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SPECIAL ISSUE ARTICLE

A nanoindentation approach for unveiling the photoplastic effects by a "light switch": A case study on ZnO

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Abstract

The nanoindentation method is a widely utilized approach for characterizing the mechanical properties of materials at the nanoscale. In typical nanoindentation tests, the mechanical responses of materials are monitored while maintaining constant environmental factors, such as lighting and temperature, in order to ensure the reliability of the results. Here, we propose a testing method that switches the light conditions during a single nanoindentation creep test to detect slight changes in the mechanical response due to weak light illumination. To achieve this, a reference sample of fused silica was employed, which is insensitive to light, in order to compensate for the thermal expansion/contraction of approximately 1 nm due to the light environment. The calibrated results revealed the instantaneous suppressive influence of light illumination on the indentation creep behavior of ZnO. It was found that upon initiating illumination, the indentation creep rate decreased by 45%, whereas terminating illumination led to a dramatic 19.4-fold increase in the creep rate. The effective testing pattern involving a "light switch" enables quantitatively visualizing the light illumination effects through the instant "jump" in the creep strain rate within a single test, facilitating the detection of minor and instantaneous effects of light environments on indentation creep behavior.

KEYWORDS
plasticity, nanoindentation, oxides, wurtzite, compound semiconductors

1 | INTRODUCTION

Nanoindentation, an instrumented indentation technique that continuously monitors the applied load and depth throughout the loading and unloading cycle, has ushered in significant developments since Oliver and Pharr established a standardized analytical framework in 1992.[1] Nanoindentation is generally used to evaluate various mechanical properties at the nanoscale, such as the elastic modulus,[1–4] hardness,[1–5] steady-state creep rate,[6] and residual stresses,[7,8] through load–displacement (P–h) curves. Classical nanoindentation experimental modes include a quasistatic protocol to obtain Young's modulus and hardness,[9] a creep pattern to study rate-dependent deformation behavior,[10] and continuous stiffness measurement to estimate dynamic contact stiffness.[1] Consequently, nanoindentation has become a standard method for exploring mechanical issues in nanomaterial science and solid-state physics,[4,11] with every innovation in nanoindentation testing sparking a new wave of research.

Over the past three decades, nanoindentation techniques have been substantially developed, with the integration of external fields constituting a crucial research branch. For example, photoindentation,[12] which combines a state-of-the-art nanoindentation tester with a fully controlled lighting system, has enabled the assessment of the influence of light on dislocation behavior in crystals. Photoindentation studies have revealed that visible light affects the dislocation motion more than the dislocation nucleation across several wide-bandgap semiconductors.[12–14] Furthermore, incorporating electric and magnetic fields during nanoindentation tests provides insights into how these external stimuli affect the mechanical properties of the material.[15–18] Integrating thermal fields into nanoindentation testing promotes the investigation of temperature-dependent mechanical properties.[19,20] These advancements have expanded the fundamental understanding of how materials behave under different external fields and provided new possibilities for designing novel materials with properties tailored by environmental variables.

Generally, nanoindentation tests are performed in an environment with constant conditions, such as under unchanged light conditions or a fixed bias voltage; this consistent setting is crucial to ensure the reliability and repeatability of the results. Typically, large amounts of data must be collected to validate the influence of external stimuli. For instance, to examine the impact of light on ZnS, nanoindentation creep curves and dislocation behaviors in darkness and under other light conditions were acquired and compared.[12] We recently reported the suppressive effect of light illumination on dislocation motion in (0001) ZnO crystals, based on nanoindentation tests together with transmission electron microscopy (TEM) observations.[13] At that time, the difference in the nanoindentation creep curve due to the light environment was not remarkable, and the instantaneous effects of light conditions were unclear. As a result, the effect of light illumination on dislocation motion could not be fully explained based on nanoindentation creep curves alone.

Oshima et al.[21] employed a method of changing the light conditions during the deformation of bulk samples and succeeded in quantifying the effect of light illumination on dislocation motion. However, in nanoscale tests, the method of switching the light conditions within a single test has rarely been attempted. In this study, we introduce a “light switch” during nanoindentation creep deformation for quantitatively evaluating the effect of light on materials. Here we succeeded in extracting the transient mechanical response associated with switching the light conditions for the (0001) ZnO single crystals, taking into account thermal expansion/contraction. This has enabled us to clarify the instantaneous effects of light illumination on creep deformation within a single test. The approach proposed in this study will enable the instantaneous effects of light on the mechanical properties of materials to be evaluated in an efficient and reliable manner.

2 | MATERIALS AND METHODS

In this study, the same (0001) ZnO single crystals used in a prior study [13] were used. In addition, a plate of fused silica (FS) provided by ELIONIX, Inc. was used as a reference material for correcting thermal expansion/contraction. The FS sample measured 10.0 by 10.0 mm, with a thickness of 3 mm, and possessed a root mean square roughness of ~0.6 nm. Fused silica is insensitive to visible light, and the volume change of the FS sample under the light intensity adopted in this study was calculated to be negligible, making it a good candidate for calibrating volumetric changes due to switching light conditions.

Nanoindentation creep tests were conducted on the ZnO and FS using an ENT-NEXUS (ELIONIX Inc.) equipped with a diamond Berkovich indenter. The maximum loads P_{max} for ZnO and FS were set at 300 and 285 μN, respectively. Here, a slightly smaller 285 μN was employed for the FS so that the indentation depth in the FS would be equivalent to that of ZnO. The loading duration (t_1) was 10 s for ZnO and 9.5 s for FS, with a consistent loading rate of $\dot{P} = 30$ μN/s. All the experiments were conducted under load-control mode at a constant temperature of 30°C.

Figure 1 illustrates the load-time profiles under four different light conditions: (i) in darkness for 300 s, (ii) in light for 300 s, (iii) switching the light on after 50 s in darkness, and (iv) switching the light off after 50 s in light. For all lighting conditions, a light with a wavelength of 405 nm (slightly below the band gap of ZnO[22]) and an intensity of 40 μW/cm^2 was employed. The light intensity is also the same as that used in a prior study[13]; such weak light intensity does not cause detectable changes in the ambient temperature. However, a sudden change in light conditions could induce thermal expansion/contraction, affecting the monitored indentation depth, as detailed in the Supporting Information Material. For conditions (i) and (iii), the samples were placed in the dark for at least 6 h before performing any tests to provide sufficient time to remove the effects of ambient room light absorbed by the sample during preparation. Since ZnO, unlike ZnS[23] (as discussed later), does not exhibit phosphorescent properties, the effect of light illumination is not expected to last long. For conditions (ii) and (iv), the testing samples were illuminated with light 10 min before the tests to ensure the thermal equilibrium of the system at the start of the

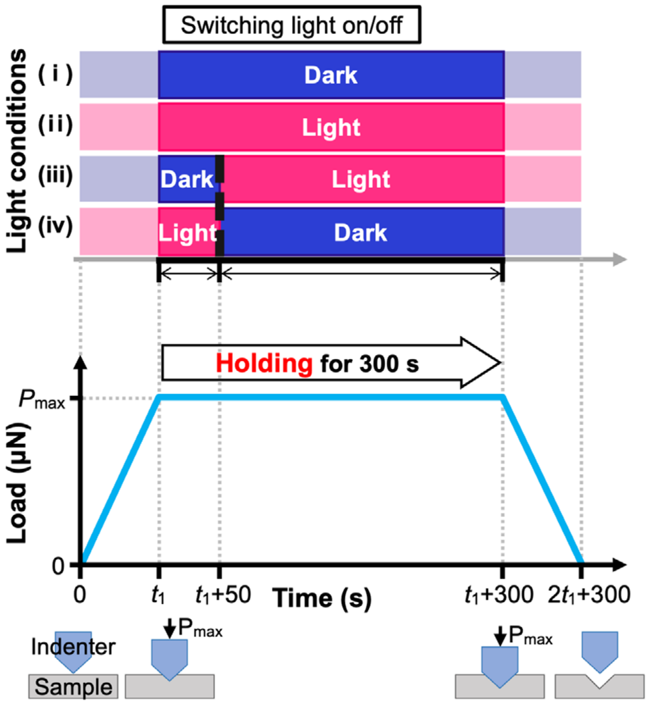

FIGURE 1 Load function and four light conditions in nanoindentation creep tests: (i) in darkness for 300 s; (ii) in light for 300 s; (iii) switching the light on after 50 s in darkness; (iv) switching the light off after 50 s in light.

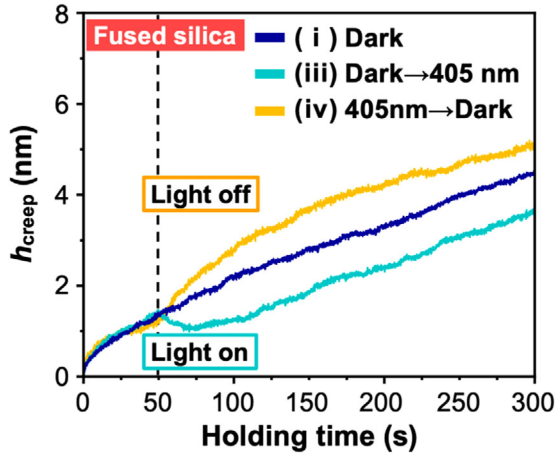

FIGURE 2 Monitored indentation creep depth as a function of holding time in 285 μN nanoindentation creep tests on fused silica (FS). The navy, light-blue, and orange curves show the results obtained under conditions (i), (iii), and (iv), respectively. Each curve represents the average of at least 30 curves.

experiment. Thermal drift was measured by monitoring the indenter tip displacement over 300 s at a constant load (10% of the maximum load). The calculated drift, less than 3.0×10^{-3} nm/s, was calibrated into all acquired curves.

3 | RESULTS AND DISCUSSION

Figure 2 presents the monitored indentation depth as a function of the holding time for the FS sample under different conditions: (i) 300 s in darkness, (iii) switching the light on after 50 s in darkness, and (iv) switching the light off after 50 s in light. The monitored indentation creep depth (h_{creep}) was calculated using (Equation 1):

$$h_{creep} = h - h_0, \tag{1}$$

where h represents the real-time monitored indentation depth, and h_0 denotes the monitored indentation depth at time zero in Figure 2 (t_1 in Figure 1). Each curve represents the average of at least 30 curves, with dashed black vertical lines marking the moment of switching the light conditions. As depicted in Figure 2, the three curves almost overlap during the initial 50 s, indicating little influence of light on creep deformation in the FS, possibly attributed to the large band gap of FS (9.0 eV[24]) compared with the photon energy of 405 nm light (3.1 eV), which can hardly excite carriers in FS. When the light conditions were switched at 50 s, a deviation between the three curves became evident. Under condition (iii), the creep depth decreases promptly after the introduction of light at 50 s, continues to decrease until 75 s, and then shifts to an increasing trend until the end of the creep test. Under condition (iv), when the light illumination was terminated at 50 s, the creep depth increased rapidly and then transitioned to increase at a relatively slow rate. Immediate deviations of approximately 1 nm at 50 s in the monitored indentation creep depths under the two light conditions (iii) and (iv), which are unexpected in conventional indentation creep tests, may result from the thermal expansion/contraction effects caused by the switching of the light conditions, suggesting the necessity of calibration to obtain the actual indentation depth. After ~100 s, the difference between the curve obtained in complete darkness (navy curve) and those subjected to altered light conditions during testing (light-blue or orange curve) hardly changed, suggesting stabilization of the thermal expansion/contraction effect.

The evolution of the monitored indentation creep depth with respect to the holding time for ZnO under the four light conditions is shown in Figure S1. It should be noted that the monitored indentation creep depth under different light conditions reflects not only the material's response but also the effect of thermal expansion/contraction due to the change in light conditions. As explained in detail in the Supporting Information Material Section 2, the switching of light illumination mainly causes the expansion/contraction of the Ti shaft for the indenter. On the other hand, the volume change of the diamond indenter

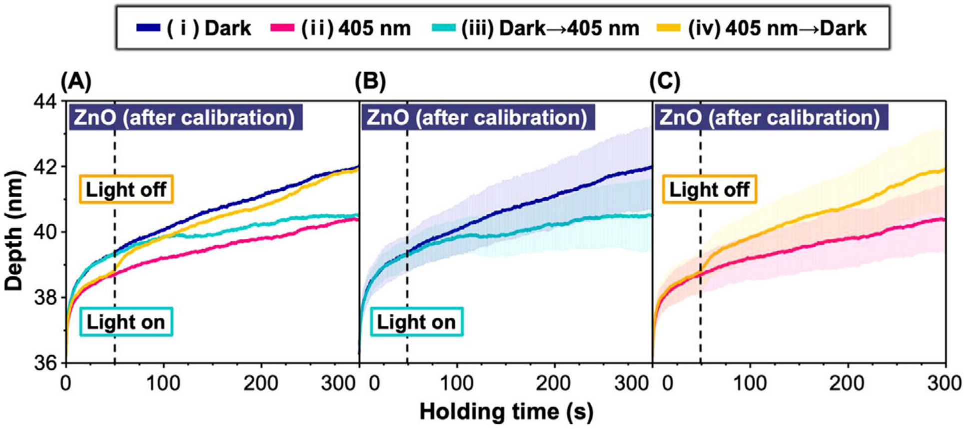

FIGURE 3 Calibrated indentation depth as a function of holding time of (0001) ZnO. (A) Calibrated depth–time curves under all four light conditions. (B) Calibrated depth–time curves for conditions (i) and (iii). (C) Calibrated depth–time curves for conditions (ii) and (iv).

and the ZnO sample is negligible. Therefore, we used the results from FS shown in Figure 2 to isolate the effect of the light-induced volume change of the Ti shaft on the experimental data.

The following Equations (2) and (3) were employed to compensate for the thermal expansion/contraction effects caused by the switching light conditions:

$$h'_{\mathrm{ZnO(iii)}} = h_{\mathrm{ZnO(iii)}} + (h_{\mathrm{FS(i)\_creep}} - h_{\mathrm{FS(iii)\_creep}}), \quad (2)$$

$$h'_{\mathrm{ZnO(iv)}} = h_{\mathrm{ZnO(iv)}} + (h_{\mathrm{FS(i)\_creep}} - h_{\mathrm{FS(iv)\_creep}}), \quad (3)$$

where h'_{ZnO}, h_{ZnO}, and $h_{\mathrm{FS\_creep}}$ represent the calibrated indentation depth of ZnO, the monitored indentation depth of ZnO, and the monitored creep depth of FS, respectively. The subscripts (i), (iii), and (iv) indicate the light conditions corresponding to (i) in darkness for 300 s, (iii) switching the light after 50 s in darkness, and (iv) switching the light off after 50 s in light.

Figure 3 shows the calibrated indentation creep depths for the (0001) plane of ZnO, which were corrected based on the results of FS. Figure 3A shows the average indentation depth under the four aforementioned light conditions. To facilitate data comparison, Figure 3B presents the results of conditions (i) and (iii), while Figure 3C presents the results of conditions (ii) and (iv). Each curve depicted in Figures 3A–C was derived by averaging at least 40 individual curves, where the black dashed vertical lines indicate the moment of transition in a light environment. The shaded regions, which match the colors of the curves and indicate the error bars in Figure 3B,C, refer to ± a standard deviation. In Figure 3A, the calibrated indentation depth in darkness for 300 s (navy curve) is notably larger than that under 405 nm for 300 s (pink curve), revealing the suppressive effect of light illumination on ZnO plasticity, consistent with the results of previous studies.[13,14,25]

In Figure 3B, before switching the light on, the light-blue curve aligns well with the navy curve, showing good reproducibility of the experiments in darkness. In Figure 3C, within the first 50 s in light, the orange curve overlaps with the pink curve, demonstrating the high reproducibility of the experiments in light. Notably, Figure 3B,C demonstrate a significant decrease/increase in indentation depth upon switching the light on/off, respectively, revealing that creep deformation decelerates upon the initiation of illumination and instantly accelerates without light. This instantaneous influence of light on plastic deformation has also been reported for the uniaxial compression of cubic ZnS single crystals.[21] Thus, the instantaneous suppressive effect of light on dislocation-based plasticity at the nanoscale in hexagonal ZnO was successfully determined through a single test.

To evaluate the nanoindentation creep rate (related to the mobility of dislocations), the indentation creep strain rate was further calculated using the scaling relationship $\dot{\varepsilon} \sim \dot{h}/h$, where h denotes the indentation depth.[26] Figure 4A illustrates the variations in the indentation creep rate with the holding time for conditions (i) and (iii), whereas Figure 4B shows the results under conditions (ii) and (iv). In both darkness (navy line) and light (pink line), the indentation creep rate initially drops sharply and then stabilizes at a steady state of constant decrease. The extremely low diffusion coefficient of 1.28×10^{-62} $\mathrm{cm^2/s}$ at 30°C[27] suggests that diffusion creep is unlikely to govern the room temperature nanoindentation creep of ZnO. Meanwhile, the stress exponents in darkness and light calculated from our experiments indicate that the dominant mechanism of indentation creep is dislocation creep, as detailed in Supporting Information Section 3. In Figure 4A, the curves for conditions (i) and (iii) overlap during the initial 50 s. Upon the initiation of illumination, the indentation creep rate depicted by the light blue

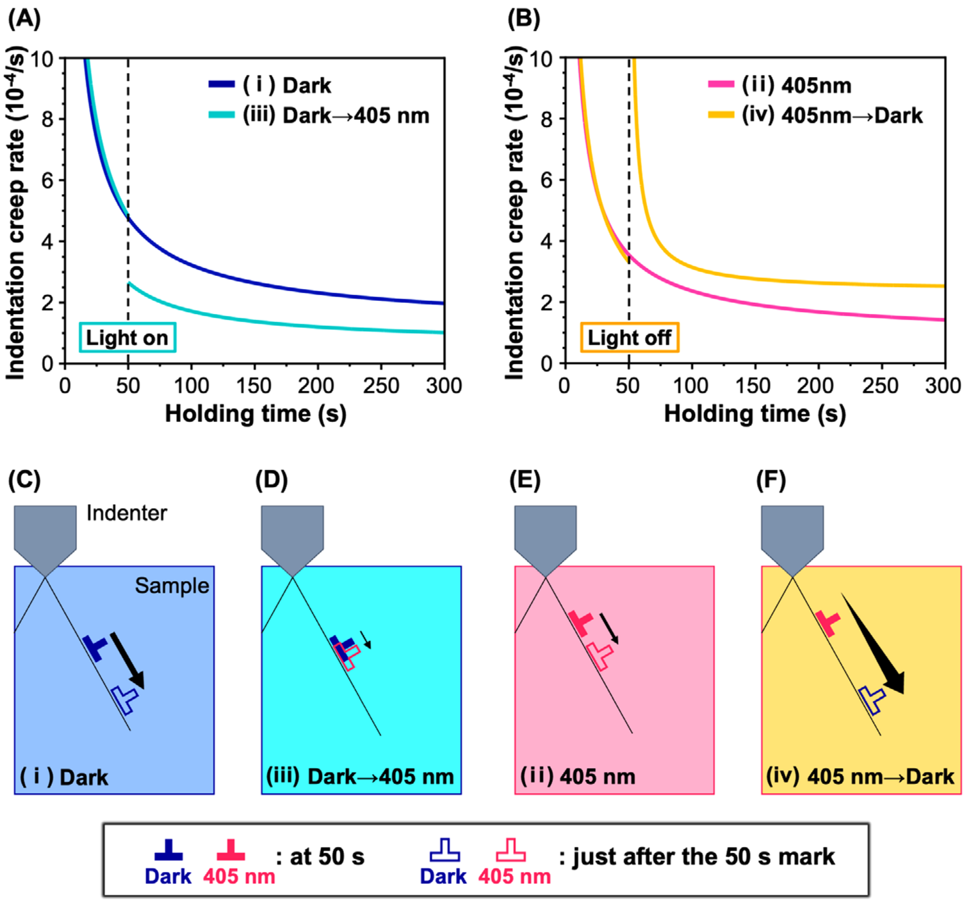

FIGURE 4 Indentation creep rate as a function of holding time in 300 μN nanoindentation creep tests: (A) for conditions (i) in darkness for 300 s and (iii) switching the light on after 50 s in darkness. (B) for conditions (ii) in light (405 nm) for 300 s and (iv) switching the light off after 50 s in light. A schematic of dislocation motion at 50 s and just after the 50 s mark for (C) condition (i), (D) condition (iii), (E) condition (ii), and (F) condition (iv). Solid and outlined dislocation symbols represent the location of typical dislocation at 50 s and just after the 50 s mark, respectively.

line dramatically falls by 45% before settling into a steady decrease at approximately 150 s. At the end of the creep test ($t = 300$ s), the indentation creep rate under condition (iii) is lower than that observed in light for 300 s. In contrast, Figure 4B reveals that upon the termination of illumination at 50 s, the indentation creep rate (orange line) momentarily surges by a factor of 19.4 before decreasing sharply from 50 s to ~100 s, followed by a gradual decrease at a nearly constant rate. The indentation creep rate at 300 s for condition (iv) is higher than that in continuous darkness for 300 s. As illustrated by comparing conditions (i) and (ii) in Figure 3A, the impact of light on the dislocation-based plasticity of ZnO was significant throughout the entire holding time. Switching the light conditions at the beginning of the holding time may include a slight influence from the transient creep during the initial stage of the test. However, switching the light conditions in the later phase of the holding time results in an already low indentation creep rate. Introducing light at this point would further reduce the creep rate, but the change may be too subtle to detect. Given the trade-off between reaching an equilibrium state and keeping a high indentation creep rate, 50 s would be an optimal choice for switching light conditions. As shown in Figure 4A,B, the sudden change in light conditions shall lead to an immediate "jump" in the indentation creep rate, revealing the instantaneous impact of light. The "light switch" proposed here enables the visualization of the instantaneous influence of light on the mechanical properties of materials through such a "jump" in indentation creep rate within a single test.

In a previous study on room-temperature creep tests of bulk ZnS crystals, a significant incubation time was observed when creep deformation resumed in the dark (light-off) after being loaded under light (where creep deformation hardly occurred[21]) because ZnS exhibits distinct phosphorescence owing to the long lifetimes of the photoexcited electrons and holes.[23] In contrast, ZnO does not exhibit such phosphorescence significantly, and thus the photoexcited carriers in ZnO tend to disappear immediately. Previous research on photoindentation tests of (0001) ZnO revealed that low-load nanoindentation mainly induces pyramidal dislocations, with light illumination found to inhibit their motion.[13] Postmortem cross-sectional TEM analysis demonstrated that nanoindentation-induced dislocations were present much deeper in darkness than in light.[13] Furthermore, Matsunaga et al.[28,29] reported that dislocations in compound semiconductors have the potential to capture photoexcited carriers through local electrostatic fields around the dislocation cores, leading to the reconstruction of these cores. Such structural changes result in an increased Peierls barrier, making the dislocation motion more difficult under light illumination. An increased Peierls barrier under light illumination was also demonstrated in photoindentation tests of ZnO samples with other orientations.[14] That is, the core structure of each dislocation in ZnO can also change reversibly depending on the light conditions such as in light or in darkness, and it is thought that there are no memory or history effects in the glide motion of each dislocation. Therefore, the memory or history effects due to the history of the light environment are considered to be negligible in the indentation creep behavior shown in this study.

Based on these insights, Figures 4C–F depict schematics of dislocation motion beneath the indenter at 50 s and just after the critical 50 s mark to elucidate the mechanism underlying the abrupt "jump" in indentation creep rate. At 50 s, the dislocations induced by indentation in the dark penetrate deeper than those in light. Accordingly, the solid navy symbols in Figures 4C,D are located deeper than the solid pink symbols in Figures 4E,F. For condition (iii), upon the initiation of illumination at 50 s, despite the minor change in the stress field,[14] the Peierls stress required for dislocation motion increases instantaneously. Such a rise leads to an instant decrease in the indentation creep rate just after the 50 s mark—illustrated by comparing Figures 4C,D. Conversely, for condition (iv), upon the termination of illumination at 50 s, the Peierls stress required for dislocation motion decreases instantly, resulting in a sharp increase in the indentation creep rate just after the 50 s mark, as illustrated by comparing Figures 4E,F.

For materials such as ZnO, light illumination tends to suppress dislocation motion[13]; hence, the initiation of illumination at 50 s would lead to an immediate decrease in the creep rate. Moreover, as the creep gradually transitioned to a steady state, the indentation creep rate inherently decreased over time. For condition (ii), the combined effects of natural creep evolution and the suppressive effect of light may lead to an inconspicuous transition in the indentation depth curve. In contrast, the increase in the indentation creep rate caused by the termination of illumination under condition (iv) was opposite to the time-dependent decrease in the indentation creep rate, resulting in a distinct inflection point at 50 s. This inflection point acts as an indicator to assess the influence of light. In materials such as ZnO, which display suppressed dislocation motion under light illumination, terminating the illumination during the creep process may offer a more notable method to discern the influence of light.

4 | CONCLUSION

In conclusion, we introduced a "light switch" midway through nanoindentation creep tests as an indentation pattern. The instantaneous suppressive influence of light illumination on the indentation creep behaviors of wurtzite ZnO was revealed by using fused silica (FS) as a reference sample. It is noteworthy that the "light switch" enables the visualization of the instantaneous influence of light on nanoindentation creep through a sudden jump in creep rate within a single test. The proposed method will facilitate the convenient and straightforward evaluation of the instantaneous effects of external fields, such as light illumination, on various ceramic materials.

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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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