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Precession Electron Diffraction (PED) Services

Precession Electron Diffraction (PED) analysis provides visualization of material phase, crystal structure and strain level at nanometer (nm) scale.

Due to the nature of electron beam (e-beam) and crystal materials interaction, e-beam, when hitting a crystal structure, gets diffracted to certain directions and form diffraction patterns, which carry various structural information. For example, diffraction spots arrangement reveals crystal orientation information, while spacing between diffraction spots carries crystal lattice distance information.

Using this principle, PED technique uses a focused e-beam in Transmission Electron Microscopy (TEM) setting to scan through an area pixel by pixel on a thin TEM lamella, which is typical less than 30nm thick and prepared by Focused Ion Beam (FIB) method. At each pixel in the 2D area, an electron diffraction pattern is generated and then recorded by a camera. For grain orientation mapping application in poly crystalline materials, diffraction pattern of each pixel is compared with the user given theoretical model, and thus crystal orientation of this diffraction pattern can be determined. Having the crystal orientation of each pixel in the 2D analysis area determined and been represented in different color for different orientation, we then can generate the crystal grain orientation maps. Similarly, for strain mapping application in single crystalline materials, diffraction spot spacing of each pixel is compared to strained free area and the difference of the spacing is calculated to generate the strain level data, which is then used to create the strain level maps.

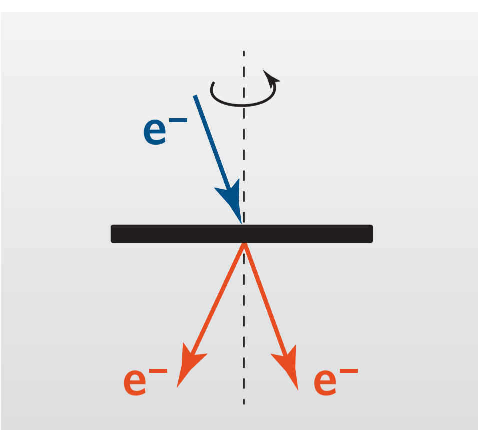

Figure 1: General schematic of how PED works

Strengths

- Can provide visualization of materials structure at nm scale
- High strain detection sensitivity (< 10^-3 level)

Limitations

- Significant sample preparation and analysis time (several hours to days)
- Relatively small sampling volumes (<5X5µm)
- Plan-view characterization can be challenging
- Materials with complex crystal structure, e.g. some ceramics, can be challenging
- Requires prior knowledge of materials phase/ structure, which can be determined by other techniques, e.g. EDS, EELS, XRD, etc

Common Applications

- Structural analysis for poly-crystalline materials, such as semiconductor metal line, via deposition, metal/ceramic film, poly-Si film:
    - Visualization of materials nanostructure through phase mapping and crystal grain orientation mapping

- Crystal grain size distribution and average grain size analysis
- Preferential growth direction characterization/texture information through pole figures

- Strain level mapping for single crystal, such as strained Si/SiGe, III-V materials quantum well devices

Industry Sectors and Technologies

- Consumer Electronics
- Batteries/Energy
- Semiconductors

Case Study: Nano-scale Grain Orientation and Grain Size Analysis of Metal Line in an Integrated Circuit (IC) Chip

In the past a few decades, the semiconductor technology node/transistor size has been decreasing. However, for metal interconnect lines that carry the current/signal, as the metal line size decreases, the resistance increases, leading to more heat and bigger power consumption. Especially, the size of metal lines has shrunk to tens of nanometer recently, and thus the problem becomes much more severe. Several approaches have been proposed to mitigate the problem. One of them is to manipulate the grain size of the metal line, as the electrons tend to scatter at grain boundary leading to resistance. However, characterizing and direct observation of the grain size at nanometer scale has been challenging until now due to the resolution limit by other techniques, e.g. SEM based EBSD. And here is how PED can help to characterize grain size at nm scale of different metal lines (Fig. 2).

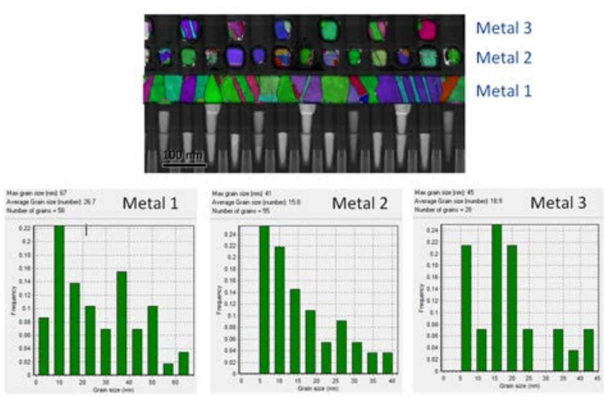

Figure 2: Grain size distribution of different metal lines of 7nm System On a Chip (SOC) application processor from a smart phone by PED analysis

Beside the grain size and grain orientation mapping, PED can also provide statistical quantification of the preferential growth direction (or called texture information) of the metal lines. Using pole figures, whether the grain has preferential growth direction or not can be accessed. In this case, the Cu <111> direction (Fig. 3) has preferential vertical growth direction, shown here in red cycle. This direction is 7 times higher than average direction, which is considered statistically significant.

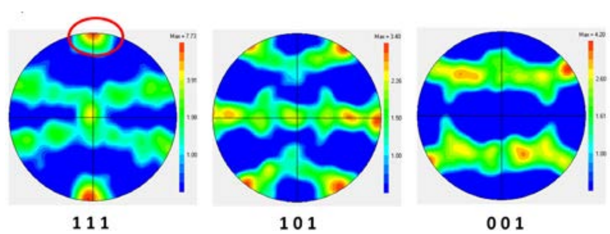

Figure 3: Pole figures of metal line (Cu)

Case Study: Nano-scale Strain Analysis of Strained Si

The speed that an IC chip can compute and process the data greatly depends on how fast the basic building blocks of the chip – transistor can switch on and off or how fast the signal can pass through the gate. By squeezing the Si lattice or in the other word introducing strain inside Si, the hole – the charge carrier of PMOS can move faster and hence increase the transistor speed. And how to characterize this small strain at nanometer scale is challenging until now. Fig. 4 shows strained level in 45nm technology node transistor with high sensitivity and high resolution by PED.

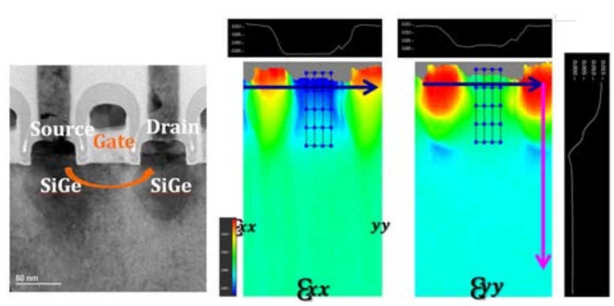

Figure 4: Strain mapping of 45nm technology node transistor

Complementary Techniques

PED analysis is a great complement to other structural analysis techniques, such as X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) based Electron Backscatter Diffraction (EBSD). XRD is a nondestructive technique, which provides very accurate and quantitative structural information, e.g. determination of phases, lattice constant, crystal symmetry information, etc. But, due to the nature of X-ray, the spatial resolution of XRD is relatively low (bigger than tens μm). Therefore, XRD is often use as a technique to screen the sample and provide phase candidates for further EBSD/PED analysis if microstructural analysis is needed. Having known the phase candidates, EBSD can be used to provide structural information, like grain orientation distribution mapping, at hundreds nm to hundreds μm level. If higher resolution is needed, PED, performed in Transmission Electron Microscopy (TEM) setting, can be used to visualize the structural information at the resolution of nm level, but often requires more complex sample preparation.

PED at EAG

At Eurofins Nanolab, we invest in new tools and technologies to provide our clients with the best data. We are currently the only commercial laboratory in the US offering PED analysis services, and that is just one of the many techniques and services we have to offer. With our years of expertise, knowledge, and wide range of analytical tools, we partner with our clients to help identify and solve problems. Contact us today to learn how we can help.