s5.m3.o3 **Solving and Refining Crystal Structures Using Electron Diffraction Data.** C.J. Gilmore and A. Stewart *Department of Chemistry, University of Glasgow, Glasgow G12 8QQ, Scotland, UK.*
Keywords: electron diffraction, refinement, maximum entropy.

Electron crystallography is still the Cinderella of the crystallography world: specimen preparation can be difficult; data are hard to collect and have problems with sample thickness, dynamical scattering and sampling; structure solution is problematic, and refinement and structure validation are also non-trivial. Yet there is an ever-increasing body of knowledge in structural science derived from this method, and many samples can only be studied in this way.

In this talk we discuss two complementary problems:

1. Structure solution especially using maximum entropy methods which are well suited for this purpose.
2. Structure refinement using least squares. Unconstrained and unrestrained refinement is not usually successful[1] and we will report on the use of:

- Constraints such as rigid bodies.
- Restraints such that bond lengths are constrained to be equal and constrained also to a chemically sensible value and yet still allowed some freedom. of refinement
- Matrix manipulations using singular value decomposition and eigenvalue filtering. These methods enable the user to determine which parameters are sensitive to the experimental data. Quite often this is not obvious.

The structures come from the literature and are available on a database in cif format.

We acknowledge support from Eastman Kodak, Rochester, USA.

[1] Dorset,D. & Gilmore,C.J. (2000), *Acta Cryst.* **A56,** 62-67.

s5.m3.o4 **ELECTRON POWDER DIFFRACTION – An old topic rediscovered for structure determination of nanocrystalline materials.** T.E. Weirich, *Darmstadt University of Technology,Materials Science Department, Petersenstraße. 23, D-64287 Darmstadt (Germany).*
Keywords: electron crystallography, small molecules, organic materials.

The use of electron diffraction for determining the crystal structure of polycrystalline materials has a long tradition. For example, data from electron powder diffraction (EPD) were already employed in 1933 by V.E. Laskarev & I.D. Usyskin to locate the hydrogen atoms in NH_4Cl. Just a few years later, Z.G. Pinsker and B.K. Vainshtein started first systematic investigations with the aim to turn EPD into an independent method for structure determination [1, 2]. One of the most significant contributions from this group was the development of the *oblique texture technique* which facilitated the determination of dozens of structures from minerals, organic and inorganic compounds [3, 4]. However, in contrast to the oblique texture technique there has been only limited efforts in the past to develop methods for structure determination from non-textured EPD data. Major reasons for this are the well known difficulties for structure determination from powder diffraction data, *e.g.* unit cell determination and extracting sufficiently good structure amplitudes from the overlapping reflections. Caused by these complications only a few (non-cubic) crystal structures have been solved up to now from non-textured EPD data [4].

Nevertheless, the strong interaction between electrons and matter makes EPD extremely attractive as a tool for determining the atomic structure of nanocrystalline powders and thin films. To test the potential of EPD for this purpose an electron diffraction study was carried out for nanocrystalline TiO_2. The investigation proved that Rietveld refinement of EPD data can yield results in good agreement with neutron diffraction data from coarse grained materials. Moreover, the study showed that EPD data (like X-ray powder data) can also employed for solving crystal structures [5]. In spite of these hopeful results further developments will be necessary in this field before EPD on non-textured samples can be considered as a new stand-alone method for determining the atomic structure of nanocrystalline materials. Strategies how to reach this goal together with recent results will be discussed.

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[5] Weirich T.E., Winterer M., Seifried S., Hahn H., Fueß H., "Rietveld analysis of electron powder diffraction data from nanocrystalline anatase, TiO_2. *Ultramicroscopy* 2000 (81), 263-270.