Precession electron diffraction: New instrumentation for TEM electron diffraction structure analysis

Arturo Ponce[1] and Stavros Nicolopoulos[2]

[1]Departamento de Materiales Avanzados. Centro de Investigación en Química Aplicada. Blvd. Enrique Reyna Hermosillo, 140. Saltillo, Coahuila. aponce@ciqa.mx
[2]President NanoMEGAS sprl ,Blvd Edmond Machtens 79 B-1080 Brussels, Belgium.
www.nanomegas.com

Precession electron diffraction (PED)is a new very promising technique that allows collect electron diffraction patterns very close to kinematical condition (like in x-ray diffraction) allowing this way to solve ab-initio crystal structures of nanocrystals. The pattern is collected while the TEM electron beam is precessing on a cone surface; this way, only few reflection are simultaneously excited at the same time, therefore dynamical effects are strongly reduced. Compared to the normal selected area electron diffraction PED has the following advantages:

- Oriented ED patterns can be obtained even if the crystal is not perfectly aligned to a particular zone axis (ZA). We can then collect reliable ED patterns very quickly without need to accurately align the crystals along ZA, which is very useful when dealing with beam sensitive materials (zeolites, pharmaceutical, proteins)
- Geometry of PED patterns contain full information on the crystal symmetry (space and point group) of unknown nanocrystals (no CBED is needed for accurate SG determination).
- Intensities extracted from PED patterns contain reliable integrated intensities in comparison with conventional SAED patterns.
- PED intensities are kinematical, (X-ray like), up to a thickness of 100 nm and they can be used to solve the structure of very small nanocrystals in a semi-automatic way.
- PED technique can be used in older or new TEM (100-400 kV) and is very effective tool to upgrade older instruments to modern electron diffractometry equipments.

The combination of the precession and scanning of the ED pattern reduces the sensitivity of ED intensities to crystal thickness, reduces the effect of Ewald sphere curvature and can also reduce a lot of multiple beam dynamical effects contribution to the reflections (see fig. 1).

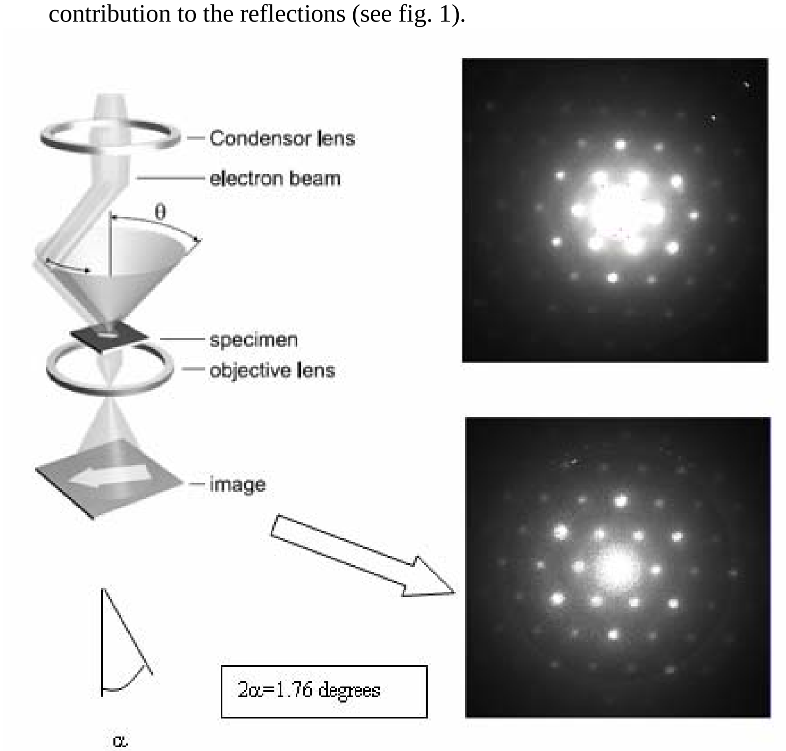

Figure 1. (a)Beam movement in a TEM after applying "conical dark field/precession" mode (b)electron diffraction (ED) pattern of a $Li_xMn_2O_4$ nanocrystal in (111) orientation, with no application and after application of "precession mode": as it can be observed dynamical contribution to all reflections is greatly reduced.

This way all ED reflections are much more kinematical in character e.g. a quality of structure analysis can be greatly improved. as has been shown also in recent publications by several authors[1-4].
The use of precession can also be helpful to observe wider areas of reciprocal space than can be usually observed without precession, or can be helpful to check zone axis orientations (see fig. 2 as example).

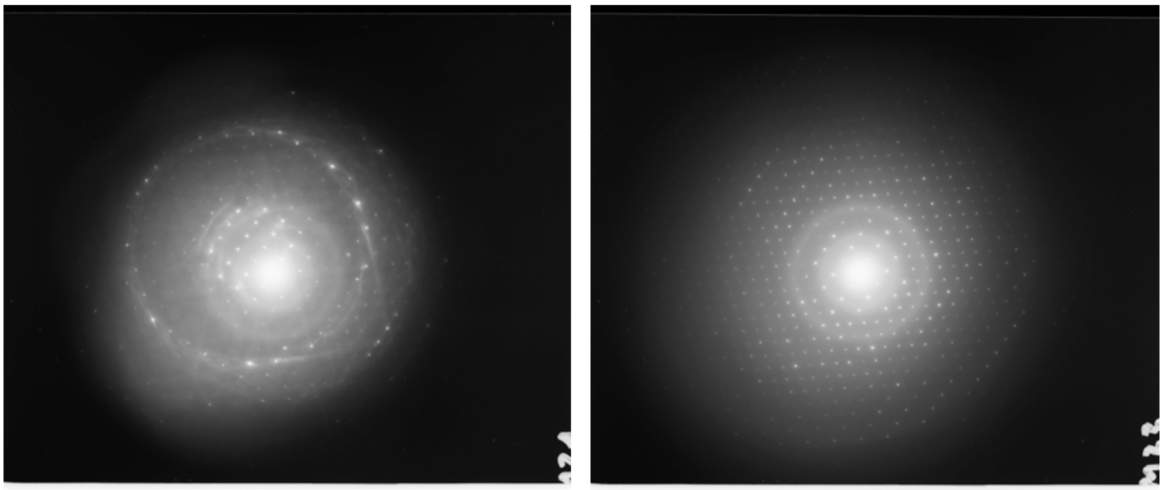

Fig 2. (left)Nd Al3(BO3)4 electron diffraction pattern near zone axis orientation, without precession (right) same orientation with precession

One important aspect in order that precession mode can work satisfactory in the TEM, is that the ED pattern must remain stationary through the precession. In order to do so, scanning and de-scanning of the beam must be exactly compensated.

REFERENCES

1. M.Gemmi Precession electron diffraction in EUROSUMMER SCHOOL: “Electron Crystalloggraphy and cryoelectron microscopy on inorganic materials and organic and biological molecules”, Barcelona (Spain), July 23-27, 2001
2. C.S.Own, A.K Subramanian ,L.D.Marks L. Marks ,Microscopy and Microanalysis 10 , 96-104, (2004)
3. K. Dokko, M. Nishizawa, M. Mohamedi, M. Umeda, L. Uchida, J. Akimoto, Y. Takahashi, Y. Gotoh, S. Mizuta, Electrochem. and Solid State Lett:: “Electrochemical studies of Li-ion extraction and insertion of LiMn2O4 single crystal”
4. J.Gjonnes, V.Hansen , A. Krerneland Microscopy and Microanalysis 10 , 16-20, (2004)