

Editorial

Special Issue: Electron Diffraction and Structural Imaging—Volume I

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In recent years, electron diffraction (ED) and structural imaging have undergone a major resurgence in the scientific community, driven by continuous advancements in transmission electron microscopy (TEM) instrumentation, such as Cs correctors, direct detection cameras and automation, and the development or expansion of analytical methods, such as cryo-EM, beam precession, 4D Scanning Electron Diffraction, 3D electron diffraction, 4D-STEM, and ptychography. These innovations have enabled the atomic-scale structural characterization of a wide range of nanocrystalline materials, spanning functional materials, zeolites, energy storage materials, minerals, organics, pharmaceuticals, and proteins.

One of the key strengths of ED lies in its low-dose capabilities compared to conventional imaging techniques, allowing for the study of beam-sensitive and nanocrystalline materials that would otherwise be inaccessible by laboratory or synchrotron X-ray techniques. Today, ED is widely applied in atomic structure determination, phase/orientation/strain mapping, electric field analysis, and even in the study of amorphous materials. Furthermore, the integration of in situ TEM holders (gas, liquid, and heating holders) has allowed the analysis of materials in real-time and near-natural conditions. All these applications benefit from the understanding and exploitation of symmetry at the atomic scale, from conventional space groups to higher-dimensional formalisms in incommensurate structures and dynamic symmetry evolution under external stimuli [1–13].

This Special Issue was published in two parts. Volume I includes 10 articles authored by international researchers, while Volume II features 9 original contributions that highlight the versatility, depth, and ongoing innovation in the field of electron diffraction and structural imaging.

The first paper by Orlova et al. presents a comprehensive study of LiNi_{0.8}Mn_{0.1}Co_{0.1}O₂ (NMC811) cathode materials using a combination of PXRD, HAADF-STEM, and electron diffraction tomography (EDT). The authors investigate cation disorder, particularly Li⁺/Ni²⁺ mixing, which strongly influences electrochemical performance. The defect formation mechanisms were studied through Rietveld refinement and various structure-sensitive metrics, while local variations were mapped using STEM. This multi-scale approach provides a full picture of disorder and demonstrates how complementary techniques can bridge the gap between bulk structure and local defect environments [14].

The second paper by Rauch et al. reviews the development and capabilities of precession-assisted ACOM-TEM (commercially known as ASTAR)—an automated tool for electron diffraction pattern indexing and orientation mapping in TEM using scanning precession electron diffraction data. Initially designed as an alternative to EBSD and XRD pole figures with nanometer-scale spatial resolution, ACOM/TEM has since evolved into



Received: 26 July 2025

Accepted: 30 July 2025

Published: 11 August 2025

Citation: Das, P.P.; Ponce-Pedraza, A.; Mugnaioli, E.; Nicolopoulos, S. Special Issue: Electron Diffraction and Structural Imaging—Volume I. *Symmetry* **2025**, *17*, 1288. <https://doi.org/10.3390/sym17081288>

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a powerful platform for phase identification and crystallographic analysis in TEM and is used in many material science labs. The paper addresses key technical challenges such as indexing complex diffraction patterns with overlapping contributions from multiple phases and resolving 180° orientation ambiguities. Future perspectives are outlined, including the 3D reconstruction of crystallographic phases within materials, further expanding applications in the field of advanced materials [15].

The third paper by Quintelier et al. applies precession electron diffraction (PED) and 3DED to the study of $\text{Li}_{1.2}\text{Ni}_{0.13}\text{Mn}_{0.54}\text{Co}_{0.13}\text{O}_2$ cathodes used in lithium-ion batteries. Due to the nanoscale structured domains and the beam sensitivity, traditional XRD and imaging fall short in quantifying phase changes. The authors developed a method to determine the volume ratio of spinel-like phases of honeycomb structures using low-dose electron diffraction. After 150 charge/discharge cycles, 4% of the volume converted irreversibly, providing insight into the degradation mechanisms in high-capacity cathodes [16].

The fourth paper by Yang et al. investigates whether cryogenic conditions are essential for 3DED of small organic molecules. The structures of sucrose and a newly reported compound, azobenzene tetracarboxylic acid (H4ABTC), were determined at both room and cryogenic temperatures. Despite common assumptions, the results show that when samples are stable in vacuum, room temperature data can yield structures of comparable quality, including hydrogen atom positions. The study emphasizes merging data as a key strategy to improve resolution and reliability [17].

The fifth paper by Tamari et al. resolves a long-standing debate regarding symmetry in high-temperature “Al₃Mn” T-phase alloys extended to the Al–Mn–Pt system. Using crystallographic and metallurgical evidence, the authors determine that the Al-rich phase is non-centrosymmetric (Pna21) while the Al-poor variant is centrosymmetric (Pnam). This work highlights the correlation between composition and symmetry in complex metallic alloys and provides a framework for further investigations [18].

The sixth paper by Mythili et al. focuses on retained austenite in reduced activation ferritic martensitic (RAFM) steels used in nuclear fusion reactors. The combination of synchrotron XRD, Mössbauer spectroscopy, and precession-assisted ACOM-TEM/ASTAR reveals a subtle retained austenite that is not visible with conventional methods. This study underscores the importance of microstructural homogeneity in optimizing the mechanical properties of RAFM steels [19].

The seventh paper by Klein et al. introduces precession-assisted low-dose electron diffraction tomography (LD-EDT) as a structure solution method for beam-sensitive nanocrystals. Examples include two synthetic oxides, a mineral, and an MOF, all solved and refined using dynamical diffraction data at doses below $0.1 \text{ e}^-/\text{\AA}^2$. This method enables the reliable structure determination of complex materials previously inaccessible by X-rays or traditional ED approaches [20].

The eighth article by Singh et al. examines the creep behavior of Nb- and Ta-rich γ -TiAl alloys using a suite of electron diffraction and imaging techniques, including precession-assisted ACOM-TEM/ASTAR, EBSD, and TEM. Creep tests at high temperatures reveal distinct microstructural instabilities and intermetallic phase formation (e.g., τ phase) in Ta-rich alloys. The results highlight how alloying elements influence phase evolution under stress [21].

The ninth article by Yang et al. explores Ruddlesden–Popper (RP) fault structures and interface chemistry in NdNiO_3 thin films grown via molecular beam epitaxy. Using aberration-corrected STEM and spectroscopy, the authors show that elemental intermixing and local strain lead to Ni valence variations at the faults. This microstructural understanding is essential for tuning functional properties in nickelates [22].

The tenth paper of the first volume by Das et al. provides a systematic evaluation of global optimization methods for solving structures from incomplete 3DED datasets collected with and without precession. In cases where traditional methods fail due to limited tilt range, beam damage, or dynamic scattering, the proposed approach enables successful structure determination. This study benchmarks global optimization against conventional structure solution paths, showing that it is a reliable alternative for difficult cases [23].

Conflicts of Interest: The authors declare no conflicts of interest.

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