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Scanning Precession Electron Diffraction and What We Get Out of Such Data in Studies of Aluminium Alloys

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The TEM Gemini centre in Trondheim, Norway has the last years had a strong focus on developing the scanning precession electron diffraction (SPED) technique. In particular, we have focused on using the technique to study nucleation and phase development of precipitates in age-hardenable aluminium (Al) alloys. In this talk, the experimental set-up, along with the acquisition parameters and the data analysis tools we use to study precipitation in Al alloys by SPED will be presented, as well as the results we have obtained. John Spence helped me doing TEM on Al alloys during my MSc thesis in Trondheim in 1990 and guided me on diffraction analysis during my stay as a PhD student in Arizona a few years later [1]. His wide and eager interests in materials and his deep knowledge of diffraction physics have inspired me during my years in electron microscopy☺.

Age-hardenable Al alloys are important structural materials for construction and automotive applications due to properties like high strength/weight ratio and good formability. These properties are often attributed to nanoscale precipitates in the Al fcc host lattice. The crystal phases, morphologies and distributions of these precipitates can be manipulated by chemical composition and thermo-mechanical treatment [2,3]. Precipitation starts out with clustering of solute elements and vacancies from a supersaturated, metastable solid solution in the Al matrix. Solving the structure, quantifying the distribution and determining phase fractions of clusters and precipitates are important for basic understanding and design of better alloys. SPED provides good statistics on the relative occurrence of the precipitates and hence have become a valuable tool for TEM studies of heat-treatable Al alloys.

For the acquisition, we use a JEOL 2100F microscope operating at 200 kV in the nanobeam diffraction (NBD) mode using the NanoMEGAS DigiSTAR and P1000 scanner scan generator. The convergence angle is ~1 mrad. The precession angle and frequency are tuned according to the experiment, typical values being 0.5-1.0° (~9-17 mrad) and 100 Hz, respectively, while the probe size is typically ~ 1 nm. Due to precession and aberrations, the actual probe size is somewhat larger. The double-rocking probe is aligned according to the approach described by Barnard et al. [4]. The scanning step size is set to ~1-2 nm, and the scan regions can be up to 1 µm^2. The 4D SPED datasets are aquired using a Medipix3 MerlinEM camera with a single 256 × 256 Si chip from Quantum Detectors. Diffraction patterns are recorded in 12 bits mode with an exposure time of ~20-50 ms per pixel. Since the precipitates have certain orientation relationships to Al, the SPED data are acquired in one of the major zone axes of Al, depending on the alloy system. This gives limited numbers of diffraction patterns for each precipitate type and simplifies the analysis.

The 4D SPED data stack is analyzed using the open-source python libraries hyperspy [5] and pyxem [6]. The first step of the analysis typically involves so-called virtual imaging, where a virtual aperture is placed in the SPED data stack and by integrating the intensity of the diffraction patterns within that aperture, a virtual image is created. The virtual image can either be a virtual bright-field (VBF) image, by placing the aperture onto the central beam, or it can be a virtual dark-field (VDF) image, by placing the aperture away from the central beam. To obtain phase maps of the precipitates in Al alloys we have tested different data analysis methods to assess their performance in terms of speed and accuracy. The methods include non-negative matrix factorization, template matching, a vector-based approach, and neural networks. The goal is to establish a fast, robust, and unbiased way of obtaining phase maps.

For the Al-Mg-Si alloy system, SPED has been a valuable tool for investigating the evolution of precipitates during artificial ageing [7]. We have also studied the result of adding low amounts of Cu, which significantly affects the phase fraction by an increase of the Cu-containing Q′-phase. In deformed Al-Mg-Si materials, SPED indicates that β” precipitates are sheared several times in single steps on different planes by dislocations during deformation [8]. In Al-Mg-Zn alloys, SPED is used in combination with density functional theory to study fine details of GP zones [9]. These and other results obtained from SPED will be discussed in the presentation [10].

References:

[1] R Holmestad (1994) Quantitative Electron diffraction. Energy filtering and studies of bonding effects in TiAl, Dr. Ing thesis, NTH.
[2] SJ Andersen et al., Adv. in Physics X **3**(1) (2018), p. 1479984. doi:10.1080/23746149.2018.1479984.
[3] T Saito et al., Adv. Eng. Materials **20** (2018), p. 1800125. doi:10.1002/adem.201800125.
[4] JS Barnard, DN Johnstone and PA Midgley, Ultramicr. **174** (2017), p. 79. doi: 10.1016/j.ultramic.2016.12.018
[5] https://github.com/hyperspy/hyperspy
[6] https://github.com/pyxem/pyxem
[7] JK Sunde et al., Mat. Characterization **142** (2018), p. 458. doi: 10.1016/j.matchar.2018.05.031
[8] E Christiansen et al., Sci. Reports **9** (2019), p. 17446. doi: 10.1038/s41598-019-53772-4
[9] E Thronsen et al., Mat. Characterization **185** (2022), p. 111675. doi: 10.1016/j.matchar.2021.111675
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