range of the 10 MeV proton beam was calculated using the Stopping and Range of Ions in Matter (SRIM) program.<sup>28</sup>

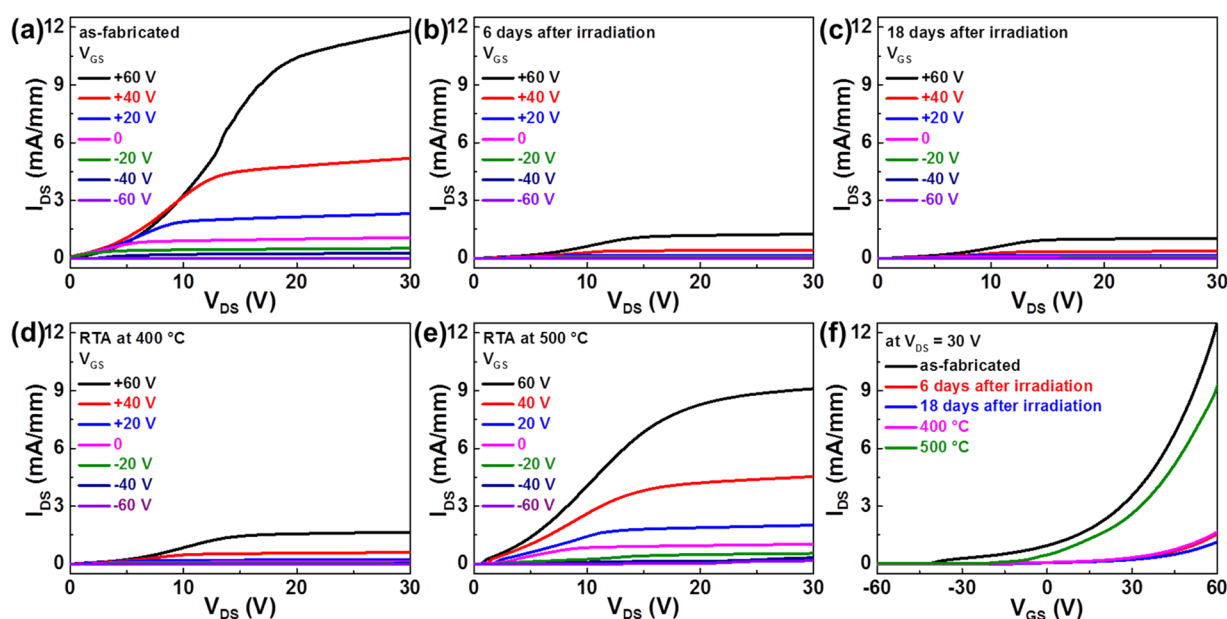
## RESULTS AND DISCUSSION

Figure 1c shows the projected range of the proton beam, estimated from the SRIM program. The penetration depth of the 10 MeV proton beam for  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> is approximately 332  $\mu$ m, which exceeds the thickness of the exfoliated  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> flake ( $\sim$ 400 nm). Therefore, most of the protons pass through the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> (thickness of 400/300 nm) structure, as shown in the inset of Figure 1c. These simulation results indicate that the defects induced by the 10 MeV proton beam should be uniformly distributed throughout the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> flake. The proton-induced damage was also evaluated by micro-Raman spectroscopy, before and after 10 MeV proton irradiation. The Raman spectra of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> flakes show several Raman active modes, which correspond to  $\sim$ 142 (B<sub>g</sub>),  $\sim$ 169 (A<sub>g</sub>),  $\sim$ 201 (A<sub>g</sub>),  $\sim$ 348 (A<sub>g</sub>),  $\sim$ 416 (A<sub>g</sub>),  $\sim$ 662 (A<sub>g</sub>), and  $\sim$ 767 (A<sub>g</sub>) cm<sup>-1</sup>, as shown in Figure 1d.<sup>29</sup> No noticeable change in these modes was observed after proton irradiation, which indicates that defects generated by the proton flux did not produce any strain-induced Raman shift.

Proton-dose-dependent output (drain-source current vs drain-source voltage,  $I_{DS}$ - $V_{DS}$ ) and transfer characteristics (drain-source current vs gate-source voltage,  $I_{DS}$ - $V_{GS}$ ) of the quasi-2D  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FET with back-gate configuration are shown in Figures 2 and S1. Its output characteristics with



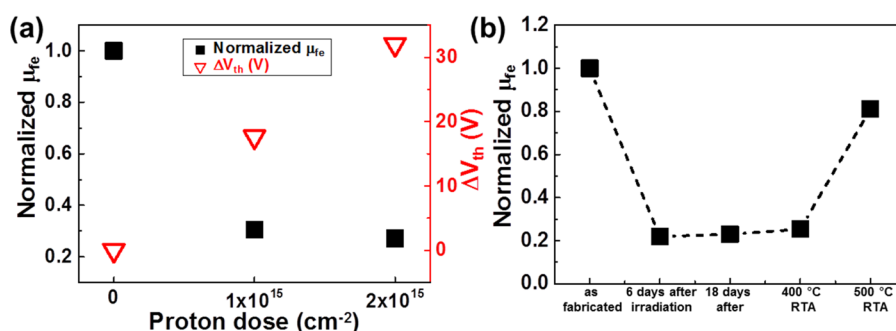
**Figure 2.** Output characteristics ( $I_{DS}$ – $V_{DS}$ ) of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FET before and after 10 MeV proton irradiation at different doses: (a) as fabricated, (b)  $1 \times 10^{15} \text{ cm}^{-2}$ , and (c)  $2 \times 10^{15} \text{ cm}^{-2}$ . (d) Transfer characteristics ( $I_{DS}$ – $V_{GS}$ ) of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FET at  $V_{DS} = +30 \text{ V}$  before and after 10 MeV proton irradiation at different doses.



**Figure 3.** Output characteristics of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FET with time and temperature of RTA after 10 MeV proton irradiation at a fluence of  $1 \times 10^{15} \text{ cm}^{-2}$ : (a) as fabricated, (b) 6 days after proton irradiation, (c) 18 days after proton irradiation, (d) after RTA at  $400^\circ\text{C}$ , and (e) after RTA at  $500^\circ\text{C}$ . (f) Time and temperature dependent transfer characteristics of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FET at  $V_{DS} = +30 \text{ V}$ .

time and temperature of RTA after 10 MeV proton irradiation at a fluence of  $1 \times 10^{15} \text{ cm}^{-2}$  are represented in Figure 3. A typical n-type behavior, with a decrease in  $I_{DS}$  with decreasing  $V_{GS}$ , was observed in the as-fabricated devices. In general, unintentionally doped  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> shows n-type conductivity owing to its oxygen vacancies and unintentional impurities.<sup>30,31</sup> The conductivity of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> flakes decreased with increasing the proton dose to  $2 \times 10^{15} \text{ cm}^{-2}$ . The removal of the free carriers due to the introduction of radiation-induced deep traps leads to the deterioration of the electrical

conductivity,<sup>1,2,32</sup> as reported previously for samples irradiated with both electrons and protons.<sup>26,27</sup> Another electrical parameter influenced by the proton irradiation is the field-effect electron mobility ( $\mu_{fe}$ ).  $\mu_{fe}$  of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FET was estimated by the following equation,  $\mu_{fe} = (g_m \cdot L) / (W \cdot C \cdot V_{DS})$ , where  $g_m$ ,  $L$ ,  $W$ , and  $C$  are the transconductance, channel length, channel width, and oxide capacitance, respectively. The  $\mu_{fe}$  value decreased from 2.9 to  $0.78 \text{ cm}^2/\text{Vs}$  with an increasing the proton dose to  $2 \times 10^{15} \text{ cm}^{-2}$ . The normalized  $\mu_{fe}$  as a function of proton doses is shown in Figure 4a.  $\mu_{fe}$  was reduced



**Figure 4.** (a) Normalized  $\mu_{fe}$  and  $\Delta V_{th}$  as a function of proton doses, (b) RTA-dependent normalized  $\mu_{fe}$  with respect to time and temperature.

by 73% after proton irradiation at a fluence of  $2 \times 10^{15} \text{ cm}^{-2}$ . The decrease of  $\mu_{fe}$  can be explained by increased Coulombic scattering by the radiation-induced defects.<sup>2,5</sup> A positive shift in the threshold voltage ( $\Delta V_{th}$ ) and reduction of  $I_{DS}$  were observed in the  $I_{DS}$ – $V_{GS}$  curve, as shown in Figures 2d and S1.  $\Delta V_{th}$  increased with increasing proton dose, which can be explained by the removal of electrons by trapping and introduction of acceptor-type radiation-induced states. The  $\Delta V_{th}$  can be estimated by the sum of the  $V_{th}$  shift caused by the oxide trap ( $V_{ot}$ ) and interfacial trap ( $V_{it}$ ), which can be expressed as  $\Delta V_{th} = \Delta V_{ot} + \Delta V_{it}$ . For n-channel transistors, an increase in the interfacial trap density results in a positive shift in  $V_{th}$ , whereas an increase in the oxide trap density typically leads to a negative shift in  $V_{th}$ .<sup>33–35</sup> Because the proton-irradiated quasi-2D  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FETs showed a positive shift in the  $V_{th}$ , the effect of the interfacial traps seems dominant compared to that of the oxide traps. Although proton irradiation induced a large change in  $\mu_{fe}$ ,  $V_{th}$ , and the drain–source on-resistance ( $R_{DS(on)}$ ) values (measured in the linear region before knee voltage), only a nominal change in the on/off ratio was observed, as shown in Figure S1. Proton-dose-dependent electrical parameters,  $\mu_{fe}$ , normalized  $\mu_{fe}$ ,  $R_{DS(on)}$ ,  $V_{th}$ , and the on/off ratio, are summarized in Table 1. The

**Table 1.** Summary of the Electrical Parameters of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> Nanobelt FET before and after 10 MeV Proton Irradiation at Different Doses

	as fabricated	10 MeV, $1 \times 10^{15} \text{ cm}^{-2}$	10 MeV, $2 \times 10^{15} \text{ cm}^{-2}$
field-effect mobility ( $\mu_{fe}$ , $\text{cm}^2/\text{V s}$ )	2.9	0.88	0.78
normalized $\mu_{fe}$	1	0.31	0.27
$R_{DS(on)}$ (k $\Omega$ mm)	1.4	4.8	11
$V_{th}$ (V)	–8.2	9.4	23.7
on/off ratio	$7.0 \times 10^7$	$2.3 \times 10^7$	$7.5 \times 10^7$

degradation of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FETs caused by proton irradiation is comparable to those of AlGaIn/GaN/GaN-based high electron-mobility transistors.<sup>36,37</sup> This indicates the great potential of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> for application in radiation-hard semiconductor devices.

The Y-function method was used to evaluate the effects of the high-energy proton irradiation on the channel resistance ( $R_{ch}$ ) and contact resistance ( $R_c$ ) of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FETs. The Y-function method is applicable to 2D material-based FETs because it enables an assessment of the transport characteristics of nanoscale materials. The Y-function method that was originally developed to analyze a metal-oxide-semiconductor FET allows us to extract the device character-

istics from a transfer curve when  $V_{DS}$  is much smaller than  $V_{GS}$ . The Y-function is defined as,  $Y = I_{DS}/g_m^{1/2} = (\mu_0 \cdot C_{OX} \cdot W \cdot V_{DS}/L)^{1/2} \cdot (V_{GS} - V_{th})$ , where,  $\mu_0$  is the low-field mobility.<sup>38,39</sup> Proton-dose-dependent electrical properties extracted by the Y-function method are summarized in Table 2.  $\mu_0$  decreased by

**Table 2.** Summary of the Electrical Parameters of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> Nanobelt FET Extracted by the Y-Function Method before and after 10 MeV Proton Irradiation at Different Doses

	as fabricated	10 MeV, $1 \times 10^{15} \text{ cm}^{-2}$	10 MeV, $2 \times 10^{15} \text{ cm}^{-2}$
low-field mobility ( $\mu_0$ , $\text{cm}^2/\text{V s}$ )	238	89.7	32.6
normalized $\mu_0$	1	0.38	0.14
$R_{ch}$ ( $\Omega$ mm)	36.7	132	504
$R_c$ ( $\Omega$ mm)	606	716	837
$R_{ch}/R_{total}$ (%)	3	8	23

86% after proton irradiation at a fluence of  $2 \times 10^{15} \text{ cm}^{-2}$ . The  $\mu_0$  value, obtained by the Y-function method is higher than  $\mu_{fe}$  because  $\mu_{fe}$  is generally underestimated owing to  $R_c$ . From the calculated  $\mu_0$  and  $V_{th}$ ,  $R_{ch}$  can be obtained.<sup>40,41</sup> Because the total resistance ( $R_{tot}$ ) is composed of  $R_{ch}$  and  $R_c$ , the contribution of each resistance at different conditions of proton irradiation can be explored. Although both  $R_c$  and  $R_{ch}$  increased after proton irradiation, a noticeable change in  $R_{ch}$  was observed compared to that in  $R_c$ . The proportion of  $R_{ch}$  in  $R_{tot}$  increased from 3 to 23% with increasing proton dose, which indicates that the conductive channel of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> is more susceptible to proton irradiation compared to the contact between the metal and  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>. Therefore, the main cause of the decline in the electrical performances of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FETs is inferred to be radiation-induced damage in its conductive channel.

Radiation-induced defects can often be eliminated by recombining with other defects or impurities, even at room temperature.<sup>1,2</sup> We also investigated the thermal removal of the radiation defects. The time-dependent output and transfer characteristics of the quasi-2D  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FET are shown in Figures 3 and 4b. The value of  $\mu_{fe}$  decreased by 78% after 10 MeV proton irradiation at a fluence of  $1 \times 10^{15} \text{ cm}^{-2}$ . The quasi-2D  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>-based FET showed no change in the electrical properties when it was measured 18 days after the proton irradiation, indicating no self-recovery at room temperature. Further, RTA was performed to accelerate the restoration of the radiation-induced defects. The electrical characteristics of the quasi-2D  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>-based FET recovered dramatically with increasing RTA temperatures. The  $\mu_{fe}$  value reached 81% of its original value after RTA at 500 °C. The  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt



FETs did not operate normally at the higher RTA temperature (600 °C) owing to high-temperature-induced degradation.

## CONCLUSIONS

High-energy protons were used to produce radiation-induced defects in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelts obtained by mechanical exfoliation. Optical and electrical characterizations were performed on the fabricated  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FETs before and after proton irradiation at an energy of 10 MeV, with a fluence of up to  $2 \times 10^{15}$  cm<sup>-2</sup>. The  $\mu_{fe}$  value decreased by 73% after proton irradiation but recovered to 81% of its initial value after RTA at 500 °C. However, the on/off ratio was hardly affected by proton irradiation. The contribution of  $R_{ch}$  to  $R_{total}$  increased with increasing proton dose, which indicates that the degradation in the conductive channel is greater compared to that in the contact between the metal electrode and  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> under proton irradiation. The decline in the electrical performance of the exfoliated  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FETs is comparable to the results previously reported for GaN-based devices. The radiation-induced defects were hardly repaired at room temperature, whereas they were almost fully recovered in terms of the electrical performance after thermal annealing at 500 °C. The robust radiation tolerance of the quasi-2D  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt renders it a potentially useful material for space applications.

## ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsami.7b13881.

Transfer characteristics of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> nanobelt FET before and after 10 MeV proton irradiation (PDF)

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### Notes

The authors declare no competing financial interest.

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