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# Atomic-scale characterization of structural damage and recovery in Sn ion-implanted $\beta$ -Ga<sub>2</sub>O<sub>3</sub>

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## ABSTRACT

 $\beta$ -Ga<sub>2</sub>O<sub>3</sub> is an emerging ultra-wide bandgap semiconductor, holding a tremendous potential for power-switching devices for next-generation high power electronics. The performance of such devices strongly relies on the precise control of electrical properties of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>, which can be achieved by implantation of dopant ions. However, a detailed understanding of the impact of ion implantation on the structure of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> remains elusive. Here, using aberration-corrected scanning transmission electron microscopy, we investigate the nature of structural damage in ion-implanted  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> and its recovery upon heat treatment with the atomic-scale spatial resolution. We reveal that upon Sn ion implantation, Ga<sub>2</sub>O<sub>3</sub> films undergo a phase transformation from the monoclinic  $\beta$ -phase to the defective cubic spinel  $\gamma$ -phase, which contains high-density antiphase boundaries. Using the planar defect models proposed for the  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, which has the same space group as  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>, and atomic-resolution microscopy images, we identify that the observed antiphase boundaries are the {100}1/4 (110) type in cubic structure. We show that post-implantation annealing at 1100 °C under the N<sub>2</sub> atmosphere effectively recovers the  $\beta$ -phase; however, nano-sized voids retained within the  $\beta$ -phase structure and a  $\gamma$ -phase surface layer are identified as remanent damage. Our results offer an atomic-scale insight into the structural evolution of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> under ion implantation and high-temperature annealing, which is key to the optimization of semiconductor processing conditions for relevant device design and the theoretical understanding of defect formation and phase stability.

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Power electronics represents a technology that capitalizes on the use of power-switching devices for energy-efficient control of electric power. A key requirement of power-switching devices is high break-down strength, which determines both the figure-of-merit and device performance limit.<sup>1</sup> As the breakdown voltage is scaled with the bandgap energy, semiconductors with a wide bandgap energy are needed to enable high voltage operation and reduce the power conversion loss for a given breakdown voltage.<sup>2</sup> The monoclinic, beta-phase (space group C2/m) Ga<sub>2</sub>O<sub>3</sub> ( $\beta$ -Ga<sub>2</sub>O<sub>3</sub>) has, therefore, garnered tremendous interest as a promising contender for supporting next-generation power electronics due to its ultrawide bandgap of ~4.8 eV and a theoretical breakdown electric field of ~8 MV cm<sup>-1,3-6</sup> In addition, recent advances in bulk synthesis and thin-film technologies along with the availability of high-quality, cost-effective substrates

have encouraged a surge in the development of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>-based power devices for high-capacity power supplies, mass power transmission, and electric transportation.  $^{7-11}$  These benefits have led to a demonstration of a range of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>-based power-switching devices, including Schottky diodes,  $^{12-14}$  metal–oxide–semiconductor field effect transistors (MOSFETs),  $^{6.15-17}_{0.15-17}$  and metal–semiconductor field effect transistors (MESFETs).  $^{18,19}$ 

Critical to successful implementation of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> in powerswitching devices with the targeted performance is the precise control over its electrical properties via doping. Incorporation of dopant elements in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> during film growth, often referred to as *in situ* doping,<sup>20</sup> has been demonstrated using a variety of growth techniques such as Si, Ge, and Sn doping with molecular beam epitaxy (MBE),<sup>21–23</sup> Si and Sn doping with metal-organic vapor phase epitaxy

(MOVPE),<sup>24</sup> and Sn doping with mist chemical vapor deposition (mist CVD), all of which result in n-type conductivity.<sup>25</sup> Delta doping of Si in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>, which leads to the formation of a two-dimensional electron gas, has also been attempted to fabricate MESFETs with promising device performance.<sup>18</sup> Ion implantation is another powerful, well-established method to dope semiconductors with potential benefits, such as low temperature processing conditions and excellent doping profile and dosage control, which have been proven beneficial for fabricating power electronics based on Si and conventional wide bandgap semiconductors (GaN and SiC) over the last few decades.<sup>26–28</sup> Si ion implantation in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> has been used to form lowresistance source/drain contacts.<sup>29,30</sup> In addition, deep acceptor levels can be created with Mg and N ion implantation in n-type  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> to define a high-resistance region for suppressing edge leakage current in vertical devices and achieving isolation of active device.<sup>31–33</sup> A major concern for the practicality of ion implantation is the structural damage induced by collisions between energetic dopant ions and host atoms as well as the effectiveness of post-implantation thermal annealing for damage recovery and dopant activation.<sup>34</sup> Prior work in Si and Sn ion implantation revealed that structural damage and remanent strain in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> by x-ray diffraction (XRD) and transmission electron microscopy (TEM) are not fully recovered with thermal annealing.<sup>35</sup> In addition, residual damage and the sign of stress in the irradiated layer depend on surface orientation of Si<sup>+</sup> implantation.<sup>36</sup> The accumulation of damage during ion irradiation also depends on the dose and ion flux density.<sup>37–40</sup> In the case of  $O_2$  annealing, there was extensive redistribution of the Si, Ge, and Sn implanted ions across the entire dose range, while in sharp contrast, the use of N<sub>2</sub> annealing suppressed the dopant diffusion,<sup>41,42</sup> which was ascribed to the influence of Ga vacancies.<sup>43</sup> Electron diffraction was used to show that Ge ion implantation induces a phase transformation of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> to the  $\kappa$ -phase, a lesser-known subset of the  $\varepsilon$ -phase.<sup>44,45</sup> Early studies of radiation effects studies conducted on  $\beta$  polymorph show that amorphization does not occur at room temperature with low energy ions (i.e., high percentage of displacement damage), even at high fluences.<sup>39</sup> Instead, the backscattered yield saturates at  $\sim$ 0.9, due to the formation of point defects and defect clusters as well as the recombination of these defects, which lead to the saturation in observed damage.<sup>39</sup> The material is expected to become amorphous at low temperature due to accumulation of damage without dynamic annealing that occurs at room temperature, but this has not yet been demonstrated.

The current literature still lacks an atomic-scale investigation of the nature of implantation-induced structural damage and remanent defects after the thermal annealing process. This knowledge is vital to produce a fundamental understanding of the impact of structural defects on dopant diffusion/activation and achieving process optimization of ion implantation for desired electrical properties.

Here, we investigate the structural damage and recovery of Sn ion-implanted  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> films using aberration-corrected scanning transmission electron microscopy (STEM), which can resolve individual atomic columns with sub-Å spatial resolution. Using this atomic-resolution imaging technique, we find that the structural damage induced by Sn ion implantation is the  $\gamma$ -phase Ga<sub>2</sub>O<sub>3</sub> (cubic, space group  $Fd\overline{3}m$ ) featuring antiphase boundaries (APBs). High-temperature thermal annealing results in the recovery of the  $\beta$ -phase; however, remanent structural defects at different locations within the film are identified.

The 500 nm thick  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> epitaxial film was grown on an edgedefined, film-fed grown Fe-doped (010)  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> using ozone-assisted MBE, obtained from Novel Crystal Technology. In order to achieve a near-uniform Sn profile with the targeted concentration  $\sim 10^{19} \, \mathrm{cm}^{-3}$ , the film was then implanted with Sn ions at  $2 \times 10^{13}$  cm<sup>-2</sup>/60 keV +  $3 \times 10^{13}$  cm<sup>-2</sup>/100 keV +  $4 \times 10^{13}$  cm<sup>-2</sup>/200 keV. To minimize channeling of the implanted ions, implantation was performed 7 degrees off axis. The as-implanted film was then subject to a 60-s rapid thermal annealing at 1100 °C in N2 ambient. Cross-sectional TEM specimens of as-grown, as-implanted, and post-annealed samples were prepared for the  $[001]_m$  projection of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>, where the subscript indicates the monoclinic structure, using an FEI Helios Nanolab 600 Dual Beam focused ion beam/scanning electron microscope (FIB/SEM). High-angle annular dark-field (HAADF) imaging in STEM was conducted on a Themis Z (Thermo Fisher Scientific) operated at 200 keV, equipped with a fifth order probe spherical aberration corrector. Energy dispersive x-ray spectroscopy (EDS) was performed using a Super-X detector system in a Themis Z. For HAADF-STEM imaging, an electron probe convergence semi-angle of 20 mrad and a HAADF detector range of 58-200 mrad were used. To minimize the image distortion arising from sample drift during imaging and enhance image contrast, HAADF-STEM images were obtained using the rigid registration method by which 20 fast-scan images were sequentially recorded, aligned using cross correlation, and averaged.

Figure 1(a) shows the representative HAADF-STEM image of an as-grown  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> film (unimplanted) imaged along the  $[001]_m$  zone axis. As shown in the corresponding schematic in Fig. 1(b), this zone axis allows for imaging crystallographically inequivalent Ga<sub>T</sub> and Ga<sub>O</sub> positions, which have tetrahedral and octahedral coordination, respectively. Since the image intensity in HAADF-STEM images is sensitive to atomic number (Z), high-intensity region in Fig. 1(a) corresponds to the atomic columns consisting of the heavy constituent element (i.e., Ga in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>), while the signal from oxygen atomic columns is barely detected.

Shown in Fig. 2(a) is the atomic-resolution HAADF-STEM image of a nominal  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> film after Sn ion implantation, i.e., as-implanted Ga<sub>2</sub>O<sub>3</sub>, revealing unique atomic arrangements that are distinct from that of an unimplanted, as-grown  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>. This implantation-induced damage region extends over  $\sim 120$  nm from the film surface, which matches with the extent over which an appreciable number of Sn dopants (1% of target concentration) are located in asimplanted  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> (Fig. S1). Two distinct structural features are determined in this image. First, Fig. 2(b) displays a hexagon-like feature, guided by the red hexagon, consisting of ten atomic columns with the high-intensity single atomic column in the center of each hexagon. This atomic arrangement corresponds to the  $\gamma$ -polymorph (cubic spinel) of  $Ga_2O_3$  viewed along the  $[110]_c$  projection, where the subscript refers to the cubic structure. The model structure used for constructing the  $\gamma$ -phase structure is based on magnetite, Fe<sub>3</sub>O<sub>4</sub>,<sup>46</sup> containing cation vacancies, which adopts a defective spinel structure with the same space group  $(Fd\overline{3}m)$  as  $\gamma$ -Ga<sub>2</sub>O<sub>3</sub>, as suggested by Chang et al.<sup>47</sup> In this structure, the anion (oxygen) sublattice is fully occupied, while the cation (Ga) sublattice contains vacancies that balance the [Ga]/[O] ratio to be 2/3 and lead to a small displacement of constituent atoms (thus termed "defective"). The structure of the  $\gamma$ -phase has not been extensively studied with regard to the bulk or film geometry as the  $\beta$ -phase is the most thermodynamically stable among reported





polymorphs of Ga<sub>2</sub>O<sub>3</sub>; however, there have been reports on this cubic spinel structure in nanoparticles/nanocrystalline structures.<sup>48,49</sup> In addition, the presence of  $\gamma$ -phase inclusions was recently reported as a common structural defect in as-grown, doped  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> and alloyed  $\beta$ -(Al<sub>x</sub>Ga<sub>1-x</sub>)<sub>2</sub>O<sub>3</sub> during film growth, especially at low temperature growth conditions.<sup>47</sup>

The second notable feature in as-implanted Ga<sub>2</sub>O<sub>3</sub> is the defect phase, which is characterized by the diagonally streaking, high-intensity atomic columns and a succession of three atomic columns (triplets) in between those streaking atomic columns, marked by a red rectangle and a yellow arrow in Fig. 3(a), respectively. The atomic structure of this new defect phase does not match any projections from known polymorphs of Ga<sub>2</sub>O<sub>3</sub>, but rather resembles overlapped sheets of the  $\gamma$ -phase, which was previously observed in as-grown, doped  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>.<sup>47</sup> Antiphase boundaries (APBs) were reported as a common planar defect in the defective spinel structure (e.g.,  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> that has the same space group as  $\gamma$ -Ga<sub>2</sub>O<sub>3</sub>) that can produce lattice shifts.<sup>50</sup> The APBs for  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> that were experimentally determined using x-ray diffraction and conventional transmission electron microscopy analysis are characterized by the displacement vector  $1/4\langle 110 \rangle_c$  in the  $\{100\}_c$  planes of the



**FIG. 2.** (a) HAADF-STEM image of a nominal  $Ga_2O_3$  film implanted with Sn ions. (b) Schematic (top) and high-magnification HAADF-STEM image (bottom) of the  $\gamma$ -Ga<sub>2</sub>O<sub>3</sub> structure projected along [110]<sub>c</sub>, where the subscript indicates the cubic structure. Locations of the pure  $\gamma$ -Ga<sub>2</sub>O<sub>3</sub> are indicated by yellow arrows in (a).

cubic spinel structure.<sup>50,51</sup> Using this information, we constructed two model structures of the defect phase with the APBs of the type  $(001)_c 1/4[10\overline{1}]_c$  [Figs. 3(c) and 3(e)] and the type  $(001)_c 1/4[\overline{1}0\overline{1}]_c$  [Figs. 3(d) and 3(f)]. These types of APBs disrupt the coherence of the Ga sublattice, while the oxygen sublattice remains unchanged.<sup>50</sup> We note that the high intensity atomic columns, as in the red box in Fig. 3(a), are due to Ga<sub>O</sub> atoms stacked on top of each other from the overlapped sheets, as seen in Figs. 3(e) and 3(f), whereas the lower intensity triplets do not involve stacking by atoms from separate sheets. The model structures in Figs. 3(e) and 3(f) are in good agreement with the recorded HAADF-STEM images of an as-implanted Ga<sub>2</sub>O<sub>3</sub> film, strongly suggesting that the structural origins for the dominant defect phase in as-implanted Ga<sub>2</sub>O<sub>3</sub> are APBs.

Next, we examined the structural damage recovery of the implanted Ga2O3 film upon rapid thermal annealing. Figure 4(a) shows the low-magnification HAADF-STEM image of post-annealed Ga<sub>2</sub>O<sub>3</sub>. A large volume of damaged structure was recovered back to the  $\beta$ -phase. However, remanent structural defects persist. First, we observed low-intensity polygonal patches, marked by yellow arrows in Fig. 4(a), throughout the post-annealed film, mostly confined at 40-100 nm away from the surface. The high-magnification HAADF-STEM images in Fig. 4(b) revealed that the atomic structure within this low-intensity region mostly retains the  $\beta$ -phase structure. Thus, the relatively low intensities in the nominal Ga<sub>T</sub> and Ga<sub>O</sub> atomic columns in those polygonal regions arise from Ga vacancies, which is also supported by our EDS result (supplementary material Fig. S2). While similar structural features have been reported in prior literature as both vacancy clusters and voids,<sup>52-54</sup> based on the size and low atomic density of polygonal patches observed in our work, we hereafter refer to those features as nano-sized voids. Furthermore, Sn dopants were identified in the vicinity of these nano-sized voids. Since the Sn dopants have a higher Z than the host (Ga), the Sn-containing Ga atomic columns show higher intensities in HAADF-STEM images. Figures 4(c) and 4(d) show a high-magnification HAADF-STEM image near a nano-sized void and a corresponding atomic-column intensity map. Using the statistical criterion used in previous studies,<sup>55,56</sup> atomic columns having distinctly higher intensities than those of neighboring atomic columns are marked by white arrows. Given that these atomic columns are atomically isolated, it is highly likely that high image intensities arise from heavy Sn dopants rather than a gradual change in number of Ga atoms across the interface between



FIG. 3. (a) and (b) High-magnification HAADF-STEM images of a nominal Ga<sub>2</sub>O<sub>3</sub> film implanted with Sn ions. (c) and (d) Projected unit cells of a cubic spinel structure along [110]<sub>c</sub> that represent the lattice shifts,  $1/4[101]_c$  and  $1/4[101]_c$ , respectively. (e) and (f) Projected model structures along [110]<sub>c</sub> constructed based on the lattice shifts in (c) and (d), which match the recorded HAADF-STEM images in (a) and (b).

nano-sized voids and  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>. As shown in Figs. 4(c) and 4(d), Sn dopants, indicated by white arrows, are predominantly located at the octahedral coordinated Ga atomic columns (Ga<sub>0</sub>), which is consistent with previous theoretical prediction<sup>57,58</sup> and experimental results.<sup>47,59</sup>

Another major feature in post-annealed Ga<sub>2</sub>O<sub>3</sub> is the incomplete recovery of the  $\beta$ -phase near the surface of the Ga<sub>2</sub>O<sub>3</sub> film. Figure 4(e) shows the combination of the pure  $\gamma$ -phase structure and the defect phase similar to the as-implanted Ga<sub>2</sub>O<sub>3</sub>, shown in Figs. 2 and 3. This





remanent  $\gamma$ -phase extends about 5–10 nm from the film surface, which is consistent with the thickness of the remaining defect layer observed in post-annealed Ge, Sn, and Si implanted  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> films,<sup>35,41</sup> although these early studies did not identify the nature of this surface layer. Note that, in prior studies, the formation of the  $\gamma$ -phase has been observed at the surface of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> during film growth.<sup>47,60</sup> Moreover, as mentioned earlier, the  $\gamma$ -phase has been identified in nanostructures,<sup>48,49</sup> where the surface-to-volume ratio is high. These experimental results along with our observation of the  $\gamma$ -phase on the surface suggest that the surface energy plays an important role in the stability of the  $\gamma$ -phase over the  $\beta$ -phase. Current theoretical studies on the surface energies of Ga<sub>2</sub>O<sub>3</sub> polymorphs are limited to the  $\beta$ -phase,<sup>61,62</sup> and, thus, it would be interesting to computationally investigate the landscape of surface energies of different polymorphs of Ga<sub>2</sub>O<sub>3</sub> with respect to crystallographic planes and annealing conditions.

It is worth noting that HAADF-STEM imaging is less sensitive to light elements than relatively heavy elements (e.g., oxygen in Ga<sub>2</sub>O<sub>3</sub>). However, given the extent (4-20 nm) and atomic structure of the detected nano-sized voids observed in Fig. 4(a), it is believed that this region may contain oxygen vacancies as well to maintain reasonable stoichiometry with local charge neutrality. Clustering of cation and anion vacancies was previously reported for implanted and annealed wide bandgap semiconductors, such as GaN.<sup>63</sup> These nano-sized voids are typically formed when vacancies are mobile, and there are no sinks such as dislocations and free surface. Based on our result, showing that no clusters were observed near the top 40 nm of the thin film, we speculate that the vacancies present in the  $\gamma$ -phase (as-implanted structure) are annealed to the surface, but those present at deeper region within the film agglomerate into a stable nano-sized void. Further systematic computational investigations are needed to produce an improved understanding of the mechanisms of vacancy clustering (and Sn ion segregation near nano-sized voids) and its impact on the electronic properties of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>. Additionally, another imaging technique available in STEM, namely, annular bright-field (ABF) imaging, which has been used to detect light elements (i.e., oxygen) in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>,<sup>64</sup> may prove useful in future studies for locating regions of dopant segregation. As demonstrated in Fig. S3, ABF-STEM imaging is sensitive to relatively light concentrations of dopants, especially heavy dopants like Sn in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>. Quantitative analysis of both ABF and HAADF images in combination with image simulation is needed to further elucidate the evolution of point defects in Ga2O3 after implantation and postthermal annealing.

To summarize, we have performed HAADF imaging in an aberration-corrected STEM to investigate the impact of high-energy ion implantation and rapid thermal annealing on the structure of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>. We identified that Sn ion implantation induces the phase transformation from the  $\beta$ -phase to the  $\gamma$ -phase with high density of the defect phase. This result is in stark contrast to the reported phase transformation of Ge-implanted  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>, where the  $\beta$ -phase is transformed into the  $\kappa$ -phase.<sup>45</sup> The origin of the defect phase in the  $\gamma$ -phase was determined to be APBs on {100}<sub>c</sub> planes with the lattice displacement of 1/4 (110)<sub>c</sub>. High-temperature rapid thermal annealing was proven effective to recover the  $\beta$ -phase from the defective  $\gamma$ -phase; however, nano-sized voids embedded in  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> and the  $\gamma$ -phase on the film surface were identified as remanent structural defects. These are likely the structural origins for incomplete recovery of the lattice parameters and increased XRD rocking curve full width at half

maximum that is observed in Sn ion implanted  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> after annealing.<sup>35</sup> The atomic-scale insights into the structure/defect evolution presented in this work provide invaluable constraints for future theoretical studies on the formation and diffusion of point defects (vacancies and dopants) and the phase stability of competing polymorphs ( $\beta$ - and  $\gamma$ -phases). In addition, our results emphasize the need to optimize the conditions for implantation and annealing. In particular, approaches to avoid the formation of the surface  $\gamma$ -phase are urgently needed as the properties of this region, which interfaces source/drain contacts, diode junctions, and channels, have profound influence on the performance of power-switching devices.

See the supplementary material for the secondary ion mass spectroscopy (SIMS) of the Sn profile, the EDS line profile across the nano-sized void, and ABF-STEM results.

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# AUTHOR DECLARATIONS

#### **Conflict of Interest**

The authors have no conflicts to disclose.

#### **Author Contributions**

Timothy Yoo: Data curation (lead); Formal analysis (lead); Investigation (lead); Visualization (equal); Writing – original draft (lead). Xinyi Xia: Investigation (supporting); Writing – original draft (supporting). Fan Ren: Supervision (supporting); Writing – original draft (supporting). Alan Jacobs: Investigation (supporting); Writing – original draft (supporting). Marko J. Tadjer: Investigation (supporting); Writing – original draft (supporting). Stephen J. Pearton: Conceptualization (equal); Funding acquisition (equal); Investigation (supporting); Supervision (supporting); Writing – review and editing (supporting). Honggyu Kim: Conceptualization (equal); Supervision (lead); Visualization (equal); Writing – original draft (supporting); Writing – review and editing (lead).

#### DATA AVAILABILITY

The data that support the findings of this study are available within the article and its supplementary material.

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