NTNU Noregs teknisk-naturvitskaplege universitet

Yingda Yu, Phone: +47-7359-4863 Fax: +47-7355-0203 E-mail: yingda.yu@material.ntnu.no Tor Arild Nilsen Phone: +47-7359-4085 Fax: +47-7355-0203 E-mail: tor.nilsen@material.ntnu.no

Fakultet for naturvitenskap og teknologi Institutt for Materialteknologi

Gløshaugen

7491 Trondheim Norway

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Memo, the UltraCCD Diffraction and Magnification Precise calibrations

Two typical Gatan DM (DigitalMicrograph) modes were precisely calibrated by using pure aluminium (diffraction calibration) and "fish-bone" nano-fibre (HRTEM calibration) samples for the future easy-using reference.

For diffraction mode, the TEM camera length 30 cm (300 in DM) is chosen for calibration, which gives a good diffraction pattern overview for most of crystalline orientations. SADE (selected area electron diffraction) patterns before (miscalibrated) and after the Diff calibrations by using Al [211] are shown below.



For getting accurate SAED patterns, please try to follow the diffraction alignment procedures as outlined in the **JEM-2010 Handbook Operation**, i.e. in the image mode, to focus the sample by using Z (sample height) control, and at the same time to keep the objective lens at the standard current (at DV +0). Then insert the SA aperture, change to the Diff mode, and sharpening the diffraction spots by adjusting Diff Focus knob.

For recording the correct SAED patterns onto the Ultra CCD, the illuminating beam should be as weak/parallel as possible. In such case, the Diff exposure time could be as longer as possible to avoid the bright tail line on the SAED pattern, (the white tail line caused by beam delaying from the beam electronic shutter (i.e. TEM beam deflector used as the CCD shutter).

For HRTEM mode, the TEM magnification 500 K is precisely calibrated by using the "fishbone" graphite lattice of 0.34 nm. This TEM magnification could be a good start-point for unknown phase HRTEM investigations, and the HRTEM images before (miscalibrated) and after the present HRTEM calibrations are shown below.



While for the well know phase such as shown at <u>http://folk.ntnu.no/yingday/IG/fromOursCCD.jpg</u>, the HTREM image could be directly recorded at higher magnification then calibrated by using the phase lattice distance.

For the low TEM magnification: If really the precise calibration is necessary for a low TEM magnification, it could take a CCD micrograph at 500 Kx first with some fine structures from the sample such as a nano-particle shown at <u>http://folk.ntnu.no/yingday/IG/ABO_particle.jpg</u>. Then low down to the wanted magnification to do calibration by using the same fine structure (e.g. the particle diameter in the above web link). By using such kind method of internal reference, the calibration precision should be much better than that from so called standard sample, since any tiny localised tilting from the TEM holder would result a countable error into the standard sample calibration.

alibration Table	Calibration Table
Microscope Setting	Microscope Setting
Mode: Diffraction	Mode: Imaging 💌
Beam Energy [kV]: 200.0	Beam Energy [KV]: 200.0
Device Name UltraScan 💌	Device Name UltraScan 💌
150->350.026 300->337.341	2000->2773.33 6000->8319.99
🖶 Edit Magnification 🛛 🗙	30000->17928.3 40000->23931.4
Indicated: 300	500000->737684
Actual: 337.3413022	Edit Magnification
	Indicated: 500000
	Actual: 737683.9837
Edit Remove Add	OK Cancel
OK Cancel	OK Cancel

For the future easy using, these two calibrated conditions maybe modified accidentally! In such case, please just enter the DM Calibration Table as shown in the above figure, to check these two mode parameters are correct! If not, for the **Diff** camera length 300 mm, choose edit then key in 337.24, while for the **Imaging** 500 K, in the Edit floating window, key in 737684.

Thanks for the TEM specimens from Manping Liu (Al) and Sten Yngve Larsen ("fish-bone" fibre).