Solid State Phenomena
ISSN: 1662-9779, Vol. 376, pp 47-54
doi:10.4028/p-KMZo3u

Submitted: 2024-11-13
Revised: 2025-05-19
Accepted: 2025-05-19
Online: 2025-09-10

Polarization Superimposed Phase Contrast Microscope Inspection of Dislocations in SiC Epitaxial Layer

R. Hattori*[1,a], Y. Yao[2,3,b], and Y. Ishikawa[2,c]

[1]Ceramic Forum Co. Ltd., Japan

[2]Japan Fine Ceramic Center, Japan

[3]Mie Univ., Japan

[a]hattori@ceramicforum.co.jp, [b]yao@icsdf.mie-u.ac.jp, [c]yukari@jfcc.or.jp

Keywords: phase contrast microscope, polarization, synergy, dislocation, SiC, TSD, TED

Abstract. We introduce a polarization superimposed phase contrast microscope (PS-PCM) for wide-gap semiconductor wafers as a new analytical technique that enables non-destructive and three-dimensional characterization of threading dislocations; TSDs and TEDs in SiC epilayers and substrates, such as discrimination each other or detection of their inclination in the depth direction.

Introduction

SiC power devices are establishing themselves as highly effective energy converters owing to their splendid material features. However, further improvements in the production yield and their reliability are required to achieve that. The major killer defects in the epitaxial layer for SiC device operation are carrots, triangle defects, downfalls, half-loop-arrays, and micropipes [1-3]. Although their origins and formation mechanisms have not yet been fully clarified, most of them are thought to be related to the combination or decomposition of BPDs and TSDs extended from the substrate into the epitaxial layer. It has been confirmed that screw dislocations with large Burgers vectors become hollow cores and form micropipes [4]; however, it is still challenging to identify the screw dislocations with Burgers vectors of small: 2c-3c or mixed with "a" component [5] and has not been sufficiently verified. Therefore, a more detailed TSD related dislocation inspection is required.

For dislocation inspection, X-ray white beam topography [6], KOH etching technology [7], and transmission electron microscopy (TEM) are generally applied as reliable inspection methods. However, they have limitations for application because of their operational expertise or sample breaking for inspection. Rapid and simple operation dislocation analytical methods for epitaxial growth or device process manufacturing are monochromatic beam X-ray topography or polarization observation, but they have limitations in terms of depth resolution or ability to identify dislocation types. We developed a phase contrast microscope (PCM) designated for wide-gap semiconductor wafers as a three-dimensional nondestructive inspection tool discriminating TSDs and TEDs in SiC crystals [8]. The phase contrast images of TSD and TED are distinguishable enough for automatic classification with image processing. However, the phase contrast contrasts of dislocations are very sensitive to the uniqueness of each dislocation for a more detailed characterization of the dislocations.

In this paper, we propose a new technique that enables the identification of detailed features of TSDs and TEDs in SiC epitaxial layer by polarization superimposing on PCM optics. The principle of the PCM method is quite different from that of the polarization method; however, adding the polarization effect on PCM gives a further function for the dislocation inspection as a synergy effect, which is entirely different from adding two images.

Phase Contrast Microscopy for Wide-gap Semiconductor Wafers

PCMs are generally called "biological microscopes", widely used in microbial research and bio-cell structural analysis. Frederik Zernike, a Dutch Nobel prize physicist, developed PCM in 1932, which allows the observation of the internal structure of cells of moving microbes alive. However, semiconductor engineers were not familiar with phase-contrast microscopes. The primary reason for this was that semiconductor wafers such as Si and GaAs do not transmit visible light, so reflection microscopes were the mainstream for optical observation, and transmission microscopes were not familiar. However, wide-gap semiconductors such as SiC and GaN transmit visible light, making it possible to apply phase-contrast microscopes.

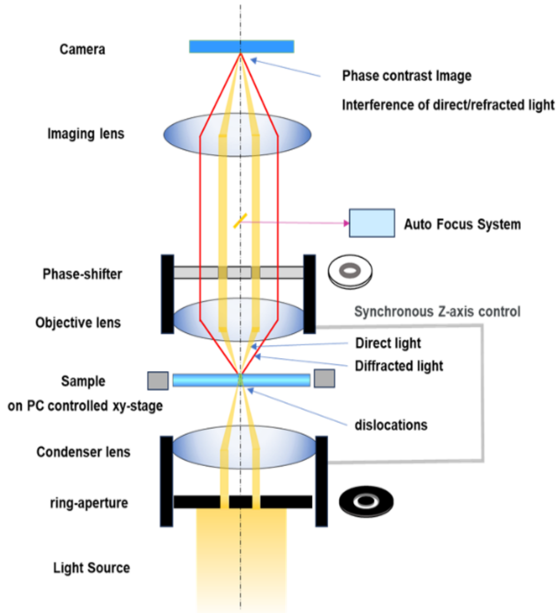
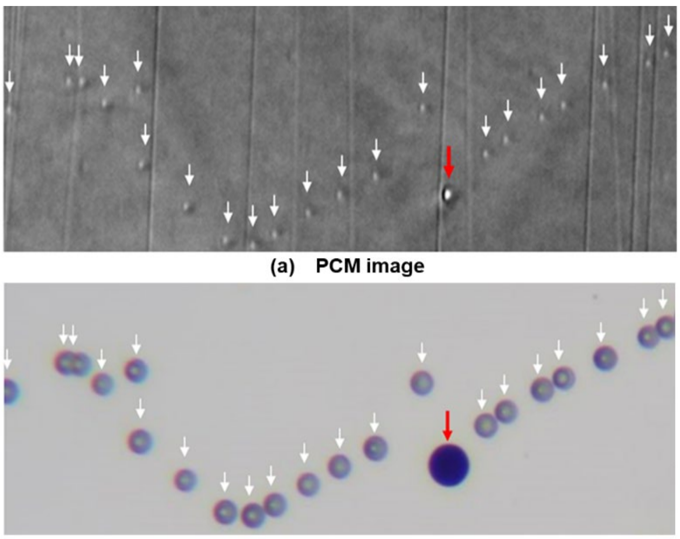


Figure 1 Optical system of Phase Contrast Microscope (PCM) for widegap semiconductor crystals

Fig.1 shows the schematic view of the PCM optics developed for semiconductor application, which implements an autofocus (AF) system on the transparent wafers, PC controlled x-y stage covering up to Φ200mm wafer, synchronized z-axis control system of objectives and condensers for wafer depth profiling, and image processing to extract and identify dislocations.



(a) PCM image

(b) KOH etch pits

Figure 2 PCM image and KOH etch pits of the same position of SiC 10μm thick epilayers.

Fig.2 shows the (a) PCM image and (b) KOH etch pit photo of the same position of slow-grown (10µm/hour) epilayer. Red arrows and white arrows indicate TSD and TEDs, respectively, which are in complete agreement in (a) and (b). This confirmed that the phase contrast image indeed detected the threading dislocations in the SiC crystal.

Another essential feature of the PCM method is the non-destructive tracking of the depth profile of dislocations inside the crystal by synchronous up/down of the focal positions of both the objective and condenser systems within the crystal.

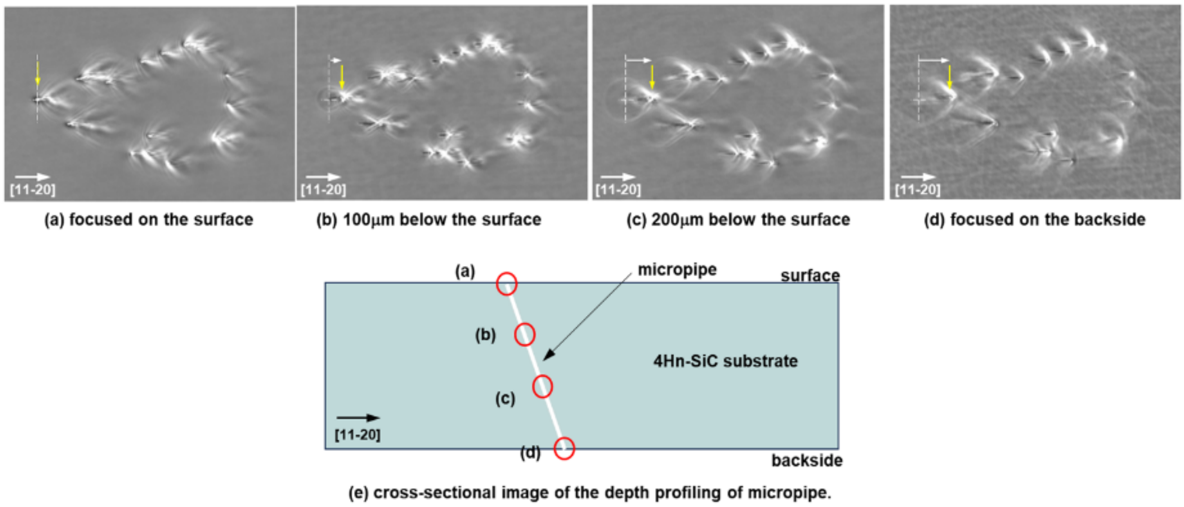
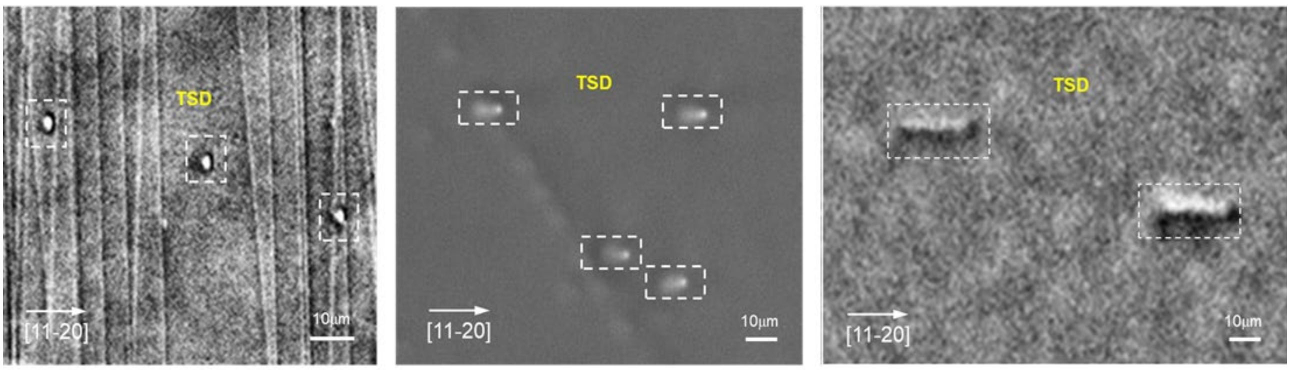


Figure 3 Non-destructive depth profiling of micropipes from top to back of SiC substrate with PCM. PCM images focused on (a) the surface, (b) 100mm below the surface, (c) 200mm below the surface, and (d) the backside surface, as indicated in (e) a cross-sectional schematic diagram of the depth profiling.

Fig.3 shows the depth profiling of the micropipes in the SiC substrate from (a) the top surface to (d) the backside, as described in (e) the cross-sectional view. We confirmed with SEM that there were actual holes on the top and backside surfaces that correspond to those MPs and that this kind of CPM contrasts should be MPs. Surface observation alone cannot provide conclusive evidence that this kind of defects are MPs, but depth profiling with CPM makes it possible to easily and non-destructively confirm which is MP [9-11].



(a) slow-grown epilayer (10µm/h) (b) medium-grown epilayer (15µm/h) (c) fast-grown epilayer (40-60µm/h)

Figure 4 Changes in TSD contrasts of PCM in response to the epitaxial growth rate.

However, it was confirmed that the PCM contrast of dislocations in the SiC epitaxial layer changed in response to the epitaxial growth rate, as shown in Fig. 4. The TSDs extend almost perpendicular to the growth surface with up to 10µm/h, but at faster growth rate than 10µm/h, they appear to be pulled in the down-step direction.

On the step-flow growth process, the Si-C bonds in the epi. growth reactor of each atomic step on the inclined surface repeatedly bonds laterally on each atomic terrace and overlaps, as a conclusion, which results in vertical growth. Higher growth experiences faster lateral stacking of Si-C bonds on the atomic terrace. We think such a lateral stacking process may affect TSD contrasts of the phase contrast microscope (PCM). Some parts of this result are qualitatively consistent with the contents of JEITA EDR-4712/200, the Japanese standard on the optical inspection of SiC defects.

Faster-growth epitaxy is becoming widespread in recent years, for higher throughput in single-wafer growth with uniformity of the epitaxial layer within the wafer surface. To investigate the reality of the horizontal segment containing TSD in this fast-grown epilayer, depth profiling was performed at two locations.

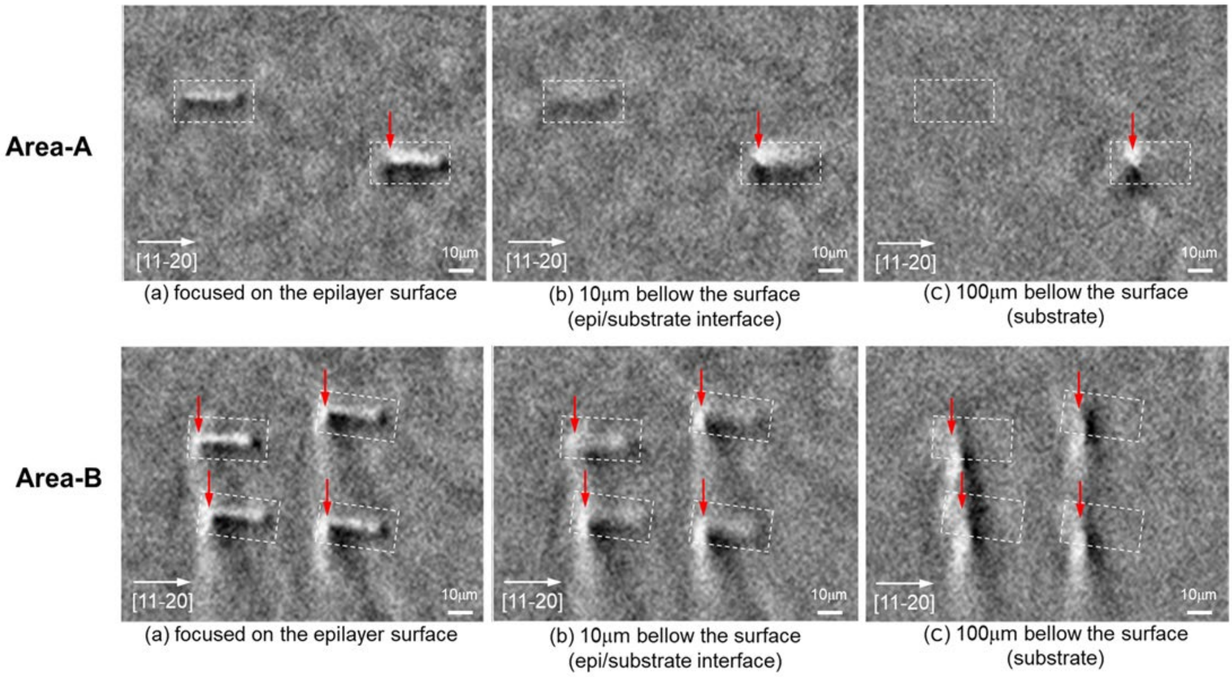
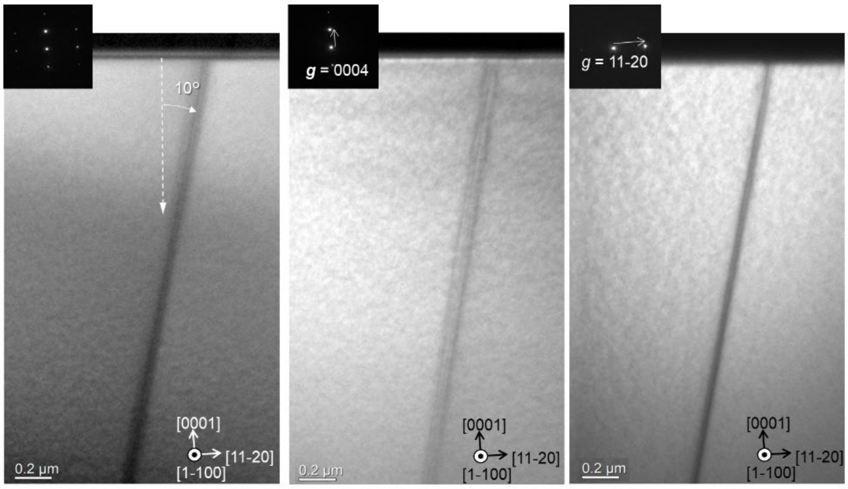


Figure 5 PCM depth profiling of continuity of TSD in fast-grown epitaxial from TSD in the substrate of area-A and B (a) Focused on the epilayer surface, (b) 10µm below the surface (epi./sub. interface), and (c) 100µm below the surface (inside of substrate).

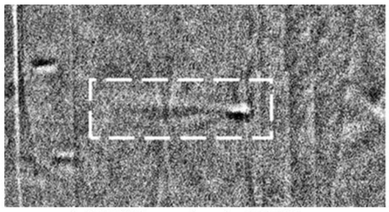
Fig. 5 shows the depth-profiling of area-A and area-B of fast-grown (60µm/hour) 10µm-thick epilayer; (a) focused on epilayer surface, (b) 30µm below the surface (epi./sub. Interface), and (c) 100µm below the surface (inside of the substrate). In area A, one of two segments appearing on the surface annihilates in the substrate, which we think may be caused by some surface roughness at the early stage of epitaxy, and the other one is connected with TSD contrast in the substrate. In area B, four segments are connected to the inclined dislocations in the substrate.

As shown in Fig. 6, to investigate the dislocation connected below the segment, the segment site was sliced in the [11-20] direction, and the cross-section was observed TEM in [1-100] direction and two beam excitation conditions of g=11-20 and g=0004. Consequently, the dislocation was the complex dislocation containing burgers vector of c component and a component and inclined 10° from the normal of epilayer surface, as described in Fig.7. Then, the direction of the inclination seems to vary for each dislocation.

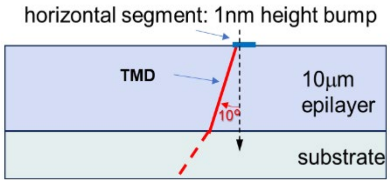


(a) [1-100] incidence (b) g = 0004 two-beam (c) g=11-20 two-beam

Figure 6 TEM inspection of TSD in horizontal segment defect in fast-grown SiC epilayer



(a) PCM contrast of TSD



(b) Schematic cross-sectional view of TSD as determined by TEM analysis

Figure 7 PCM contrasts of TMD in fast-grown (60µm/h) epilayer

Polarization Superimposed Phase Contrast Microscopy

Actual dislocations in epilayers and substrates take various forms, such as inclination, decomposition to partial dislocations, or mixing each other, and should appear in various optical features. To decipher the detailed characteristics of dislocations, we tried to superimpose the polarization effect on the PCM. Fig.8 shows the optics of polarization superimposed PCM (PS-PCM). There are three options for polarizers: linear polarizers, right-handed circular polarizers, and left-handed circular polarizers. As shown in the inset of Fig.8, the major setting angles of the polarizer and analyzer have many setting angles such as the [11-20] and [1-100] directions and the three a-axis of SiC crystal: ***a***1, ***a***2, and ***a***3.

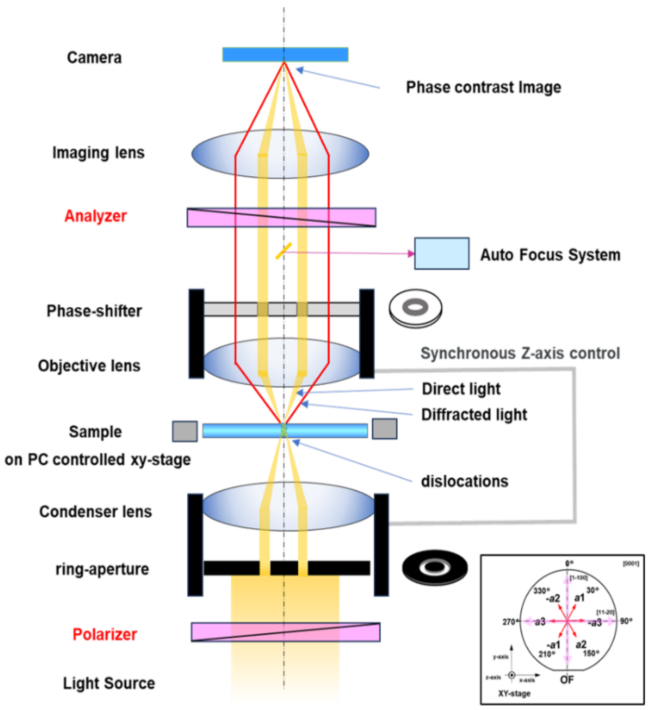


Figure 8 Polarization superimposed PCM

Since the irradiation from the PCM condenser is condensing through a ring slit, the incident wavefront is not parallel to the wafer, and the contrast seen in the transmitted polarization image hardly appears.

Fig.9 shows the PS-PCM images at the same position on the 10µm thick epilayer under various polarization conditions with (a) non-polarization as the reference, (b) cross-polarization with a polarizer in [1-100] and analyzer in [11-20], (c) cross-polarization with a polarizer in [11-20] and analyzer in [1-100], (d) right-handed circular polarizer and analyzer in a1, (e) right-handed circular polarizer and analyzer in a2, and (f) right-handed circular polarizer and analyzer in a3, where (a)~(f) with objective lens 10x of the depth resolution of < 2µm show selectively dislocations in the epilayer. (g) right-handed circular polarizer and analyzer in ***a***1, (h) right-handed circular polarizer and analyzer in ***a***2, and (i) right-handed circular polarizer and analyzer in ***a***3, where (g)~(i) with objective lens 4x of the depth resolution of > 50µm show dislocations both in the epilayer and in the substrate below the interface.

Yellow arrow contrast is TSD confirmed with KOH etch pit, and other contrasts may be TEDs. TED shows strong analyzer orientation dependence. Although further research is required to confirm whether it is related to the orientation of the Burgers vector of the dislocation, we think that the circularly polarized light irradiated on the dislocation is transmitted through it and polarized in the analyzer direction. By investigating the relationship between the direction of TED displacement and the transmitted light, it may be possible to estimate the direction of TED displacement from the dislocation contrast of PS-PCM.

The polarization direction dependence of dislocation phase contrast has been confirmed in various samples, but their interpretation has not yet been taken. Further investigation is required.

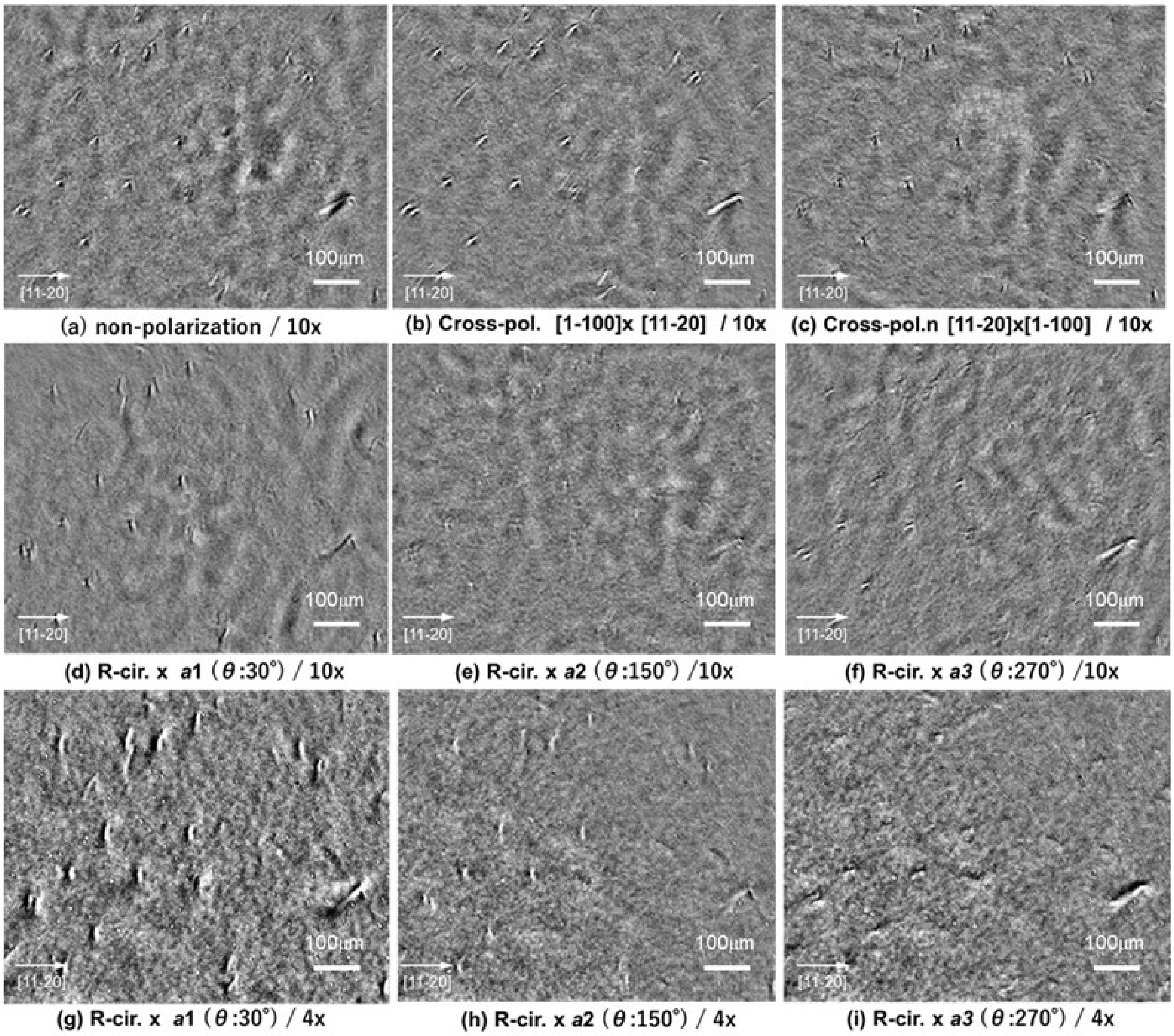


Figure 9 Polarization superimposed PCM images at the same position on the 10µm thick epilayer under various polarization conditions with (a) non-polarization as the reference.

Discussion

It is necessary to discuss how PCM or PS-PCM detects dislocations. Fig.12 shows the cylindrical tube model for simulating elastic distortion around the dislocation core of TSD and TED [12].In the model of elastic strain caused by dislocations, the stress diverges to infinity at the center of the dislocation core, so the region with a radius r0 of about five times the Burgers vector from the dislocation core is considered hollow and excluded from the calculation. The birefringence image that appears in polarized light observation is due to the modulation of the dielectric function due to this elastic stress, and is reproduced by analyzing the crystal strain due to elastic stress.

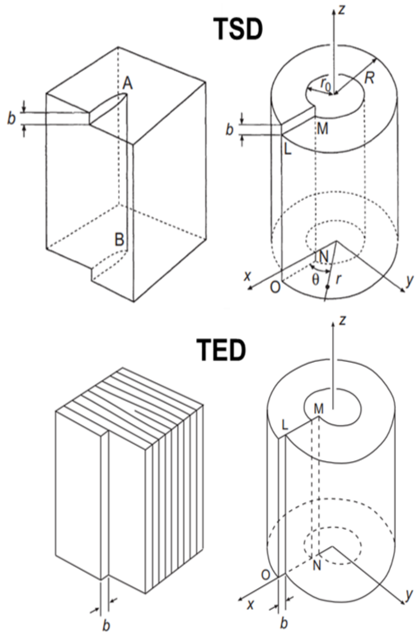


Figure 10 TED and TED lattice image (left) and cylindrical tube model for elastic stress analysis (right)

On the other hand, the dislocation contrast in the PCM image is thought to be caused by the refractive index modulation in the inelastic stress region inside r_0. However, the size of r_0 in TSD and TED is on the order of a few nm, and it is unclear whether it is effective for visible light optical systems. In addition, the analyzing-angle(θ) dependence of dislocation appearance, as shown in Fig.9 for circular polarized light irradiation, was not observed by birefringence images without the condenser for PCM. The synergistic effect of polarization on PCM may be the appearance of the uniqueness of each dislocation core's optical properties, rather than the crystal distortion effects.

Further research, such as analyzing the polarization characteristics of light passing near the dislocation core, may enable us to decipher the individual characteristics of the dislocation.

Conclusion

We developed a polarization superimposed phase contrast microscope (PS-PCM) for wide-gap semiconductor wafers and demonstrated that PS-PCM is sensitive to identifying the detailed uniqueness of dislocations in SiC crystals.

Acknowledgment

This study was partially supported by the METI Monozukuri R&D Support Grant Program for SMEs Grant Number JPJ005698.

References

[1] E. Van Brunt et al., Mat. Sci. Forum, vol. 924, pp 137-142.

[2] T. Neyer et al., Proc. IRPS 2021, pp 1-6.

[3] P. Fiorenza et al., Mat. Sci. Forum, vol. 1004, pp 433-438.

[4] S. Nakashima, et al., Appl. Phys. Lett. 77, 3612–3614 (2000).

[5] Y. Yao, et al., Materials Science Forum 858 (2016), pp389-392

[6] M. Dudley et al., Journal of Electron Materials, Vol.44 No.5 (2015)

[7] Y. Ishikawa et al., Materials Science Forum 717-720 (2012), pp367-370

[8] R. Hattori, et al., JAppl. Phys. Express 11, 075501 (2018).

[9] F.C. Frank, Acta Crystallographica, Vol.4, No.6, pp.497-501, 1951

[10] J.L. Liu, Material Letters, Vol.59, pp. 2374-2377, 2005

[11] J. Giocondi, J. Cryst. Growth, Vol. 181, pp. 351-362, 1997

[12] D. Hull and D.J. Bacon, chapter 4, Introduction to Dislocation, Elsevier