

CASE STUDY

PED and DPC

Introduction

Precession Electron Diffraction (PED) and Differential Phase Contrast (DPC) are novel techniques recently installed at Covalent and we've already been using these techniques to tackle tough R&D problems.

What is PED?

Precession electron diffraction (PED) is carried out in a TEM or STEM by introducing a small tilt to the incoming electron beam and then sweeping it in a circular, conical motion about the microscope's optical axis while the diffraction signal is acquired.

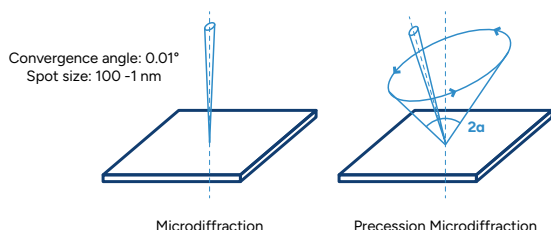


Figure 1: PED Diagram, courtesy of Nanomegas

By recording a PED pattern at each scan position in STEM, the data can be assembled into 2D maps of crystal phase, orientation, and local lattice parameters. Compared with conventional selected-area or nanobeam diffraction—where measured intensities often vary strongly due to dynamical (multiple-scattering) interactions—PED effectively averages over a range of incident angles, making results less dependent on exact zone-axis

alignment and sample thickness. The resulting intensities tend to be closer to the kinematical ideal (single scattering events instead of multiple scattering events), which generally improves the robustness of crystallographic indexing and quantitative interpretation.

What is DPC?

Differential Phase Contrast (DPC) STEM is a way to directly visualize internal electric or magnetic fields in and around a material while you scan it in STEM. It works by measuring tiny beam deflections caused by specimen charge at each pixel. To quantify the beam deflection from pixel to pixel, DPC uses a segmented STEM detector with 4 quadrants. As the sample's local polarization deflects the beam, the diffraction disk shifts slightly, and opposite detector segments see intensity changes. Those intensity differences are converted into field direction and relative strength maps

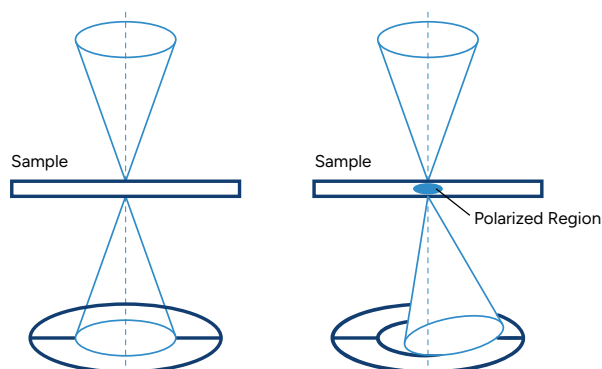


Figure 2: Illustration demonstrating beam deflection using DPC.

Case 1: Why did our GaN stack drift out of spec?

Gallium nitride (GaN) has emerged as one of the most important III–V semiconductors due to its exceptional combination of wide bandgap strength, high electron mobility, and thermal stability. These attributes enable efficient, high-voltage operation and support compact, high-power device architectures, making GaN a key material driving innovation across power electronics, RF communications, optoelectronics, and next-generation display technologies.

GaN is commonly used in High Electron Mobility Transistors (HEMT's), a type of field-effect transistor that gets its current-carrying channel from a heterojunction (an interface between two different semiconductors).

GaN is piezoelectric, which means it builds up charge when it's squeezed, stretched or bent. In layered AlGaIn/GaN devices, this feature helps create the HEMT channel. Piezoelectric polarization appears because the AlGaIn barrier is strained when it's grown on GaN. The two materials possess different lattice spacings and the strain that's induced as the lattices try to match each other creates a charge at the interface.

The piezoelectric qualities of GaN allow engineers to tune the electrical properties by designing layers that are meant to apply a given strain and therefore induce a given charge polarization.

However, the piezoelectric qualities can introduce failure modes because anything that changes stress/strain can change polarization fields. That means packaging stress, wafer bow, thermal cycling, and even local process-induced stress can show up as device variability and amplify reliability issues.

At Covalent, we use PED to perform nanoscale strain mapping to quantify strain gradients across the GaN/AlGaIn multilayers at the length scale most relevant to defects and interfaces.

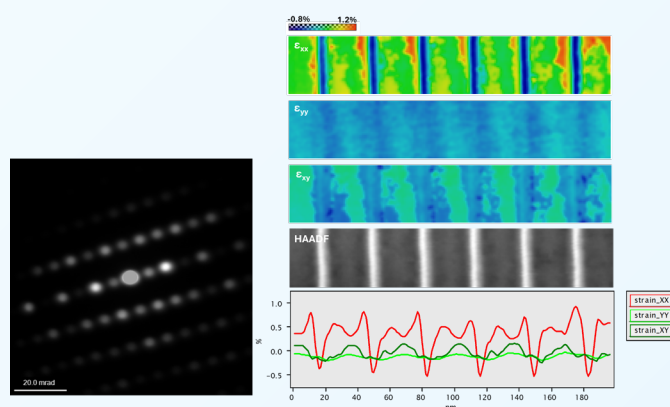


Figure 3: PED data showing strain on a GaN/AlGaIn stack

With PED, we were able to identify spatially varying strain (layer-to-layer and within layers) that aligned with microstructural features like grain orientation changes and boundary density.

We also use DPC to visualize the electrostatic impact of strain in the GaN/AlGaIn heterostructure. The resulting field visualization provides direct evidence of strain-induced local electric polarization, strengthening the link between microstructure/strain variability and electrical behavior in the GaN stack.

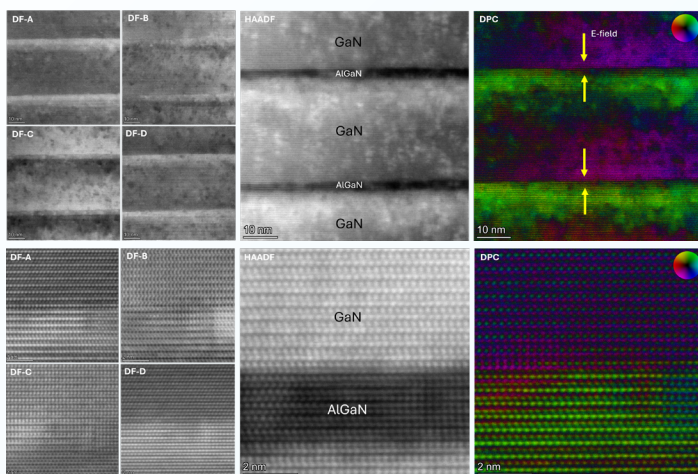


Figure 4: DPC results on GaN/AlGaIn stack

In the images above, the color wheel is a legend for the beam deflection at each pixel, and the colors encode the direction and strength of the beam deflection, which we interpret as a map of local electric-field variations.

In short, PED provides information about strain while DPC shows the electrostatic consequence of strain. Our clients can then use this evidence to adjust process conditions and tune their layers to reach the desired electrical performance.

Case 2: Are grain size and orientation the root cause of our NMC variability?

NMC (nickel–manganese–cobalt) materials are one of the main cathode chemistries used in lithium-ion batteries. It's crucial to understand grain size and orientation in NMC because these characteristics can drive rate capability, capacity fade, impedance growth, and safety margin for battery manufacturing.

Grain characteristics can impact:

1) Lithium transport and power performance

Smaller primary grains shorten diffusion distances inside each grain, which can improve fast charge/discharge performance. However, if grains are too small, the number of grain boundaries increases, which can become resistive or reactive depending on chemistry and cycling conditions.

2) Cracking and mechanical durability

NMC particles expand and contract as lithium moves in and out. That repeated strain can cause cracking along grain boundaries in small grains or cracking within grains in larger grains under stress. Cracking is undesirable because it creates fresh surfaces that react with electrolyte and accelerates degradation. Understanding grain size allows researchers to determine which failure mode dominates.

4) Grain boundaries as “highways” (and sometimes “trouble zones”)

Grain boundaries can act as fast diffusion pathways for ions or defects and/or initiation points for microcracks. Therefore, grain size is a proxy for grain boundary density, which is often what researchers are trying to control.

If performance varies between lots, grain size is one of the first microstructural suspects. SEM-based techniques like EBSD lack the resolution needed to resolve nano-scale grains.

At Covalent, in addition to EBSD, we use PED to quantify grain size distribution, crystal orientation and boundary density down to a minimum measured grain size of ~85nm.

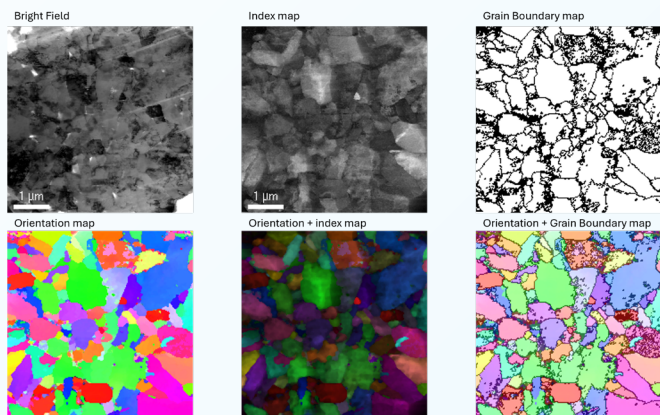


Figure 5: PED results showing grain boundaries, crystal orientation and index maps.

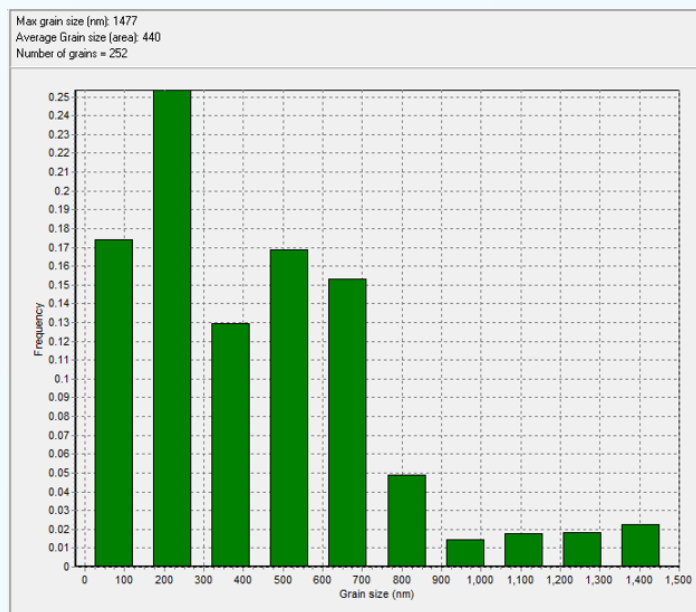


Figure 6: Histogram of grain sizes in sample.

For NMC cathode powders, small differences in grain size and orientation can translate directly into big differences in battery performance—fast-charge behavior, impedance growth, cracking, and cycle life. When a customer sees lot-to-lot variation but bulk methods and standard SEM imaging don't explain it, the missing variable is often internal grain structure. Covalent uses precession electron diffraction (PED) to map grain size distribution, crystallographic orientation, and grain-boundary density at the nanoscale (down to ~85 nm), well beyond the practical resolution of SEM-EBSD for these materials. This information enables our customers to compare processing variants, identify degradation-prone grain boundary networks, and tighten manufacturing conditions to achieve more consistent electrochemical performance.

Case 3: Is our “amorphous” film truly random—or does it have short-range order that is impacting material properties?

Amorphous materials are solids without long-range atomic order. However, being amorphous doesn't mean “atoms are placed with zero pattern.” Most amorphous materials still have consistent bond lengths and short-range structure. These features can strongly affect properties such as density, conductivity, hardness and refractive index.

Researchers in fields including semiconductor, photonics and energy storage occasionally create amorphous materials and need to know whether the atoms are arranged totally randomly or there is a specific short-range ordering that explains properties.

At Covalent, we use PED to address this question with electron pair distribution functions.

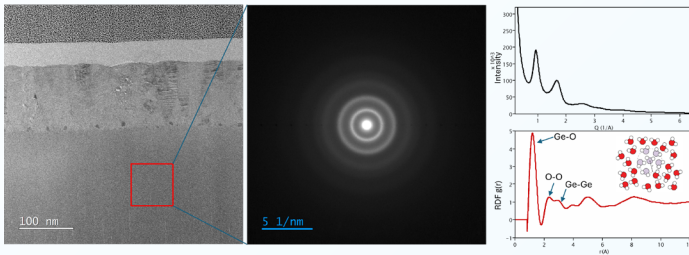


Figure 7: PED for e-PDF. Left: TEM image, Center: Diffraction pattern, Right: Radial Distribution Function

An electron pair distribution function (e-PDF) is a way to use electron diffraction to measure real-space atomic distances in a material and it is especially useful when the material is amorphous.

Process for collecting an e-PDF

1. Collect an electron diffraction pattern (often ring-like for amorphous materials).
2. Convert that diffraction intensity into a total scattering function.
3. Perform a Fourier transform to turn reciprocal space (diffraction) into real space → the PDF, typically written as $g(r)$.

This creates the radial distribution function (RDF) which shows the likelihood of finding another atom at a distance r from another atom. Peaks in this function occur at distances where lots of atom pairs exist (e.g., Ge-O, O-O, Ge-Ge).

Where PED fits in

Electrons strongly interact with matter, so electron diffraction can suffer from multiple scattering (dynamical diffraction): electrons bounce around and intensities get “warped.” This matters because PDF analysis assumes intensities are fairly faithful to structure. If intensities are distorted, the resulting $g(r)$ can be misleading.

PED helps because it averages diffraction over a small range of beam angles (precessing the beam). This tends to reduce the technique’s sensitivity to sample thickness and make intensities behave more like single-scattering events. This improves the reliability of the signal that is transformed into $g(r)$

When properties drift but XRD still reads “amorphous,” the missing answer is often hiding in local structure. With PED-based e-PDF, Covalent converts electron diffraction into a real-space radial distribution function to quantify atomic pair distances and detect subtle short-range ordering. That lets our clients distinguish “random amorphous” from locally organized networks, connect those differences to performance, and tighten process windows before variability becomes a yield or reliability problem.

Key Takeaways

In summary, PED and DPC give researchers a practical way to connect nanoscale structure to real-world performance—whether the material is crystalline, polycrystalline, or fully amorphous. Across these examples, PED adds quantitative, spatially resolved crystallographic and strain information that is more robust than conventional diffraction, while DPC reveals the electrostatic consequences of that structure in the form of local field variations. Together, these techniques help clients move past “it looks fine” bulk or low-resolution measurements and instead pinpoint the specific microstructural and bonding fingerprints that drive drift, variability, and reliability risk. The result is faster root-cause identification, clearer process knobs to turn, and higher confidence that a chosen formulation or process window will deliver consistent electrical, optical, or electrochemical properties at scale.



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