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Influence of neutron radiation on majority and minority carrier traps in n-type 4*H*-SiC

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ABSTRACT

We report on influence of neutron radiation on majority and minority carrier traps in n-type 4*H*-SiC. Together with the increase of the well-known carbon vacancy (V_C) majority carrier related trap, neutron irradiation has introduced two deep traps, labeled as EH1 and EH3 with the activation energies for electron emission estimated as 0.4 and 0.7 eV bellow the conduction band, respectively. Based on Laplace deep level transient spectroscopy (DLTS) results, we have assigned EH1 trap to silicon vacancy (V_{Si}). Two minority carrier traps labelled as B and D-center were detected by minority transient spectroscopy (MCTS) and assigned to substitutional boron B_{Si} and B_C, respectively. Activation energies for hole emission for B and D-center are estimated as 0.27 and 0.60 eV above the valence band, respectively. We have identified two emission lines for D-center by Laplace-MCTS measurements and assigned them to B_C sitting at hexagonal ($-h$) and cubic ($-k$) lattice sites.

1. Introduction

Silicon carbide (SiC) is a wide band gap semiconductor suitable for high temperature, high-frequency and high-power applications [1,2]. Its 4*H* polytype is becoming a mainstream material for very high-voltage applications, bipolar devices such as PiN diodes neutron radiation detectors. Currently, the major problem for 4*H*-SiC bipolar devices is an effects known as “bipolar degradation” [3]. Different approaches have been proposed and applied for dealing with the bipolar degradation. The focus is on the reduction of carrier lifetimes.

Moreover, characterization of radiation induced defects in 4*H*-SiC based radiation detectors is crucial for future improvement of radiation hardness and extending the lifetime of 4*H*-SiC detectors by material engineering. Electrically active defects influence the electrical properties of the semiconductor and in general, cause a deterioration of the detector spectroscopic performance, as recombination of charge carriers via created deep levels decreases the minority carrier lifetime and consequently the charge collection efficiency of a semiconductor detector. An increase in the concentration of a suitable impurity can suppress the formation of the most prominent/influential deep levels and increase the detection efficiency [4,5].

The main lifetime killer is n-type 4*H*-SiC is carbon vacancy (V_C), a defect known as $Z_{1/2}$ center. The $Z_{1/2}$ is one of the most studied defects in SiC and numerous studies regarding the structure, energy, thermal stability etc. have been reported [6–8]. The $Z_{1/2}$ concentration can be increased either by irradiations [6,7] or high-temperature annealing [8], which yields to reduction of carriers lifetime.

Numerous DLTS studies were conducted on 4*H*-SiC after controlled introduction of defects in material by electron and proton irradiation or ion implantation. Assignment of commonly observed deep levels in irradiated n-type 4*H*-SiC is still uncertain. Two deep levels usually labeled as S1/S2 [9,10], S2/S4 [11,12] or EH1/EH3 [6,13–15] with energies around $E_C - 0.4$ eV and $E_C - 0.7$ eV [16,17] can be observed in n-type 4*H*-SiC after electron or proton irradiation and ion implantation.

Contrary to majority carrier traps (such as $Z_{1/2}$), minority carrier traps in n-type 4*H*–SiC are still far from fully exploited. A short list of studies on minority carrier traps in as-grown, electron-irradiated, and thermally oxidized 4*H*–SiC [18–21] has been reported in the literature. Most of the reported minority carrier traps in n-type 4*H*-SiC material are related to boron, which is a typical SiC impurity.

Boron is usually a p-type dopant in SiC but unintentional boron incorporation that occurs during the crystal growth and which is

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explained by the presence of boron in the graphite susceptor used for the CVD growth has already been reported [19,20]. The concentration of unintentionally incorporated boron could reach 1×10^{13} cm^{-3} yielding to the appearance of boron-related deep levels in the bandgap.

Previous experimental studies have reported two boron related energy levels [3,18,19]. One shallow level with an energy around 0.27 eV (labelled as B) and another deep level (labelled as D center) with an energy around 0.56–0.62 eV are observed in n-type 4*H*-SiC for intentional N + B doping [3] and unintentional boron incorporation [19].

These experimental findings are in good agreement with already published theoretical calculations. According to Bockstedte et al. [22] B and D-center are assigned to substitutional boron atoms occupying the Si and C-site, respectively. The shallow state, B_{Si} is off-center substitutional boron at Si-site, while for the deep state Bc, boron occupies a perfect substitutional C site.

Minority carriers in Schottky barrier diodes (SBDs) could be optically generated by use of above-bandgap light [23]. The first experimental application of the generation of minority carriers by use of a light with an energy just above the bandgap energy as a technique for manipulating the occupancy of deep states was described by Hamilton et al. [24] and it was called the minority carrier capture (MCC) method. It was further developed into minority carrier transient spectroscopy (MCTS) by Brunwin et al. [25]. In addition to MCTS, a technique called optical-DLTS, in which the employed light has an energy below the bandgap energy, has been reported [23].

In this work, we present a study on influence of neutron radiation on majority and minority carrier traps in n-type 4*H*-SiC by means of DLTS and MCTS.

2. Experimental

n-type SiC SBDs were produced on nitrogen-doped (up to 4.5×10^{14} cm^{-3}), epitaxially grown 4*H*-SiC single crystal layers, approximately 25 μm thick [26]. A semi-transparent SBDs for MCTS measurements were formed by evaporation of a thin film of nickel through a metal mask with apertures of 2 mm × 2 mm. The film thickness was measured to be 15 nm. Thick film of nickel (1 mm × 1 mm) was stacked in one corner of the thin film for wire bonding of the front contact. Ohmic contacts were formed on the backside of the SiC substrate by nickel sintering at 950 °C in Ar atmosphere.

The produced SBDs were irradiated with epithermal and fast neutrons at the Jozef Stefan Institute (JSI) TRIGA reactor in Ljubljana, Slovenia. Thermal neutrons with energy less than 0.55 eV were filtered by irradiating the Schottky barrier diodes inside a cadmium box with a wall thickness of 1 mm. The neutron energy spectrum in the irradiation location was characterized on the basis of Monte Carlo calculations with the MCNP® code [27] and activation measurements [28]. The total neutron flux in the irradiation location was monitored by activation measurements for the $^{197}Au(n,\gamma)$ reaction for each used power level. The sub-cadmium neutron flux was derived from the characterized neutron spectrum, the cut-off energy of the cadmium box (0.55 eV) and the total neutron flux. The fractions of the epithermal neutron flux (neutron energies in the interval between 0.55 eV and 100 keV) and the fast neutron flux (neutron energies in the interval between 100 keV and 20 MeV) are approximately 51% and 49%. The epithermal range displays typical 1/E behavior with negligible slope ($\alpha \approx 0$ in the parametrization $\varphi(E) \sim 1/E^{1+\alpha}$) and some small peaks and dips due to resonance absorption effects. The fast component follows a typical fission spectrum, with a peak region from around 1 MeV to around 2 MeV. The quality of the SBDs (before and after neutron irradiation) was tested by current-voltage (I-V) and capacitance-voltage (C-V) measurements. The DLTS and MCTS measurements were carried out in the temperature range from 100 up to 450 K. The voltage settings for DLTS and Laplace-DLTS measurements were reverse voltage −10 V, pulse voltage −0.1 V, and pulse width 10 ms. Reverse voltage was constant during the MCTS measurements (mostly −10 V, if not stated otherwise), while excitation light pulses were applied. The samples were cooled down to 100 K from room temperature without applied bias. A 365 nm LED powered by a Thorlabs LDC205C driver was used for optical excitation. Capacitance transients were measured by Boonton 7200. Laplace-DLTS and Laplace-MCTS spectra were calculated by the FLOG numerical routine [29]. The acquisition settings for Laplace-MCTS measurements were: number of samples 1×10^4–3×10^4, sampling rate 4–20 kHz, and number of averaged scans 50–500.

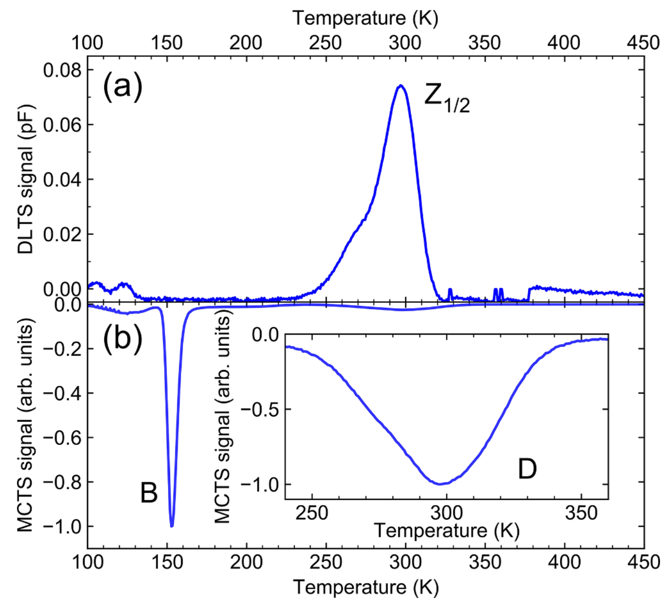

Fig. 1. (a) DLTS spectrum, and (b) MCTS spectrum for as-grown semi-transparent 4*H*-SiC SBDs. Emission rate is 10 s^{-1}.

3. Results and discussion

3.1. As-grown material

Fig. 1a shows a typical DLTS spectrum for the as-grown 4*H*-SiC material. The broad and asymmetric peak with a maximum at around 300 K with emission rate 50 s^{-1} is known as $Z_{1/2}$, and it was assigned to (=/0) transitions of V_C in 4*H*-SiC [30]. Recently, we provided direct evidence that the broad $Z_{1/2}$ peak has two components, namely Z_1 and Z_2, with activation energies for electron emission as $E_c - 0.59$ eV and $E_c - 0.67$ eV, respectively. We assigned these components to (=/0) transition sequences from negative-U ordered acceptor levels of carbon vacancy (V_C) defects at hexagonal and cubic sites, respectively [31,32].

Fig. 1b shows MCTS spectrum with two energy levels labelled as B and D center with the activation energies for hole emission estimated as $E_v + 0.27$ eV and $E_v + 0.60$ eV, respectively. These traps resemble already reported B and D-center [3] which are assigned to substitutional boron at silicon site (B_{Si}) and carbon site (B_c), respectively.

As seen in Fig. 1b, the concentration of B_{Si} (B-center) is significantly higher compared to the concentration of B_C (D-center). The concentration of B-center estimated from MCTS peak amplitude, $\sim 10^{14}$ cm^{-3}, is the same order of magnitude as the doping concentration. If SiC is grown under Si-rich conditions during the CVD, the boron incorporation is lower than in the case of C-rich conditions [33]. Under Si-rich conditions, empty C-sites are available for boron incorporation and B_c (D-center) dominates, while under C-rich conditions, empty Si sites are available for boron and B_{Si} (B) dominates. Our results (Fig. 1b) indicate that SiC was grown under the C-rich conditions.

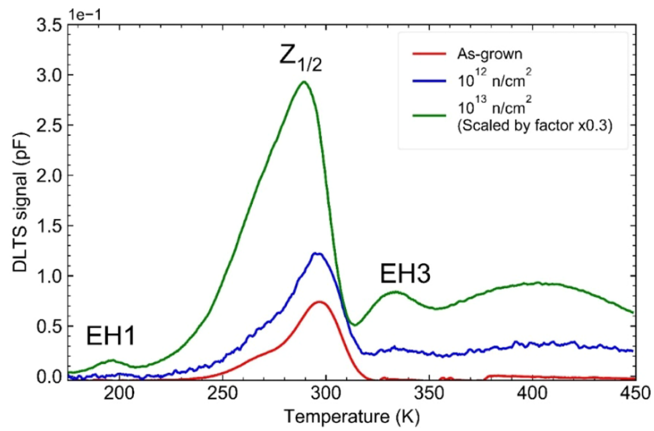

Fig. 2. DLTS spectra for neutron irradiated 4*H*-SiC SBDs. Emission rate is 10 s^{-1}.

3.2. Majority carrier traps

In this section, we will focus on the influence of neutron irradiation on majority carrier traps. Similar to our previously published study [34], two new defects labelled as EH1 and EH3 are introduced. Fig. 2 shows DLTS spectra of neutron-irradiated semi-transparent 4*H*-SiC SBDs. Activation energies for electron emission are estimated as 0.4 and 0.7 eV for EH1 and EH3, respectively.

The origin of EH1 and EH3 traps has puzzled the researchers for many years. The assignment EH1/EH3 radiation induced defects has usually been correlated with C displacements [6], particularly after the low energy electron irradiation. The possibility for the Si atom displacement [6,35] is increasing as the energy of incident particle is increasing. The general agreement is that those traps are related to intrinsic defects.

In the recent work by M.E. Bathen et al. [36] S-center, consisting of two contributions S1 and S2 which are located at 0.4 and 0.7 eV below the conduction band minimum, is reported. The S1 and S2 are introduced in n-type 4*H*-SiC samples by proton irradiation, and their intensities increase by proton fluence. They have been assigned to different charge state transitions of silicon vacancy, V_{Si}.

We have applied Laplace-DLTS to get additional information about the EH1/EH3 defects. While the Laplace-DLTS on EH3 provided us the limited information due to the strong overlap with surrounding defects, the Laplace-DLTS spectrum of the EH1 trap has revealed two components labeled $EH1_1$ and $EH1_2$ (Fig. 3).

From Arrhenius plot of electron emission, we have estimated the activation energies 0.41 and 0.42 eV for two components $EH1_1$ and $EH1_2$, respectively. The intensity ratio between $EH1_1$ and $EH1_2$ is not 1:1 as reported for S1 defect in proton irradiated 4*H*-SiC samples. However, similar effect has been reported for carbon vacancy (V_C) at hexagonal and cubic lattice sites [31,32]. We tentatively assign $EH1_1$ and $EH1_2$ components to silicon vacancy V_{Si} at hexagonal ($-h$) and cubic ($-k$) lattice sites. Our results indicate that $V_{Si}(k)$ is predominantly introduced by neutron irradiation.

3.3. Minority carrier traps

This section is devoted to minority carrier traps and on the influence of neutron radiation on those defects. As seen in Fig. 1b, the unintentionally incorporated boron has introduced two minority carrier traps. This result is consistent with our previously published study on minority carrier traps in nitrogen-doped 4*H*-SiC [18].

We have applied Laplace-MCTS to get additional information about the B and D-center. As previously reported [18], the Laplace-MCTS spectrum for B was too broad and could not provide additional information about B peak (Fig. 4). The measured capacitance transients

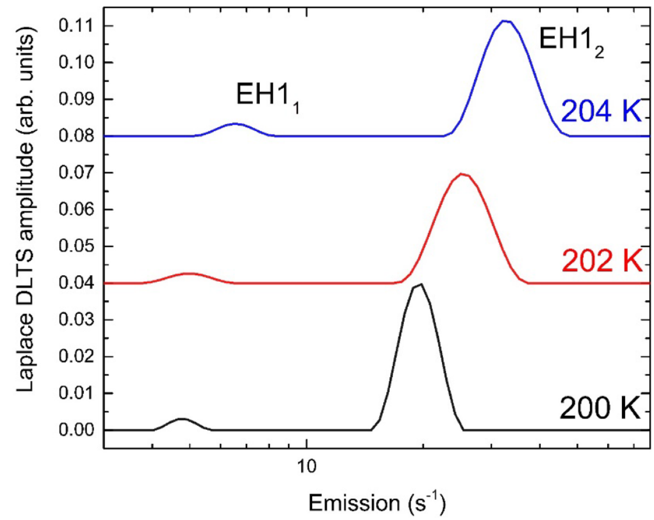

Fig. 3. Laplace DLTS spectra of the neutron-irradiated sample with fluence $10^{13}n/cm^2$ at three subsequent measurement temperatures.

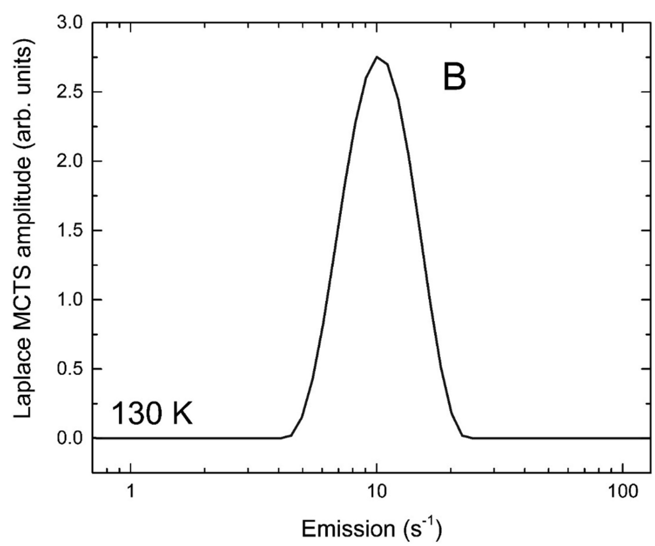

Fig. 4. Laplace-MCTS spectrum at 130 K measured on the neutron-irradiated sample with the fluence of $10^{13}n/cm^2$.

are not well-defined exponential transients, possibly due to a large concentration of B-center which is comparable to donor concentration. Additionally, a difference between the $B_{Si}(k)$ and $B_{Si}(h)$ activation energies could be too small to be resolved by MCTS measurements. Further studies, including EPR measurements and DFT calculation could provide the necessary information.

Fig. 5 shows the changes for B center induced by neutron radiation. The peak maximum is shifting to lower temperatures as the neutron fluence is increasing, while the concentration is at the same order of magnitude. The shift of B-related peak to lower temperatures is most likely due to the change in stress present in the epitaxial layer. It is well-known that neutron irradiation increases tensile stress in 4*H*-SiC due to introduced defects. According to the DFT calculations, the presence of dumbbell defects increases the cell volume, while the introduced vacancies change the volume slightly [37,38]. The stress can affect the emission rate value and cause the level splitting into multiple peaks according to the point symmetry of the defect [39,40].

Contrary to B center, we were able to provide a direct evidence that D-center has two components which we have labelled as D_1 and D_2 by Laplace-MCTS measurements (Fig. 6). Their activation energies are estimated as E_v + 0.49 eV and E_v + 0.57 eV and they are assigned to an

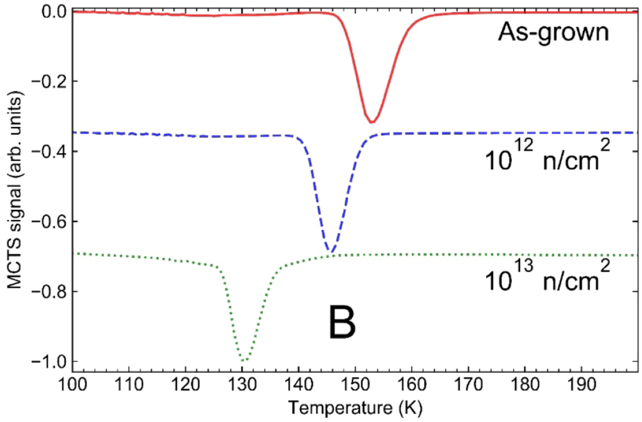

Fig. 5. B center in the MCTS spectra of as-grown and neutron-irradiated samples. Emission rate is 10 s^{-1}. The spectra are shifted vertically for the clarity.

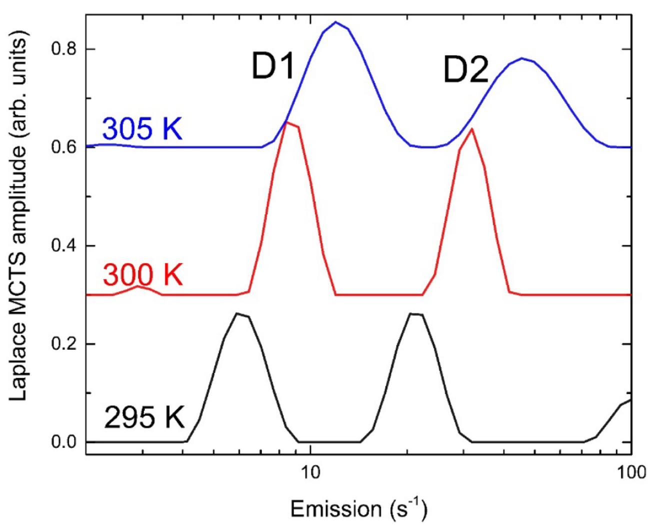

Fig. 6. Laplace-MCTS measurements on the as-grown sample, at constant reverse voltage −2 V.

isolated boron sitting at the C site (−*h* and -*k* site, respectively). The D_1:D_2 intensity ratio is roughly 1:1, suggesting that $B_C(h)$ and $B_C(k)$ sites are equally occupied.

Fig. 7 shows MCTS spectra for D-center upon neutron irradiation. As already observed and discussed for B center (Fig. 5), the D-center peak maximum is shifting to lower temperatures as the neutron fluence is increasing. This behavior is explained by the introduced stress in epitaxial layers. The concentration of D-center could not be reliably estimated due to the broadening and the strong overlap with surrounding defects.

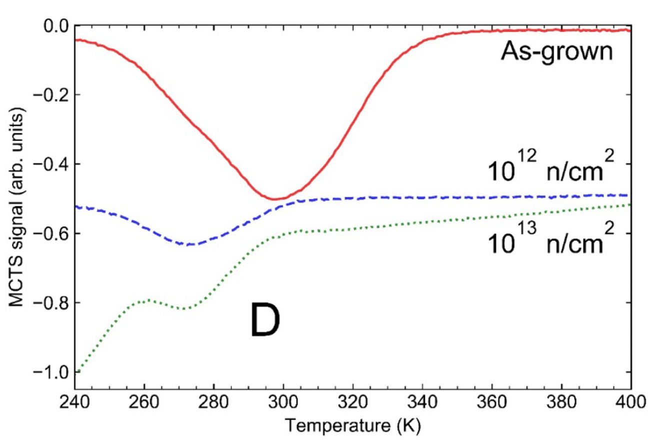

Fig. 7. D-center in the MCTS spectra of as-grown and neutron irradiated samples. Emission rate is 10 s^{-1}. The spectra are shifted vertically for the clarity.

Table 1
Activation energies for electron and hole emissions E_a and apparent capture cross-sections σ for all observed traps in as-grown and neutron irradiated semi-transparent n-type 4*H*-SiC SBD's.

Method	Trap label	E_a	σ (cm^2)	Attribution
DLTS	$Z_{1,2}$	$E_C - 0.67 \pm 0.01$ eV	7×10^{-15}	V_C (=/0)
	EH1	$E_C - 0.40 \pm 0.01$ eV	1×10^{-15}	V_{Si} (3-/=)
	EH3	$E_C - 0.70 \pm 0.04$ eV	1×10^{-15}	V_{Si} (=/-)
Laplace DLTS	$EH1_1$	$E_C - 0.41 \pm 0.03$ eV	5×10^{-16}	$V_{Si}(h)$ (3-/=)
	$EH1_2$	$E_C - 0.420 \pm 0.001$ eV	5×10^{-15}	$V_{Si}(k)$ (3-/=)
MCTS	D	$E_V + 0.60 \pm 0.02$ eV	5×10^{-16}	B_C (–/0)
	B	$E_V + 0.27 \pm 0.01$ eV	3×10^{-14}	B_{Si} (–/0)
Laplace MCTS	D1	$E_V + 0.491 \pm 0.001$ eV	5×10^{-18}	$B_C(h)$ (–/0)
	D2	$E_V + 0.567 \pm 0.005$ eV	3×10^{-16}	$B_C(k)$ (–/0)

The electronic properties and suggested attributions of the observed majority and minority carrier traps are summarized in Table 1.

4. Conclusions

We have investigated the influence of neutron radiation on majority and minority carrier traps in n-type 4*H*-SiC material. The neutron irradiation has introduced two deep traps, labeled as EH1 and EH3 with the activation energies for electron emission estimated as 0.4 and 0.7 eV bellow the conduction band, respectively. We have assigned EH1 trap to silicon vacancy (V_{Si}). Two minority carrier traps labelled as B and D-center, respectively, were detected by minority transient spectroscopy (MCTS) in as-grown material. Activation energies for hole emission for B and D-center assigned to substitutional boron B_{Si} and B_C are estimated as 0.27 and 0.60 eV above the valence band, respectively. We have identified two emission lines for D-center by Laplace-MCTS measurements and assigned them to B_C sitting at hexagonal (−*h*) and cubic (−*k*) lattice sites.

CRediT authorship contribution statement

Ivana Capan: Conceptualization, Writing - original draft, Writing - review & editing, Supervision. **Tomislav Brodar:** Investigation, Writing - review & editing, Visualization. **Yuichi Yamazaki:** Investigation, Resources. **Yuya Oki:** Investigation, Resources. **Takeshi Ohshima:** Investigation, Resources. **Yoji Chiba:** Investigation, Resources. **Yasuto Hijikata:** Investigation, Resources. **Luka Snoj:** Investigation, Resources. **Vladimir Radulović:** Investigation, Resources.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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