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## SPECIAL ISSUE ARTICLE

# Effect of light irradiation on indentation-induced dislocation behavior in GaN oriented for basal slip

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## Editor's Choice

The Editor-in-Chief recommends this outstanding article.

## Abstract

The impact of external light on dislocation-based plasticity in inorganic compound semiconductors has been increasingly recognized. Here, we investigated the effect of light on the dislocation behavior in wurtzite GaN oriented for basal slip using photoindentation. Two types of nanoindentation tests were performed in darkness and in 380 nm light on GaN single-crystal substrates oriented to maximize the Schmid factor for the basal slip. Distinct pop-in events were observed in the loading segment at loads of 150–340  $\mu\text{N}$ . Analysis of the first pop-in events revealed that they correspond to homogeneous dislocation nucleation and are largely unaffected by light irradiation. Indentation creep tests at 2 mN showed that both creep depth and creep strain rate decreased in 380 nm light. Cross-sectional ultra-high voltage electron microscopy images taken beneath the indentation imprints displayed an asymmetric dislocation distribution with the majority aligned along the  $[11\bar{2}0]$  direction. A significant reduction in the density of indentation-induced dislocations in 380 nm light was observed, indicating that light irradiation effectively suppresses dislocation glide motion and multiplication in GaN for basal slip.

## KEYWORDS

dislocation, GaN, nanoindentation, photoplastic effect, plasticity, semiconductors

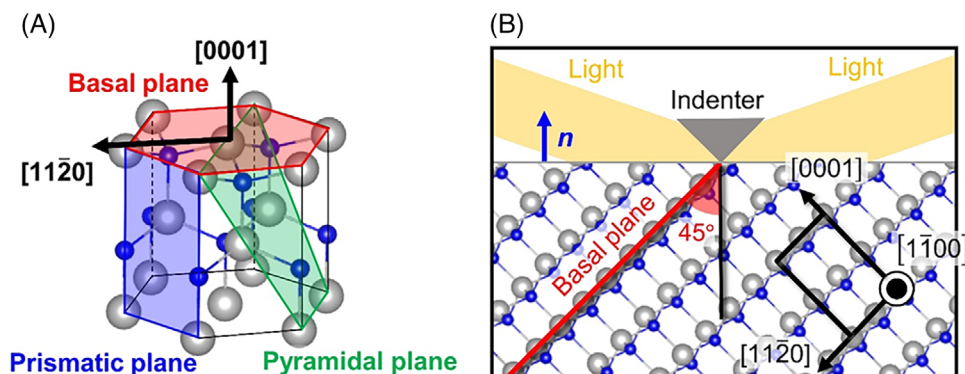
## 1 | INTRODUCTION

Inorganic compound semiconductors are regarded as brittle at room temperature due to the directional nature of their covalent and/or ionic bonds.<sup>1</sup> The inherent lack of plasticity in these materials poses substantial challenges to their fabrication and application. Therefore, various studies have been conducted over the past few decades to improve their processability and deformability.<sup>2–5</sup> In 2018, Oshima et al. reported that ZnS crystals, which were

typically brittle under regular light, exhibited extraordinary plasticity of 45% compression strain in darkness even at room temperature.<sup>6</sup> This light-induced alteration in the plasticity of ZnS is believed to result from the interaction between dislocations and photoexcited carriers,<sup>7</sup> which leads to reconstruction at the dislocation cores and increases the potential barrier for dislocation motion. Nowadays, interest in numerous historical studies on the photoplastic effect has been rekindled<sup>6–12</sup> and there is a growing expectation that controlling the external light

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**FIGURE 1** Schematics illustrating (A) wurtzite crystal structure of GaN and typical slip planes, and (B) photoindentation test on the (0001) 45° off sample. Silver-colored atoms represent Ga atoms, blue-colored atoms represent N atoms, and a red-colored plane indicates the basal plane. In (0001) 45° off samples, the basal plane (0001) and the  $[11\bar{2}0]$  direction form a 45° angle with the indentation direction (surface normal), respectively.

environment could tune the mechanical properties of semiconductors. Moreover, if it becomes possible to introduce a high density of dislocations into brittle materials without causing cracks by regulating the light condition, it paves the way for developing new materials with localized unique functionalities originating from the introduced dislocations.<sup>13–16</sup>

Gallium nitride (GaN) is a prominent III–V compound semiconductor with a wide bandgap of 3.39 eV<sup>17</sup> at room temperature. Due to its exceptional electrical and thermal properties,<sup>18,19</sup> GaN holds great potential in light-emitting and power electronics applications. GaN-based high-power and high-frequency devices are anticipated to outperform traditional Si-based power devices in efficiency and contribute to reducing global energy demand.<sup>20</sup> Yet, challenges persist in the production of high-quality bulk GaN single crystals,<sup>21</sup> leading to limited studies on the macroscopic mechanical properties of GaN. The anisotropy and complexity of the wurtzite structure further complicate the understanding of mechanical properties. The crystal structure and typical slip planes in GaN are illustrated in Figure 1a. To date, most experimental insights into the mechanical response and dislocation behavior of GaN have been gained through nanoindentation experiments performed on small and thin samples.

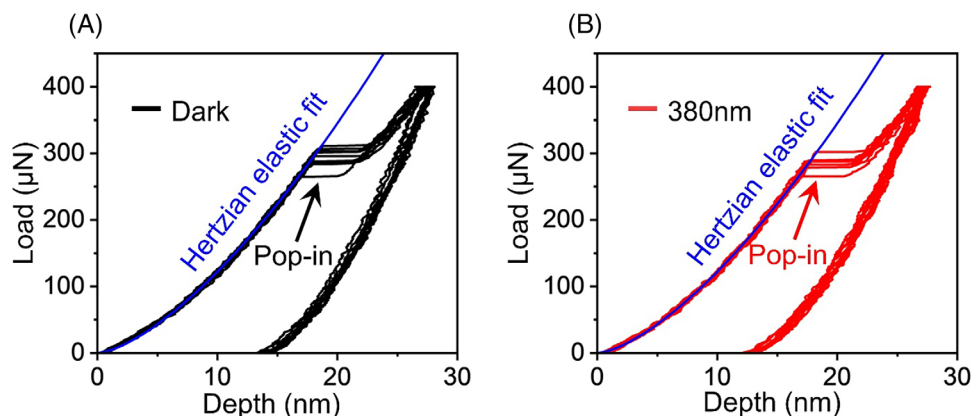
Indentation tests on GaN have mainly been conducted in the millinewton (mN) range on representative low-index surfaces such as *c*-(0001) plane<sup>22–25</sup> and *m*-( $1\bar{1}00$ ) plane.<sup>26</sup> Post-indentation transmission electron microscopy (TEM) observations reported the activation of basal and prismatic/pyramidal dislocations.<sup>23–26</sup> Recently, Cao et al. observed that the indentation hardness of the (0001) plane in GaN increases under 10 mN indentation tests with light irradiation,<sup>25</sup> providing preliminary evidence for the “photo-hardening” effect in GaN, although the specific dislocation mechanism remains unclear. In

this study, the effects of light irradiation on GaN oriented for basal slip were investigated at dislocation level through nanoindentation tests under a controlled light environment and ultra-high voltage electron microscopy observation.

## 2 | MATERIALS AND METHODS

The GaN sample used in this study was a specially designed single crystal with an unconventional orientation. As illustrated in Figure 1b, the GaN substrate surface is inclined by 45° from the (0001) basal plane; hence, referred to as the (0001) 45° off orientation. The (0001)( $11\bar{2}0$ ) basal slip system, characterized by the densely packed atoms and most widely spaced plane, represents the energetically favorable slip system in wurtzite crystals. For the designed (0001) 45° off samples, the angle between the  $[11\bar{2}0]$  axis and the indentation direction is also 45°. Consequently, the Schmid factor for the basal slip system reaches 0.5, facilitating the activation of basal slip under loading. The GaN substrates (provided by SixPoint Materials, Inc.) were grown via the ammonothermal method,<sup>27</sup> with a thickness of 1 mm. To minimize the effect of initial free electrons and holes, Mn-doped samples with a concentration of approximately  $1.0 \times 10^{19} \text{ cm}^{-3}$  were used. The sample surface was chemically mechanically polished and achieved an arithmetic average roughness of approximately 0.3 nm.

Nanoindentation testing in a controlled light environment, termed the photoindentation technique,<sup>8</sup> was adopted to evaluate the mechanical response of GaN in darkness and in 380 nm light. Two types of nanoindentation experiments were conducted: (i) Low-load quasi-static nanoindentation tests, and (ii) indentation creep tests. In the (i) tests, a load up to 400  $\mu\text{N}$  was applied with a loading rate of 40  $\mu\text{N/s}$ , followed by unloading at the same



**FIGURE 2** Representative 10 load–displacement ( $P$ – $h$ ) curves obtained from the (i) low-load quasi-static nanoindentation tests: (A) in darkness and (B) in 380 nm light. The blue solid lines represent the same Hertzian elastic fit. Out of 100 curves obtained under each light condition, more than 85 exhibited a clear pop-in event.

rate after a 0.5 s hold, and 100 tests were performed under each light condition. In the (ii) tests, a peak load of 2 mN was maintained for 100 s, and 40 tests were conducted under each light condition. During testing, the nanoindentation apparatus (Elionix Inc., ENT-NEXUS) was entirely covered by dark curtains to block light from outside the apparatus. Light from a xenon light source (Asahi Spectra Inc., MAX-350) was filtered to the wavelength of 380 nm via a bandpass filter and directed onto the sample surface from two directions. The wavelength of 380 nm was chosen because its energy of 3.26 eV is just below the bandgap energy of GaN (3.39 eV<sup>17</sup>). Previous research has indicated that light with wavelengths corresponding to energies slightly below the bandgap has the most significant impact on the mechanical properties of compound semiconductors.<sup>8,9</sup> The light intensity near the indenter tip was set to 100  $\mu\text{W}/\text{cm}^2$ . All nanoindentation experiments were performed in load-controlled mode using a Berkovich indenter with a tip radius of 120 nm. The temperature inside the apparatus was kept constant at 30°C. More details on the photoindentation techniques can be found elsewhere.<sup>8</sup> After indentation creep tests, cross-sections near the indentation imprints were extracted using a focused ion beam (Thermo Fisher Scientific, Helios G4 UX), and then examined using the bright-field scanning transmission electron microscopy (BF-STEM) mode of an ultra-high voltage electron microscope (UHVEM; JEOL Co Ltd., JEM-1000K RS) operated at 1000 kV.

### 3 | RESULTS AND DISCUSSION

Representative 10 load–displacement ( $P$ – $h$ ) curves from the (i) low-load quasi-static nanoindentation tests in darkness and 380 nm light are shown in Figure 2a,b, respectively. As indicated by arrows in Figure 2, distinct pop-in

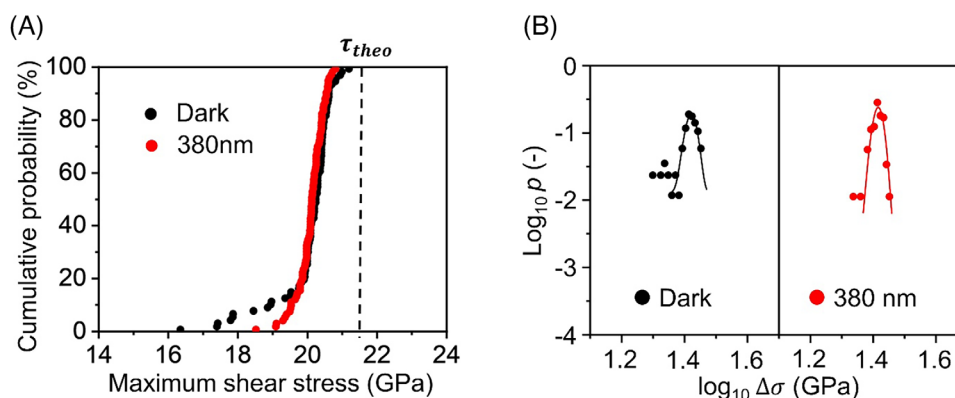
events, characterized by a sudden displacement burst at a constant load, were observed under both light conditions. Prior to the first pop-in, the GaN crystals deformed elastically, and the load–displacement curves can be described by Hertzian contact theory.<sup>28</sup> Regardless of the light conditions, almost all curves overlapped with the same Hertzian elastic fit before the first pop-in, demonstrating high reproducibility of our experiments and indicating little effect of light on elastic properties of GaN.

The occurrence of pop-in may be related to phase transition,<sup>29</sup> crack formation,<sup>30</sup> or dislocation activation.<sup>31</sup> Our subsequent UHVEM observations ruled out the first two possibilities, confirming that the first pop-in events in this study correlate with dislocation activation. Out of the 100 load–displacement curves obtained for each light condition, more than 85 curves exhibited a clear pop-in event at loads between 150 and 340  $\mu\text{N}$ . This high reproducibility of the pop-in events should be attributed to the well-polished surface of the sample, the use of an indenter with a small tip radius, and the adoption of an appropriate loading protocol. The first pop-in load  $P^{\text{pop-in}}$  and pop-in width  $\Delta h^{\text{pop-in}}$  were picked up, and the average values are presented in Table 1. The definitions of  $P^{\text{pop-in}}$  and  $\Delta h^{\text{pop-in}}$  are provided in Supporting Information S1. The average  $P^{\text{pop-in}}$  values are 285.9  $\mu\text{N}$  in darkness and 285.5  $\mu\text{N}$  in 380 nm light, with corresponding  $\Delta h^{\text{pop-in}}$  values of 3.81 and 3.82 nm, respectively. These minimal differences suggest that the first pop-in is largely unaffected by light irradiation.

The maximum shear stress  $\tau_{\text{max}}$  beneath the indenter at the first pop-in can be calculated based on Hertzian contact theory, as detailed in Supporting Information S2. Figure 3a shows the cumulative probabilities of  $\tau_{\text{max}}$  under two light conditions. Since  $\tau_{\text{max}}$  acts in the direction inclined at 45° relative to the indentation direction,  $\tau_{\text{max}}$  in this study can be considered as the maximum resolved shear stress

**TABLE 1** Measured and calculated values at the first pop-in.

	Pop-in load, $P^{\text{pop-in}}$ ( $\mu\text{N}$ )	Pop-in width, $\Delta h^{\text{pop-in}}$ (nm)	Maximum shear stress, $\tau_{\text{max}}$ (GPa)
In darkness	$285.9 \pm 32.8$	$3.81 \pm 0.47$	$20.24 \pm 0.41$
In 380 nm light	$285.5 \pm 16.6$	$3.82 \pm 0.31$	$20.12 \pm 0.36$

**FIGURE 3** (A) Cumulative probability of maximum shear stress at the first pop-in. At least 85 data points are shown under each light condition. The dashed line represents the theoretical shear strength for GaN basal slip. (B) The distribution of logarithmic probability  $p$  and the logarithmic stress drop  $\Delta\sigma$  at the first pop-in obtained by equal-width binning (0.6 GPa). The solid lines are Gaussian fitting curves.

on the basal plane. The average  $\tau_{\text{max}}$  values are 20.24 GPa in darkness and 20.12 GPa in 380 nm light. Both values are comparable to the theoretical shear strength for GaN basal slip, which is calculated to be 21.5 GPa.<sup>32,33</sup> This agreement implies that pop-in events occurring at such a high-stress level correspond to homogeneous dislocation nucleation.<sup>34</sup> Additionally, the  $\tau_{\text{max}}$  distributions under both light conditions range from 18.5 to 21.5 GPa and are closely aligned in Figure 3a, demonstrating the minor effect of light on homogeneous dislocation nucleation.

To gain a deeper insight into the mechanism underlying pop-ins, a statistical analysis of the stress drop  $\Delta\sigma$  caused by the first pop-in was conducted. The stress drop  $\Delta\sigma$  was calculated by  $P^{\text{pop-in}}$  and  $\Delta h^{\text{pop-in}}$ . Detailed calculations are provided in Supporting Information S3 and ref.<sup>35</sup> We then grouped the over 85  $\Delta\sigma$  values for each light condition into bins of equal width (0.6 GPa) and computed the frequency within each bin. Figure 3b shows the logarithmic probability distributions of  $\Delta\sigma$  at the first pop-ins, revealing a Gaussian-like distribution under both light conditions. According to Sato et al.,<sup>35</sup> a Gaussian-like distribution of  $\Delta\sigma$  typically indicates dislocation nucleation driven by thermal activation. In contrast, a power-law distribution of  $\Delta\sigma$  corresponds to dislocation network evolution, usually observed in the second or subsequent pop-in events. The pronounced Gaussian-like distributions illustrated in Figure 3b suggest that the pop-in events are linked to dislocation nucleation rather than dislocation interactions. The distributions of  $\Delta\sigma$  in darkness and in 380 nm light show some differences; the latter exhibits a

narrower and taller curve, indicating a more concentrated distribution with a smaller variance. The difference may be due to changes in thermal activation energy caused by light irradiation.

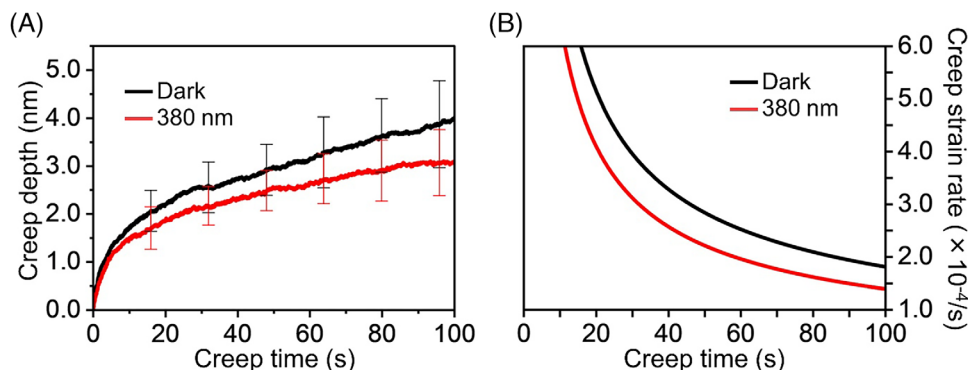
Nanoindentation creep depth  $\Delta h^{\text{creep}}$  and creep strain rate  $\dot{\epsilon}$  in the (ii) indentation creep tests are depicted as functions of creep time in Figure 4a,b, respectively. The creep depth  $\Delta h^{\text{creep}}$  refers to displacements during the load-hold period, and each curve in Figure 4a represents an average of at least 28 curves under the same light conditions. The creep strain rate  $\dot{\epsilon}$  is derived from the time derivative of the fitted creep strain  $\epsilon$  using the following equation<sup>34</sup>:

$$\epsilon = \frac{\Delta h^{\text{creep}}}{h} \approx at^b + ct, \quad (1)$$

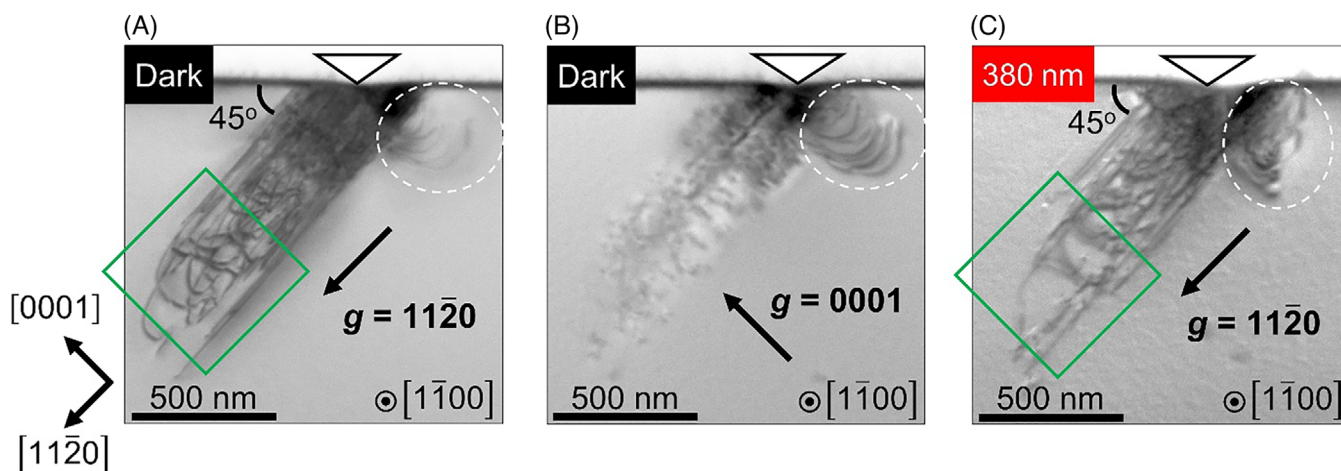
where  $h$  is indentation depth,  $a$ ,  $b$ , and  $c$  are fitting parameters, and  $t$  denotes time. As shown in Figure 4a,b, the creep depth and creep strain rate in 380 nm light are consistently lower than those in darkness. At  $t = 100$  s, differences of over 24% in both parameters are observed. Therefore, in contrast to the minor effect of light on dislocation nucleation detected in the (i) tests, the (ii) tests revealed a significant suppression of dislocation motion under light irradiation.

To further the understanding of dislocation behavior under different light conditions, the cross-sections beneath the indentation imprint after the (ii) indentation creep test were observed, using the BF-STEM mode of UHVEM. Figure 5a,b corresponds to the sample tested in darkness, while Figure 5c corresponds to the sample tested in 380





**FIGURE 4** (A) Creep depth and (B) creep strain rate as a function of creep time under each light condition. In (A), each solid curve represents an average of at least 28 curves, and error bars at several specific time points are shown.



**FIGURE 5** Cross-sectional images beneath the indentation imprints after 2 mN indentation creep tests using bright-field scanning transmission electron microscopy (BF-STEM) mode of ultra-high voltage electron microscope (UHVEM): (A and B) in darkness and (C) in 380 nm light. The triangles represent indenters during nanoindentation tests.

nm light. From Figure 5a–c, dislocations were presented within the stressed volume induced by indentation, with no dislocations observed in regions away from the imprint. Additionally, even under a load of 2 mN, no cracks or phase transitions were detected. Therefore, as mentioned above, the first pop-in events in this study are attributed to dislocation nucleation.

In order to investigate the characteristics of the indentation-induced dislocations, we analyzed the Burgers vector  $\mathbf{b}$  of these dislocations using different diffraction vectors. In this study, the Schmid factor for the basal slip system reaches the maximum of 0.5, making basal dislocations favorable to activate. In Figure 5a, when the diffraction vector  $\mathbf{g} = 11\bar{2}0$ , introduced dislocations are visible with clear contrast. Most dislocations were located in the lower-left region of the indenter and grew cylindrically to the  $[11\bar{2}0]$  direction along the (0001) basal plane. Here, the development of dislocations along the basal

plane suggests that most of the introduced dislocations are basal dislocations. In Figure 5b, these contrasts disappear under the diffraction vector  $\mathbf{g} = 0001$  due to  $\mathbf{g} \cdot \mathbf{b} = 0$ . This suggests that the Burgers vector of these dislocations lacks a [0001] component. In wurtzite crystals, previous studies have indicated that basal dislocations do not possess a [0001] component, while pyramidal dislocations do.<sup>10,23,24</sup> Therefore, it is believed that most of the dislocations in the lower-left region of the indenter are basal dislocations with  $\mathbf{b} = 1/3[11\bar{2}0]$ , belonging to the energetically favorable slip system in the present sample. Among these dislocations, straight dislocations parallel to the  $[11\bar{2}0]$  direction should be basal screw dislocations because the Burgers vector is parallel to the dislocation line. Meanwhile, other dislocations that connect and intersect the straight ones are presumed to be dislocations that have moved to other slip planes due to cross-slip, since basal edge dislocations must produce dot-like contrasts in

this observation direction. In addition, a small number of loop-shaped dislocations were induced near the surface on the opposite side beneath the indenter as enclosed by the white dashed circle. The dislocations maintained their contrast under both diffraction vectors of  $g = 0001$  and  $11\bar{2}0$ , indicating that the Burgers vector of these dislocations has both  $[0001]$  and  $[11\bar{2}0]$  components. Thus, it can be inferred that they are pyramidal dislocations.

For nanoindentation tests in 380 nm light, the induced dislocations also consist of basal dislocations cylindrically to the  $[11\bar{2}0]$  direction and pyramidal dislocations on the opposite side, as shown in Figure 5c. In other words, the dislocation substructure developed in light is similar to that formed in darkness. It should be noted that the dislocation distribution in the  $(0001)$   $45^\circ$  off GaN differs from previous nanoindentation studies conducted on low-index planes of GaN,<sup>23–26</sup> which exhibited a symmetric distribution due to the activation of several equivalent slip systems. The dislocations formed in the present  $(0001)$   $45^\circ$  off sample reflected the anisotropy of the wurtzite structure.

Figure 5a,c are compared in detail with a focus on the effects of light irradiation. Regardless of the light conditions, the deepest dislocations appeared at a depth of approximately 1000 nm from the surface. However, in the deeper region, spanning 500–1000 nm, the dislocation density in darkness was clearly higher than that in 380 nm light. When examining the region enclosed by the green square using Ham's method,<sup>36</sup> the dislocation density in darkness is more than 60% higher than that in light, as detailed in Supporting Information S4. This difference suggests that light irradiation suppresses the glide motion and multiplication of dislocations for basal slip, leading to significant differences in the results of indentation creep tests under different light conditions. Here, we use the term “dislocation multiplication” to explain the significant differences in dislocation density observed under the two light conditions. This is because the difference in dislocation density is difficult to explain solely by the difference in dislocation glide motion. However, the entire process of dislocation multiplication has not yet been identified and remains a future challenge. The occurrence of cross-slip in the basal screw dislocations may be related to the multiplication process.

Previous studies have shown that the III–V compound semiconductors such as GaP and GaAs exhibit a negative photoplastic effect (i.e., photo-softening).<sup>37,38</sup> In contrast, our study reveals that basal slip in GaN displays a positive photoplastic effect (i.e., photo-hardening). The positive photoplastic effect is similar to that observed in several II–VI compound semiconductors, such as ZnS<sup>6–8</sup> and ZnO.<sup>9–12</sup> Photoindentation studies on ZnS, ZnO, and GaN have revealed that light irradiation has only a minor effect on homogeneous dislocation nucleation, while it shows a pronounced suppressive effect on dislocation motion. This

is believed to be partly because the stress required for dislocation motion associated with the indentation creep is significantly lower than the theoretical shear strength. The interaction between dislocations and photoexcited carriers causes the stress required for dislocation motion to increase during light irradiation, which can have a significant effect on creep behavior. On the other hand, the maximum shear stress at first pop-in is mainly related to the atomic bonding of the perfect crystal, and normal light with a wavelength longer than the wavelength corresponding to the band gap should have little effect on this bonding.

It is also interesting that the effect of light irradiation on dislocation behavior differs depending on the material through differences in dislocation structure. For instance, when photoindentation tests were performed on ZnO oriented for basal slip, it was reported that the depth of indentation-induced dislocations in darkness was greater than that in light.<sup>9</sup> Intriguingly, in this study, there was almost no difference in the depth of the formed dislocations under the two light conditions, and instead, there was a pronounced difference in the density of the dislocations. This suggests that, in terms of the photoplastic effect on the introduced dislocation structure, even materials with the same crystal structure and orientation can respond differently.<sup>12</sup> Despite sharing the same crystal structure, the leading dislocations in ZnO tend to move forward, whereas the leading dislocations in GaN tend to interact with the trailing dislocations, due to the different Peierls barriers for dislocation motion.<sup>39</sup> Meanwhile, the total number of indentation-induced dislocations increases in darkness for both ZnO and GaN, indicating a similarity between them in this respect.

## 4 | CONCLUSION

In conclusion, the effect of light on dislocation behavior in wurtzite GaN was studied using two types of photoindentation tests on specially designed GaN single crystal substrates oriented for basal slip. For quasi-static nanoindentation tests, pop-in events occurred in over 85% of the acquired load–displacement curves. The statistical analysis of pop-ins indicated that the first pop-in events correspond to homogeneous dislocation nucleation. The effect of light on the incipient plasticity associated with dislocation nucleation was found to be minor. Indentation creep tests demonstrated a significant suppression of dislocation motion by 380 nm light, as evidenced by the reduction in creep depth and creep strain rate. Subsequent UHVEM observations revealed that most indentation-induced dislocations were present in a cylindrical region along the basal plane in the  $[11\bar{2}0]$  direction, which are believed to be the basal dislocations with  $b = 1/3[11\bar{2}0]$ . While the


maximum depth of introduced dislocations was largely unaffected by light irradiation, the density of indentation-induced dislocations was markedly reduced in 380 nm light. This indicates a suppressive effect of light irradiation on dislocation glide motion and multiplication in GaN for basal slip. The deeper understanding of the photoplastic effect of GaN obtained from this study contributes to overcoming the brittleness in GaN and optimizing GaN-based device fabrication, paving the way for more reliable and efficient applications of GaN in future technologies.

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## SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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