Conoscopy as a Failure Analysis Method for Single Crystals

Hyoung-Seuk Choi¹,² and Duck-Kyun Choi²,*

¹ Korea Institute of Ceramic Engineering and Technology (KICET), Seoul 153-801, Korea
² Department of Materials Science and Engineering, Hanyang University, Seoul 133-791, Korea

Conoscopy is widely used to evaluate single crystals used as substrates on which epitaxial layers are grown in the LED industry, where the quality of the single crystal affects the reliability of the final product, the LED chip, and the package. However, the application of this method is currently restricted to characterizing birefringence. We performed conoscopy measurements on single crystals with failure modes (e.g., birefringence, lineages, dislocations, polycrystallinity, and amorphpousness) and examined whether it was possible to inspect such failures using conoscopy. Sapphire (α-Al₂O₃) and silicon carbide (6H–SiC) single crystals containing failures were investigated. X-ray diffraction and transmission electron microscopy analyses were also performed; their results were compared with the conoscopy results. Conoscopy was shown to inspect birefringence as well as other failure modes. Comparison of the conoscopic patterns obtained via simulation and experiment shows that quantitative evaluation of the failure level is possible. These results show that conoscopy can be used to quickly and easily investigate various failure mechanisms in single crystals.

Keywords: Conoscopy, Single Crystal, Crystal Growth, LED, Reliability.

1. INTRODUCTION

Single crystals with hexagonal close-packed structures, such as sapphire and silicon carbide, are widely used in the LED industry as substrates for epitaxial layers because they have good hardness and insulation and have the same structure as gallium nitride, which is commonly used as an epitaxial layer material.¹,² These single crystals provide the starting material in the LED manufacturing process (which advances from substrate to chip to packaging to completed LED). Therefore, failures on the surface of the substrate can cause abnormal epitaxial growth, and abnormal epitaxial growth causes chip failure; thus the quality of the single crystal influences the reliability of the final products.³ Figure 1 shows how defects on the surface of the substrate can cause problems in the epitaxial layer. In Figure 1, the GaN layer grows in a V shape because its growth originates along the V-shaped defect line on the sapphire substrate. Such abnormal growth weakens the strength of the LED chip and decreases the lifetime of the LED. So, LED manufacturers invest considerable effort in improving the quality of single crystals. The conoscopy method is one technique used to check for defective single crystals in the LED manufacturing process. Analyses using TEM (transmission electron microscopy) and XRD (X-ray diffraction) are the more precise methods for inspecting the failures in single crystals, but these approaches require sample preparation, which damages the samples and can only be used to inspect a nano-scale region, so it is difficult to adapt these techniques to the real-time needs of manufacturing facilities.⁴ In contrast, conoscopy can measure the full field of the surface of the sample and does not require sample preparation, but its resolution is insufficient compared to that of TEM or XRD. Also, conoscopy is currently only applied to the inspection of birefringence.

Figure 2 shows the optical setup for conoscopy. The input beam passes through the polarizer and is focused by a condensing lens, after which it is incident on the sample. After passing through the sample, the beam is focused again by an objective lens, is polarized by an analyzer, and finally is transformed into a collimated beam by a Bertrand lens. The parallel beam passing through the Bertrand lens makes a conoscopic pattern on the screen or eyepieces if the sample has a periodic lattice structure. Figure 3 shows the conoscopic pattern of a uniaxial crystal. The conoscopic pattern is composed of melatopes, isogyres, and isochromes.⁵–¹⁰ A uniaxial crystal has an axisymmetric conoscopic pattern so that

* Author to whom correspondence should be addressed.
it has one melatope and a circular isochrome. However, if the crystal is biaxial, the melatope is divided into
two, and the isochrome is distorted, becoming ellipsoidal.
These properties make it possible to measure the birefrin-
gence of the crystal. There have been various attempts
to make use of conoscopy to analyze the failures of sin-
gle crystals. Conoscopy has been used to determine
the refractive indices of crystals and to investigate
their optical inhomogeneities. However, until now,
the application of conoscopy in the LED industry has
been restricted to the determination of birefringence, and
even that depends on visual inspection (that is, quali-
tative tests using eyepieces). Single crystals have many
potential failure modes, such as birefringence, amorphous-
ness, dislocation, polycrystallinity, and pores. If various
other properties can be measured with conoscopy in addi-
tion to the birefringence, the conoscopy will provide a
better method for improving the quality of single crys-
tals, and it will thus improve the reliability of the LED
chip and package. In this research, conoscopy measure-
ments were carried out on single crystals having various
internal failures, and TEM and XRD analysis were per-
formed to find the failure mechanism. Then, we found
the relationship between the failure mechanisms and the
conoscopy patterns. Finally, we analyzed the advantages
and disadvantages of conoscopy for characterizing the reli-
bility of single crystals.

2. EXPERIMENTAL DETAILS
Sapphire (α-Al₂O₃, space group: R3c) grown by the
Kyropoulos method and silicon carbide (6H–SiC, space
group: C₆ᵥ-P6₃mc) grown by HT-CVD were used as
specimens. The sapphire samples have lineages and dis-
locations, and the SiC samples have amorphousness,
polycrystallinity, and birefringence. A trait that these two
materials have in common is that they both have hexag-
onal structures, so these materials are optically uniaxial
along the c-axis (a direction that is widely used in epi-
taxy). Therefore, we investigated the c-axes of these mate-
rials. Rocking curves were measured by XRD (RIGAKU
ATX-G, Japan), and diffraction patterns in real and recipro-
cal space were measured by TEM (JEOL JEM-4010,
Japan). XRD and TEM analyses were implemented on
the same specimen to allow comparison with the results
obtained by conoscopy.

2.1. Simulation
Simulations of the conoscopic pattern were implemented
to visualize the relationship between the shape of the cono-
scopic pattern and the birefringence. Simulation of the
conoscopic pattern can be performed based on Eq. (1)

\[ I = I_0 \sin^2 2\varphi \sin^2 \frac{\delta}{2} \]  

(1)
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Here, $\varphi$ is the angle of the isogyre, and $\delta$ is the phase retardation between the two polarization axes, $x$ and $y$, which can be calculated from Eq. (2)

$$\delta = \frac{2\pi \rho}{\lambda} (n_x - n_y) \sin \theta_1 \sin \theta_2$$  \hspace{1cm} (2)

Here, $n_x$ and $n_y$ are the refractive indices along the polarization axes, $\rho$ is the density of the material, and $\lambda$ is the wavelength of the incident monochromatic beam. If the crystal is birefringent, it has two optic axes, and $\theta_1$ and $\theta_2$ are the angles between the incident beam and the optic axes. Many algorithms for simulating conoscopic patterns have been developed.\textsuperscript{16–19} In this work, algorithms from Olorunsola\textsuperscript{19} were applied after setting the silicon carbide parameters. We set the parameters of the silicon carbide such that the thickness is 0.51 mm and the refractive indices are 2.55 for light polarized perpendicular to the $c$-axis and 2.59 for light polarized parallel to the $c$-axis.

The diameter of the measurement area is 3 $\mu$m, and the wavelength of the input beam is 633 nm.

2.2. Results and Discussion

The failure modes of sapphire and SiC were measured and analyzed using conoscopy, TEM, and XRD.

2.2.1. Birefringence

It is widely known that the birefringence can be investigated by observation of the conoscopic patterns. Figure 4(a) shows the defective SiC wafer and abnormal crystal growth. Figure 4(b) presents the conoscopic pattern of Figure 4(a); it shows the typical failure pattern in that the isogyre is tilted and the isochromes are distorted, becoming elliptical. The direction of the short axis of the ellipse in the isochromes corresponds to the direction of high refractive index. Such birefringence is an intrinsic characteristic of a biaxial material but can also occur in uniaxial material when lattice distortion caused by a temperature gradient in crystal growth causes local strain. So, the interpretation of the conoscopic pattern makes it possible to evaluate whether or not the single crystal grows normally. Figure 5 shows the reconstruction of the conoscopic pattern of the birefringence failure samples, obtained by simulation. The birefringences $\Delta n = n_x - n_y$ of these samples range in value from 0 to $15 \times 10^{-8}$. The conoscopic
pattern’s distortion cannot be observed when the birefrin-
gence is $1 \times 10^8$ (Fig. 5(a)); however, it can be observed
when the birefringence is over $2 \times 10^8$ (Fig. 5(b)), and the
amount of distortion is almost the same as in Figure 4
at $5 \times 10^8$ (Fig. 5(f)). Such results indicate that under our
present approach we have judged the crystal to be a fail-
ure when the birefringence is over $2 \times 10^8$. If conoscopy
is combined with simulation, quantitative evaluation will
be possible.

2.2.2. Lineages
Lineages are narrow lines on the surface of the single-
crystal wafer. In fact, these lines are the boundaries of the
polycrystals grown individually in the crystal growth pro-
cess. It is very hard to detect the lineages in the manufac-
turing process because they are very narrow and because
there is little difference in the intensities of beams pass-
ing through lineage regions and normal regions. However,
it is known that lineages can be detected by conoscopy,
but there has been no theoretical analysis of such detec-
tion. Therefore, we have deduced the theoretical basis for
this effect as follows. Figure 6 shows the conoscopy setup
used to inspect the lineages of the wafer. The reason that
the lineages can be seen well in the conoscopy setup is
explained in Figure 6. The optical beam passes through
the polarizer, the crystal having lineages, and the analyzer,
in that order. Lineages in the crystal act like a polarizer, so
that their presence can be expressed by the Jones matrix in
Eq. (3) when $\theta$ is the angle with respect to the y-axis.20,21

$$
\begin{pmatrix}
E_x \\
E_y
\end{pmatrix} = J_{LVP} J_{LRP} J_{LHP} E_0
$$

From the right-hand side, $J_{LVP}$ is the Jones matrix that
represents a vertical linear polarizer, $J_{LRP}$ represents a lin-
ear rotated polarizer, and $J_{LHP}$ represents a horizontal linear
polarizer, and they respectively stand for the polarizer, the
crystal with lineages, and the analyzer. $(E_0)$ is the Jones
vector of the incident beam, which has an arbitrary polar-
zation, and $(E')$ is the Jones vector of the output beam.
Equation (3) is simplified to obtain Eq. (4)

\[
\begin{pmatrix}
E_x \\
E_y
\end{pmatrix}
= \begin{pmatrix}
0 & \cos(\theta) \sin(\theta) \\
0 & 0
\end{pmatrix}
\begin{pmatrix}
E_{x0} \\
E_{y0}
\end{pmatrix}
\]

\[
= \begin{pmatrix}
\cos(\theta) \sin(\theta) E_{y0} \\
0
\end{pmatrix}
\]

(4)

The intensity is the square of the electric field and can be expressed as Eq. (4)

\[
I = |E|^2 = \cos^2(\theta) \sin^2(\theta)|E_{y0}|^2 = \cos^2(\theta) \sin^2(\theta)|I_0|^2
\]

(5)

The maximum intensity attained while changing $\theta$ in Eq. (5) is $(1/4)|I_0|^2$ at $\theta = 45^\circ$. This means that the intensity transmitting the lineages is only 1/4 that of the incident beam. However, the intensity of the conoscopic pattern varies from 0 to $|I_0|^2$ depending on the position on the conoscopic pattern and the null on the isogyre. Therefore, lineages can be inspected more effectively if $\theta$ is chosen such that the lineages are in the isogyre. Thus, conoscopy can be applied to inspect the lineages that occur when rolling the crystal. For example, Figure 7 is the image of the superposition of a lineage and the conoscopic pattern. The lineage is in the isogyre in which the conoscopy intensity is zero, and the angle between the lineage and isogyre is $45^\circ$, so that the intensity of the light passing through the lineage becomes maximum.

### 2.2.3. Dislocation

First, we measured

1. the conoscopic pattern (using conoscopy),
2. the rocking curves (using XRD), and
3. the crystal structure and reciprocal lattice (using TEM) of non-defective SiC (0 0 6).

In Figure 8(a), the melatope is located in the middle of the conoscopic pattern, and the two isogyres cross each other (intersecting at right angles) while passing through the melatope. The contrast of the isochrome is clear. Figure 8(b) shows the rocking curve measured by XRD; the FWHM of the non-defective single crystal is 34 arcsec. Figures 8(c and d) respectively present the crystal structure in real space and the diffraction pattern in reciprocal space, as measured by TEM. The TEM data shows that the crystal structure of the non-defective SiC is highly periodic, with no irregularities.
Next, we investigated the defective samples. We assume that there are many dislocations near the lineage because it is the boundary between two single crystals. Figure 9 shows the enlarged version of the image in Figure 7. The conoscopic patterns were measured while varying the distance from the lineage (the results of which are shown in Fig. 9) because, if the above-mentioned assumption is correct, the greater the distance from the lineage is, the fewer dislocations there should be. As a result, the conoscopic patterns become normal when measured far from the lineage (Fig. 9(a)) and distorted on the boundary (Fig. 9(b)). The distortion decreases when measuring completely within the lineage (Fig. 9(c)). An analysis using TEM was performed to investigate the cause of the distortion of the conoscopic pattern. Figures 10(a and b) show the crystal structure in the real and reciprocal spaces at

![Figure 11](image-url)  
**Figure 11.** Comparison of conoscopzy, rocking curves, and TEM for a combination of polycrystallinity and amorphousness. (a) Conoscopy pattern. (b) Rocking curve. (c) TEM image. (d) Diffraction pattern in region 1 of (c). (e) Diffraction pattern in region 2 of (c).

![Figure 12](image-url)  
**Figure 12.** Comparison of conoscopzy, rocking curves, and TEM for a crystal with amorphousness. (a) Conoscopy pattern. (b) Rocking curve. (c) TEM image. (d) Diffraction pattern in region 1. (e) Diffraction pattern in region 2.
the same position as Figure 9(a). These figures demonstrate that the crystal structure is good. Figures 10(c and d) show crystal structures in the real and reciprocal spaces of Figure 9(b). Figure 10(c) shows that there are many dislocations near the boundary. Figure 10(d) shows the superposition of two slightly offset diffraction patterns. So, it can be inferred that the strain caused by the mismatch between the growth of two different crystals formed the local birefringence and made the conoscopic pattern elliptical. Therefore, it can be inferred that the dislocation of the single crystal can be observed by conoscopy.

2.2.4. Polycrystallinity

In the case of polycrystallinity, the parameters \( n_x, n_y, \theta_1, \) and \( \theta_2 \) have different values in each region of the polycrystal. So, Eq. (2) can be converted into Eq. (6)

\[
\delta = \sum_{i=1}^{N} \delta_i = \sum_{i=1}^{N} \frac{2\pi\rho}{\lambda} (n_{yi} - n_{xi}) \sin \theta_1 \sin \theta_2_i
\]  

Here, \( N \) is the number single crystals which constitute the whole polycrystalline region and \( n_{xi}, n_{yi}, \theta_1i, \) and \( \theta_2i \) are the refractive indices along the polarization axes and the angles of the incident beam between the incident beam and the optic axes of the \( i \)th region. The elements in the sum on the right hand side of Eq. (6) (which we term \( \delta_i \) ) each have the same form as the sine wave originally given in Eq. (2) so that increasing the number of polycrystalline regions will decrease the contrast of \( \delta \) by superposition of additional \( \delta_i \) terms. Though each polycrystalline region is uniaxial, the polycrystalline regions usually have different optic axes. Therefore, the shape of the isochrome is distorted.

Figure 11(a) shows the conoscopic pattern of the SiC (0 0 6) single crystal; in the pattern, there is a distorted isogyre, and simultaneously the contrast is low. The distortion of the isogyre means that the material is polycrystalline. The poor quality of the contrast of the isochromce indicates the presence of polycrystallinity. XRD and TEM analyses were performed in such a way to conduct the failure analyses at the same position. Figure 11(b) presents the rocking curve, in which three peaks are superposed. This means that there are three polycrystalline regions. Figures 11(c–e) confirm that there are discrete regions in the polycrystal. Therefore, it can be inferred that the polycrystallinity of the single crystal can be observed by conoscopy.

2.2.5. Amorphousness

If the material is amorphous, the refractive index is the same regardless of the direction of incidence of the light. So, in Eq. (2), \( (n_x - n_y) = 0 \), and therefore, \( \delta = 0 \) also. This means that there are no optical path differences and no conoscopic pattern. However, if amorphousness and periodicity coexist in space locally and change continuously and the optic axes are the same, then the refractive index and optic axis become functions of position, and \( \delta \) in Eq. (6) will take the form of an integral, as shown in Eq. (7)

\[
\delta = \int\int\int \frac{2\pi\rho}{\lambda} (n_x(\vec{r}) - n_y(\vec{r})) \sin \theta_1 \sin \theta_2 d\vec{r}
\]  

So, as the contrast of the isochrome decreases, we can predict that there will be more amorphousness or polycrystallinity in the crystal as a result of the integration in Eq. (8). Figure 12 shows the results of analyzing the SiC (0 0 6) single crystals with interior defects, where the analysis is performed with conoscopy, TEM, and XRD. Figure 12(a) shows that the isogyre is normal, but the contrast of the isochrome is not good compared with that of Figure 8(a). The result of the rocking curve in Figure 12(b) reveals that its FWHM is 140 arcsec, which is much larger than the 30 arcsec (Fig. 8(b)) of a typical non-defective single crystal. This means that there is a larger amorphous region. The TEM analysis results in Figure 12(c) prove that there are amorphous regions mixed with crystalline regions. Figures 12(d and e) show the diffraction patterns of regions 1 and 2 of Figure 12(c). In Figure 12(c), the amorphousness changes slowly in-plane. These results indicate that as the amorphous region increases in size, the contrast of the isochrome decreases. This property of conoscopy makes it possible to measure the amorphousness of a single crystal.

3. COMPARISON OF TECHNIQUES

The advantages and disadvantages of conoscopy, TEM, and XRD are listed in Tables I and II respectively. Table I quantifies the performance of these techniques, while Table II quantifies the costs of these techniques. These tables show that conoscopy has worse accuracy than XRD and TEM but is simpler, more cost-effective, and capable of observing more failure modes, which are features of observing more failure modes, which are features.
that are more important in the development and production stages than in the research stage.

4. CONCLUSION

In this research, we investigated the various possibilities of using conoscopy to inspect the reliability of single crystals. The results of this research proved that conoscopy can be used to inspect not only the birefringence but also the lineages, dislocation, polycrystallinity, and amorphousness. The crystal failure mechanism can be classified by interpretation of the conoscopic pattern. Birefringence, dislocations, and polycrystallinity change the angle of the isogyre and the shape of the isochrome. Polycrystallinity and amorphousness change the contrast of the isochrome. Lineages can be detected clearly by the conoscopic setup. Also, it was found that the there is a possibility of quantifying the birefringence using the conoscopic pattern simulation. Additionally, a theoretical proof of how the lineage can be inspected by conoscopy was constructed. By comparing the advantages and disadvantages of conoscopy, TEM, and XRD, we showed that conoscopy can reduce the expense and time required to inspect crystal quality, which is valuable for mass production of devices. With these advantages, conoscopy is expected to play an important role in the improvement of the reliability of single crystals.

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References and Notes


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