Gallium nitride (GaN) is one of the III–V compound semiconductor crystals and is widely used in electrical device applications. In particular, the innovation of GaN-based blue light-emitting diodes (LEDs) has brought about device applications. In particular, the innovation of GaN-semiconductor crystals and is widely used in electrical equipment. Thus, GaN is now an indispensable material to meet the demands of energy conservation in recent years.

It is known that light irradiation affects the mechanical behavior of compound semiconductors, which is called the photo-plastic effect (PPE). Among them, II–VI semiconductors are typically hardened by light irradiation near their fundamental absorption edge. Furthermore, it has recently been revealed that ZnS single crystals show brittle fracture by compression under light conditions, while they plastically deform up to $\varepsilon = 45\%$ in darkness. It is considered that the significant plasticity change is originated from the interactions of an electrical excess charge and electrons/holes excited by light irradiation at their nonstoichiometric dislocation cores.

Dislocations in GaN can have a nonstoichiometric core, and thus GaN is also expected to show a PPE. However, the PPE of GaN has not been well investigated so far. In this study, we examined the PPE of GaN by nanoindentation experiments under controlled ultraviolet (UV) light conditions. Furthermore, microstructures of the indented regions formed with and without UV irradiation were characterized by transmission electron microscopy (TEM). We will discuss the effects of UV irradiation on the mechanical behavior of GaN.

A schematic illustration of the experimental apparatus used to perform the nanoindentation tests under UV irradiation. The UV irradiation was controlled by the current flowing through the LEDs.

The plasticity of inorganic semiconductors is affected by light irradiation, which is called the photo-plastic effect (PPE). In this study, we examined the PPE of gallium nitride (GaN) by nanoindentation with controlled light conditions. The nanoindentation experiments revealed that the hardness of GaN (0001) surface is increased up to $\sim 5\%$ under ultraviolet (UV) light irradiation in comparison with one measured in darkness. Transmission electron microscopy observations showed the activation of basal slip and pyramidal slip for both samples indented with UV irradiation and in darkness. It can be suggested that the hardening of GaN is originated from the deterioration of dislocation mobility due to UV irradiation.

Key-words: GaN, Hardness, Nanoindentation, Photoplastic effect

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NOTE

Ultraviolet light induced hardening in gallium nitride

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The plasticity of inorganic semiconductors is affected by light irradiation, which is called the photo-plastic effect (PPE). In this study, we examined the PPE of gallium nitride (GaN) by nanoindentation with controlled light conditions. The nanoindentation experiments revealed that the hardness of GaN (0001) surface is increased up to $\sim 5\%$ under ultraviolet (UV) light irradiation in comparison with one measured in darkness. Transmission electron microscopy observations showed the activation of basal slip and pyramidal slip for both samples indented with UV irradiation and in darkness. It can be suggested that the hardening of GaN is originated from the deterioration of dislocation mobility due to UV irradiation.

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of NSPU510CS,⁸ it has the peak wavelength of \( \lambda = 375 \text{ nm} \) (3.3 eV) and some emission intensities in the range of \( \lambda = 350–410 \text{ nm} \) (3.0–3.5 eV), which covers the bandgap of GaN (3.4 eV). Its current-radiant characteristics is approximately linear in the range of 0–80 mA. The light intensity of the UV irradiation device was controlled to flow a constant direct current using a regulated power supply unit. The light intensity of the UV irradiation device was measured up to its absolute maximum rating of forward current \( (I = 20 \text{ mA}) \), and it was 248 \( \mu \text{W/cm}^2 \) at 10 mA, 586 \( \mu \text{W/cm}^2 \) at 20 mA at the center of the device, showing a linear relationship.

Nanoindentation tests were performed using the load-controlled indentation mode, where the applied load \( (P) \) was linearly increased from zero to a maximum value \( (P_{\text{max}}) \) in 10 s, held for 1 s, and decreased to zero in 10 s. Test conditions were \( P_{\text{max}} = 10 \text{ mN} \) and current flow from \( I = 0 \) (without UV irradiation) to \( I = 60 \text{ mA} \), increasing in 10 mA steps. For each current condition, \( N = 10 \) indentation tests were consecutively performed.

The microstructures of two indented regions formed by the conditions of \( I = 0 \) and 60 mA were examined by TEM. TEM samples were prepared using a focused ion beam system (Helios G4 UX, Thermo Fisher Scientific, Waltham, MA, USA). Each indented region was protected by carbon deposition, and a thin section was cut there and lifted out onto a copper grid. The samples were further thinned to have electron transparency. They were observed along \( \{1100\} \) direction by TEM (JEM-2100HC, JEOL, Tokyo, Japan) operated at an accelerating voltage of 200 kV.

Figure 2(a) shows typical load-displacement \( (P-h) \) curves taken by the nanoindentation experiments under the conditions of \( P_{\text{max}} = 10 \text{ mN} \) with \( I = 0 \) and 60 mA. The two curves are smooth knife-shaped and no apparent pop-in events, suggesting that the samples were mainly deformed by dislocation slip. The indentation depth \( h \) is 162.9 nm for \( I = 0 \text{ mA} \) and 160.0 nm for 60 mA. This means UV irradiation suppresses the deformation. The averaged hardness \( (H) \) evaluated from \( P-h \) curves \( (N = 10) \) taken by nanoindentation tests with the different current flows using Sawa and Tanaka’s method⁹ are shown in Fig. 2(b). Comparing the hardness measured with and without UV irradiation, the hardness increased by \( \sim 5\% \) in general with light irradiation. There is less dependence between the hardness value and light intensity, suggesting that a saturation limit of UV intensity for hardening may be achieved even at \( I = 10 \text{ mA} \).

Figure 3(a) shows a cross-sectional dark-field TEM image of an indented region formed without UV irradiation taken using the \( g_{0002} \) reflection near the \( \{1100\} \) zone axis. The dashed line indicates the indented surface. The image contrasts seen from the contact point to \( \sim 300 \text{ nm} \) inside correspond to dense dislocation structures formed by large compressive stress during indentation. In the lower region, relatively sparse linear contrasts are seen, and they can be divided into three types: ones along the horizontal line, at an angle of \( \sim 19° \) from the vertical axis, and at an angle of

![Fig. 2](image-url)  
(a) Typical load-displacement curves obtained by nanoindentation tests under darkness \( (I = 0 \text{ mA}) \) and UV irradiation \( (I = 60 \text{ mA}) \). (b) Averaged hardness \( (N = 10) \) was evaluated from the nanoindentation test with different current conditions. The error bar indicates the standard error range.

![Fig. 3](image-url)  
(a) Dark-field TEM images of samples fabricated from indented regions with and without UV irradiation (scale bars = 200 nm). (a) \( I = 0 \text{ mA} \), (b) \( I = 60 \text{ mA} \). The dashed line in each image indicates the edge of the sample. The observation direction is along the \( \{1100\} \) zone axis.
~30° from the vertical axis. A former TEM study revealed that the slip systems of \{0001\}\{1120\}, \{10\overline{1}1\}\{21\overline{1}3\} and \{11\overline{2}2\}\{11\overline{2}3\} can be activated by indentation onto the [0001] surface of GaN.\(^{10,11}\) From the crystallographic orientations, it is reasonable that the three types of linear contrasts are assigned to slip dislocations associated with \{0001\}\{1120\}, \{10\overline{1}1\}\{21\overline{1}3\}, and \{11\overline{2}2\}\{11\overline{2}3\}, respectively. Note that the slip dislocation on \{0001\} plane should not contribute to image contrast of \(g_{\overline{1}002}\) reflection because of the extinction rule for TEM imaging (\(g.b = 0\)),\(^{12}\) while Fig. 3(a) shows some image contrasts corresponding to [0001] slip dislocations. This would be because the image contrasts are contributed from a projection of many dislocations formed on a [0001] plane, and such dislocations may not follow the simple extinction rule based on the kinematical imaging theory.

Figure 3(b) shows a cross-sectional dark-field TEM image of an indented region formed with UV irradiation (\(I = 60\) mA). As with Fig. 3(a), dense dislocation structures underneath the contact point and sparse linear contrasts in the lower region are seen. Since the linear contrasts appear to be in almost the same directions, it is considered that the same slip systems were activated by indentation. This suggests that the deformation mode does not depend on UV irradiation. In addition, careful comparison of the two TEM images in Fig 3(a) and 3(b) found that the dislocations in Fig. 3(b) seem to be more winding and to have a weaker contrast. The TEM contrast of dislocation is sensitive to imaging conditions, such as incident beam direction and sample thickness, although it can reflect the structural information of a dislocation. In the present cases, the imaging conditions were not controlled enough to interpret the structural information from the contrasts. To characterize the detailed structure of dislocations formed with and without UV irradiation, a more precise TEM analysis is needed to be employed.

Our results demonstrated the UV light induced hardening of GaN. Since the same slip systems were activated regardless of UV irradiation conditions, it can be considered that the mobility of dislocations is deteriorated by UV irradiation. Former reports on PPE of ZnS suggested that electrons and holes excited by light irradiation stabilize nonstoichiometric partial dislocation cores with an excess electrical charge,\(^{6}\) and this interaction can result in the deterioration of their mobility. In the present case, electrons/holes should be induced in the GaN sample by the UV irradiation, and nonstoichiometric partial dislocations are possible to form in GaN with the wurtzite structure.\(^{7}\) Therefore, the UV light induced hardening of GaN is expected to be due to the interaction between the dislocation cores and the excited electrons/holes similar to the case in ZnS.

The light that contributes to the electron/hole generation is easily absorbed within a surface region in a crystal. For GaN, the UV light intensity exponentially attenuates with the penetration depth and drops below 1% when the depth reaches 500 nm.\(^{13}\) The effects of UV irradiation to dislocations should be limited to such a shallow region from the surface. This consideration implies that the PPE of GaN may not exert a large influence on the mechanical behavior of a bulk crystal.

The present nanoindentation experiments could detect the PPE of GaN, but there is still room to examine the quantitative relationship between the increase of hardness and UV intensity. The present method has an intrinsic difficulty: the indenter tip blocks the UV irradiation to the sample at the contact point. Therefore, the effective intensity of UV light in the deforming region during indentation is difficult to be estimated. To investigate PPE of GaN for more precise, nanomechanical tests, such as nanopillar compression under controlled light conditions, will be helpful.

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