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Enhanced nanoscale phase characterisation in modern steels using precession electron diffraction and energy filtering

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Background incl. aims
The development of lightweight and ductile Advanced High-Strength Steel hinges upon a comprehensive understanding of phase change kinetics during production. In addition to various techniques for chemical characterisation, transmission electron microscopy (TEM) is an essential tool for the structural analysis of examined phases. In this work, we showcase a significant enhancement in the acquisition of diffraction patterns (DPs) for nanostructured phases in modern steel systems, such as quenched and partitioned (Q&P) steels. We combined zero-loss energy filtering (EF) with precession electron diffraction (PED) to access crystallographic information of nano-inclusions inaccessible by standard diffraction techniques.

Methods
Selected area electron diffraction (SAED) is usually not suitable for the challenging task of nano-inclusions crystallography, which requires information about different zone axes for unambiguous phase identification. Due to the large aperture size, dynamic diffraction effects and, in particular, the intricate steel matrix containing arbitrarily oriented, strained and ferromagnetic grains, an in-depth analysis of SAED patterns is limited. To address these limitations and achieve a high lateral resolution, we employed site-specific nanobeam diffraction in PED mode with a beam diameter of approximately 1 nm and a precession angle of 1 - 3° around the central axis. Unlike standard DPs, where dynamical effects are unavoidable, PED provides quasi-kinematical results and a larger number of reflections through pattern integration, thus compensating for small variations in sample thickness or misorientation. The influence of inelastic and multiple scattering was mitigated through additional zero-loss filtering, noticeably enhancing the signal-to-noise ratio of our PED patterns. The experiments were performed with a JEOL JEM-2200FS equipped with an in-column Ω-filter and a TVIPS universal scan generator using a TemCam-XF416 CMOS camera.

Results
One application example is the investigation of iron carbides embedded in Q&P steels with varying Si or Al content, annealed at 320°C and 420°C. Our site-specific PED patterns provided enhanced clarity, revealing a multitude of carbide diffraction spots and enabling a more reliable analysis compared to results obtained via SAED. By conducting sample tilt series towards different crystallographic zone axes (Fig. 1), we achieved unambiguous identification of the carbide phase, its orientation relationship to the martensitic matrix, and the lattice parameters. The presence of orthorhombic θ-Fe_3C [2] was confirmed, in line with prior X-ray diffraction (XRD) measurements in the steel alloys annealed at 420°C. In alloys annealed at 320°C, where XRD results were inconclusive, the tilt series approach revealed the presence of transition carbides ε-Fe_3C (hexagonal) and η-Fe_2C (orthorhombic/quasi-hexagonal) [3][4]. Utilising the tilt series approach alongside the improved accuracy of the PED patterns allowed for a definitive distinction between ε- and η-transition carbides, overcoming previous challenges [1]. Further examples of PED applications are shown.

Conclusions

Our findings underscore the benefits of employing PED together with EF for characterising the crystallography of nanostructured phases in modern steel systems. Through comparison of DP quality, we demonstrate the insights attainable for complex samples by PED. The rising availability of commercial PED solutions and the convenient applicability of the PED method enhanced through EF, demonstrated on our examples, may incentivize the broader scientific community to embrace this method for investigating metals and alloys.

Graphic:

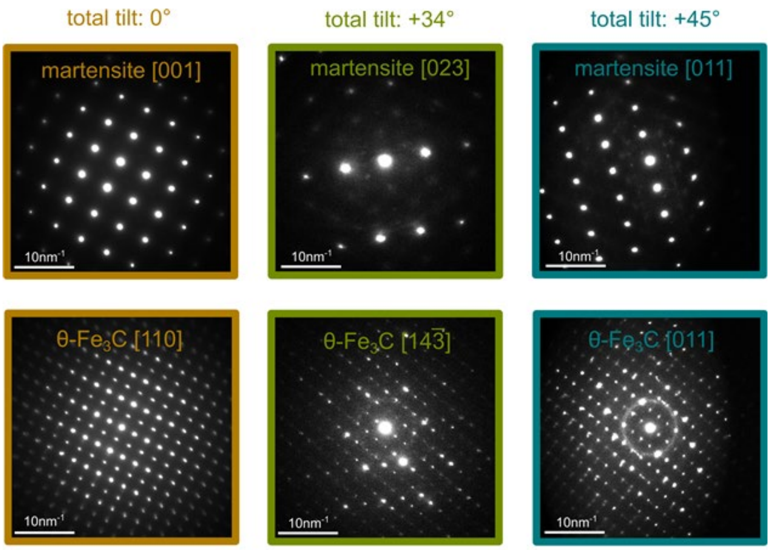

Fig. 1: Tilt series yielding DPs at three corresponding crystallographic zone axes of martensite and a θ-Fe_3C nano-inclusion.

Keywords:

PED, steel, nano-inclusions, phase characterisation

Reference:

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