Application note

Crystalline Defect Characterization in Compound Semiconductors

with Electron Channeling Contrast Imaging (ECCI)

New semiconductor applications continue to push the need for greater performance, energy efficiency, and reliability from semiconductor devices, thus driving innovations in compound semiconductors (composed of chemical elements belonging to two or more different groups in the periodic table). For devices such as logic, power, photonics, radio frequency, and sensors, compound semiconductors offer superior properties compared to silicon-based products. And while compound semiconductors have the potential to provide significant performance benefits, manufacturing them has been marked by challenges. In this application note, we discuss compound semiconductors, the impact of crystalline defects, and the need for accurate crystalline defect characterization. This application note concludes with an overview of a high-efficiency workflow using electron channeling contrast imaging (ECCI) to characterize crystalline defects.

Compound semiconductors

Compound semiconductors have been used for decades in integrated circuits, photonics, memory, and power devices. With silicon fast approaching its physical limitations, compound semiconductors have become a promising solution to many challenges.

For the semiconductor industry, the majority of compound semiconductors of interest reside in groups IV, III-V, and II-VI. Examples of group IV include silicon carbide (SiC) and silicon germanium (SiGe). Examples of group III-V include gallium arsenide (GaAs), indium phosphide (InP), and gallium nitride (GaN). Examples of group II-Vi include cadmium selenide (CdSe), cadmium sulfide (CdS), cadmium telluride (CdTe), zinc selenide (ZnSe), zinc sulfide (ZnS), and zinc telluride (ZnTe).

In recent years, wide-bandgap compound semiconductor materials such as SiC and GaN have started to be adopted for applications in electrical vehicles, portable electronics, and 5G telecommunications.

Challenges

While offering great performance attributes, compound semiconductor devices present unique manufacturing challenges that have constrained their adoption as alternatives to silicon-based devices. One of the key challenges lies in reducing or eliminating crystalline defects during the wafer fabrication processes.

Creating compound semiconductors is accomplished using epitaxial growth, in which atoms are deposited on a surface one layer at a time under vacuum conditions. Epitaxial growth takes place on a substrate, such as silicon, and creates a new layer on which device structures are built. Building device structures

on the grown epitaxial layer is advantageous, as the resulting final structures tend to contain comparatively fewer crystalline defects and impurities that might negatively influence the performance of devices. However, during the epitaxial growth process, there are considerable chances that crystalline defects on the substrate can get extended into the epitaxial layer (see Figure 1). When defects such as a threading dislocation extend to the surface of the grown layer, they can cause catastrophic failures. The most common cause of this defect extension is due to a crystalline lattice mismatch between the grown layer and its substrate. Therefore, eliminating or reducing any crystalline defects during wafer manufacturing processes is the key to improving compound semiconductor device reliability and increasing manufacturing yields.

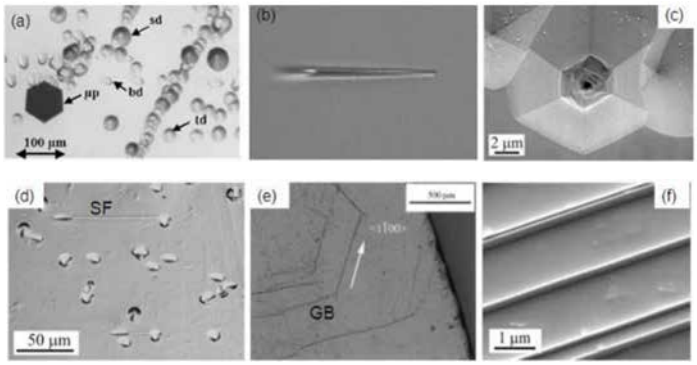

Figure 1. Examples of microstructural defects in SiC induced by epitaxial growth[1]

The first step to defect reduction involves monitoring the occurrence, density, and types of crystalline defects in the grown layers and characterizing their root causes. With conventional metrology methods, accurate and efficient monitoring and characterization of crystalline defects is a significant challenge. Each of the conventional metrology methods has critical limitations; they are either destructive and difficult to implement or simply don't have sufficient sensitivity and throughput to support wafer manufacturing. As a result, there is a need for an alternative, high-efficiency metrology method that can provide direct insights of crystalline defects and generate a better understanding of problems encountered during the wafer manufacturing processes. These insights become especially important when the production of new compound semiconductors moves on to larger-size wafers.

The solution

To solve these challenges, we have successfully developed a high-efficiency workflow using Electron Channeling Contrast Imaging (ECCI) on a Thermo Fisher Scientific™ Apreo™ 2 scanning electron microscope (SEM) system combined with Thermo Scientific Maps™ software for automated data acquisition. This workflow provides an optimal solution for crystalline defect characterization on compound semiconductors.

Principle

ECCI is an SEM-based technique in which the variation in the intensity of electrons backscattered from the crystal defect and the defect's close proximity is imaged. ECCI makes use of the strong dependence of the intensity of backscattered electrons on the orientation of the crystal lattice planes with respect to the incident electron beam. This intensity–orientation dependence is due to the electron channeling mechanism. For this purpose, the specimen lattice is oriented close to the Bragg condition for a selected set of diffracting lattice planes. Interestingly, slight deformations of the crystal lattice induced by the strain field associated with extended defects lead to a strong modulation of the backscatter intensity. Such modulations can be observed using a backscatter electron detector. Consequently, defects such as dislocations are imaged using the SEM on bulk samples, without the need to prepare electron-transparent specimens.

To achieve optimal visualization of defects, it is essential to have information on the orientation of the region of interest with respect to the incident electron beam, an equivalent of a diffraction pattern, and to be able to change this orientation in a controlled way.

With electron channeling patterns (ECP), there are two primary ways we recommend for obtaining crystallographic information from the sample: OptiPlan and Immersion modes. With both of these, the methodology is fairly straightforward and applies to large diameter (>5 mm) monocrystalline samples. In both modes, a scan is performed across the specimen in all directions. By scanning across a large area of the sample, the beam angle is varied. The change in angle produces contrast, enabling the creation of the ECP.

Procedures for Imaging of crystal defects in monocrystals by ECCI on Apreo 2 SEM via ECP

Alternative 1:
OptiPlan mode

1. Start imaging at following conditions: 20kV, 800 pA – 3.2 nA.
2. Bring the sample to WD = 2 mm, correct focus, lens alignment and stigmator. • *Do not perform lens alignment again after this step.*
3. Increase field of view to maximum (HFW=3 mm).
4. Adjust contrast and brightness so the ECP pattern is visible.
5. Use stage tilt and compucentric rotation to bring the selected diffraction condition (band in the ECP pattern) under the center cross.
6. Once the diffraction condition is set, zoom in, focus and stigmate the image. Use SE detector (T2, T3) to help adjust focus. • *Do not touch the lens alignment!*
7. Image dislocations by the T1 detector at following conditions:

    a. Field of view 10 µm or lower

    b. Dwell time 150 us (or even more)

    c. T1 brightness < 10%, contrast – set accordingly.

Use of the DBS detector for better image quality:

Once the diffraction condition is set as described in Step 5:

8. Move the stage to WD=7 mm. This will produce a zoomed-in image of the diffraction pattern, which allows for final tilt and rotational adjustments.
9. Insert the retractable DBS detector (test application will have to be used for tilt > 3 degrees)
10. Move the stage to 6 mm. Check safety on the CCD image
11. Continue according to Steps 6 and 7 (use ETD for fine tuning of focus and stigmation)

Alternative 2:
Immersion mode

1. Start imaging at following conditions: 20kV, 800 pA – 3.2 nA.
2. Bring the sample to WD = 1-2 mm, correct focus, lens alignment and stigmator. • *Do not perform lens alignment again after this step.*
3. Increase field of view to maximum.
4. Adjust contrast and brightness so the ECP pattern is visible.
5. Use stage tilt and compucentric rotation to bring selected diffraction condition (band in the ECP pattern) under the center cross.
6. Once the diffraction condition is set, zoom in, and image dislocations by the T1 detector at following conditions:

    a. Field of view 10 µm or lower

    b. Dwell time 150 us (or even more)

    c. T1 brightness < 10%, contrast – set accordingly.

7. Use SE detector (T2, T3) to focus and stigmate the image. • *Do not touch the lens alignment!*

Use of the DBS detector for better image quality:

Once the diffraction condition is set as described in Step 5:

8. Move the stage to WD=7 mm.
9. Insert the retractable DBS detector (test application will have to be used for tilt > 3 degrees).
10. Move the stage to 6 mm (5 mm if possible). Check safety on the CCD image.
11. Continue according to Steps 6 and 7.

Note:

The ECP pattern is rotated when the WD is changed in Immersion mode. The pattern center should stay under the center cross so the diffraction should be preserved. It is recommended to check by taking a low-magnification image with DBS inserted and adjusting if necessary.

Result

A typical example of ECP is illustrated in **Figure 2**. This was taken on a $Si_{0.3}Ge_{0.7}$ blanket layer with the position of diffraction conditions marked by the colored crosses. Based on the knowledge of the crystal structure and the pattern, a skilled operator can observe the dislocations under the desired Bragg conditions and evaluate their type and spatial orientation. In this case, we imaged the threading dislocations in two opposite {220} Bragg conditions, which resulted in inverse contrast. An example of the same threading dislocations imaged in two opposite diffraction conditions is depicted in **Figures 3(a)** and **3(b)** with the arrows showing the direction of contrast change.

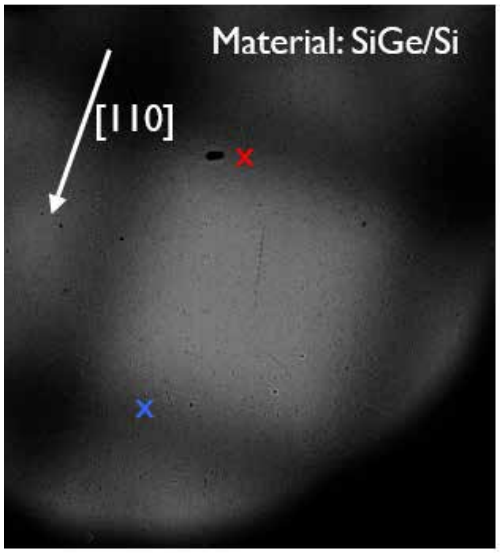

Figure 2. An ECP produced of the porous Ge layer on Si in OptiPlan mode. The crosses mark the two opposite {220} diffraction conditions used for further imaging. ***Image courtesy of Han Han, IMEC, Belgium***[1]

Summary

The deposition of novel compound layers such as SiGe and GaN on Si substrates is particularly challenging due to the large mismatch of the crystal lattice. This mismatch can lead to relaxation and extended crystalline defects, such as misfit and threading dislocations, stacking faults, and twinning planes. ECCI is a powerful, yet instrumentally undemanding, technique capable of imaging and identifying linear and planar crystal defects in semiconductor devices. This enables rapid characterization of defects and their density.

Reference

1. *Enhancing the defect contrast in ECCI through angular filtering of BSEs*, Han Han et al., **Ultramicroscopy**, Volume 210, 2020, 112922, ISSN 0304-3991, https://doi.org/10.1016/j.ultramic.2019.112922

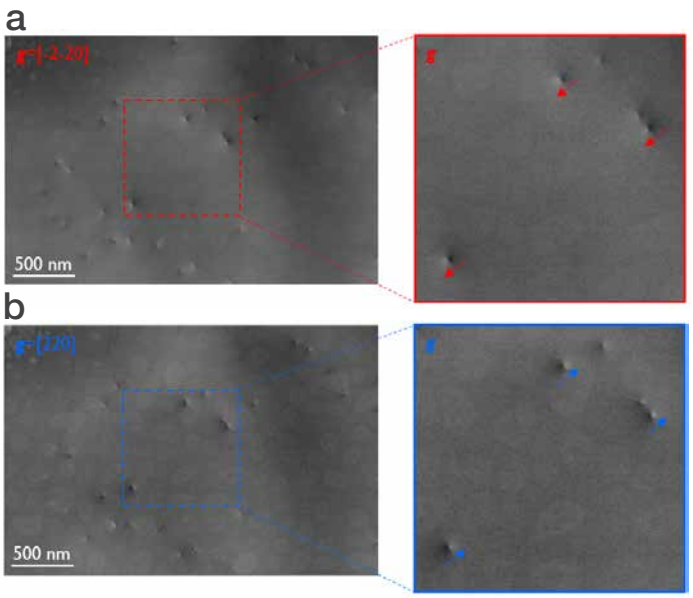

Figure 3. Threading dislocations imaged via ECCI method from diffraction conditions displayed in Figure 2 of the porous Ge layer on Si. The threading dislocations are photographed in (a) [-2-20] Bragg condition and (b) [220] Bragg condition, showing reverse contrast, which is highlighted by the arrows in the right images.
Images courtesy of Han Han, IMEC, Belgium[1]

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