# Design and Verification of an Electron Precession Device

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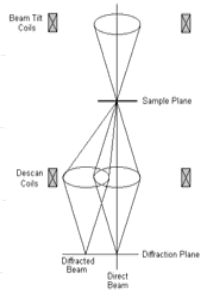
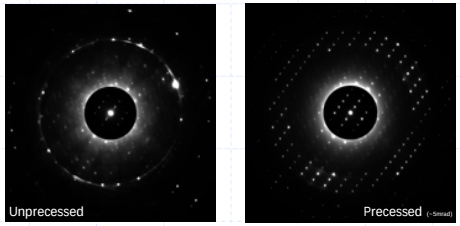



- Vincent-Midgley Precession technique†:
  - A tool for structural crystallography
- Good data requires reduced dynamical error.
- Precession should improve data quality:
  - Avoids exact Bragg condition
  - Lowers probability for dynamical coupling

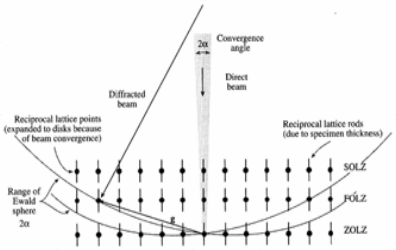
†(Vincent & Midgley. (1994) Ultramicroscopy. **53**, 271.)

## Precession pattern from Hitachi H-9000



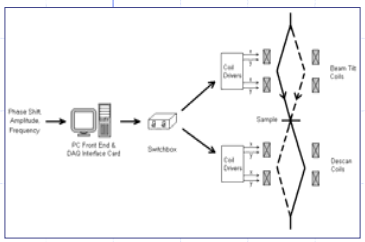

- 532 ZAP of very thick $Mg_3V_2O_8$ crystal.
  - ZOLZ reflections expanded, less diffuse scattering.
  - Kikuchi lines replaced by radial blend.
  - Many more FOLZ reflections excited.
  - HOLZ evident and measurable.

- Theoretically, non-systematic dynamical effects reduced:
  - Momentarily excited at points throughout revolution.
  - Averaged out in the background.
  - Most strong coupling avoided.
- Pattern may be indexed as a ZAP.
- Many more HOLZ reflections illuminated.
  - Expands number of measurable reflections.
  - Can provide 3D data set.



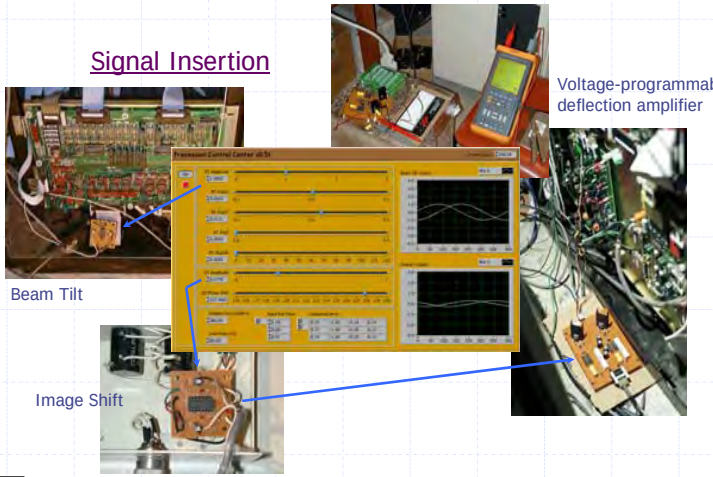
(D.B. Williams and C.B. Barry, (Plenum Press, New York, 1996).)

## The Instrument



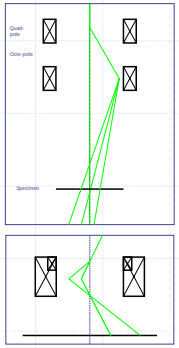

- Uses SA mode:
  - Sharp spots
  - Probed area controllable
  - Low irradiation
- Fast operation to >1KHz.
- Distortion corrections:
  - 3rd order objective pre-field and condenser stigma MUST be compensated for.
  - Software includes 2nd and 3rd-order compensations.
- ***Portable***
  - PC-based front end
  - Ultra-flexible

## Signal Insertion

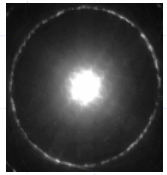



Voltage-programmable deflection amplifier

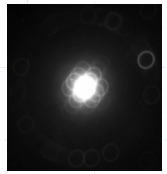
## Beam Deflection Geometry




Beam Tilt



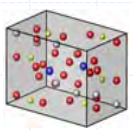
Descan

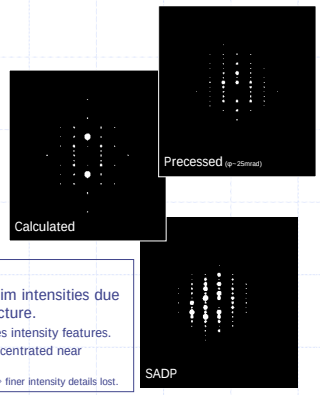


## $La_4Cu_3MoO_{12}$: A Novel Cuprate†

- LCMO a challenge to study.
  - Structure from DP is not the expected structure.
    - Faint reflections (h=odd) missing in experimental DP ⇨ evidence of slightly different phase.
  - Most measurable reflections cannot be used with 2-beam theory.
    - Most LCMO structure factors match large $A_{hkl}$.
    - 2-beam approximation breaks down for large $A_{hkl}$.

a=6.86Å, b=10.98Å, c=7.9147Å
ß=90.02° Monoclinic (barely)






- 001 zone axis.
- Alternating bright/dim intensities due to complicated structure.
  - Precession preserves intensity features.
  - SADP: intensity concentrated near transmitted beam.
    - On-zone coupling ⇨ finer intensity details lost.

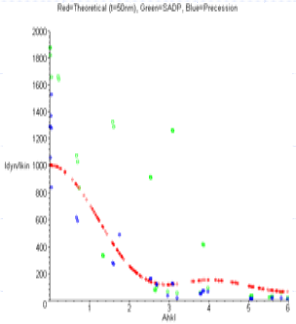
†(Griend, et al. (1999) J. Am. Chem. Soc. **121**, 4787.)

## Does precession really follow the 2-beam approx?

- Vincent & Midgley (1994): 2-beam is theoretically correct for SA precession:†

$$\frac{Idyn}{Ikin} = \sqrt{1-\frac{g}{2R_0}} \cdot \frac{1}{A_{hkl}} \int_0^{A_{hkl}} J_0(2x)dx$$

- Data exhibits blackman-like behavior.
  - Precession correlates with 2-beam much better than SAD.
  - However, this data set does not definitively follow Blackman curve.



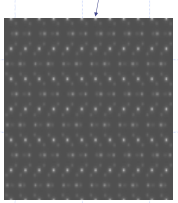
Note: Each point represents average of 4 symmetry-equivalent intensities.

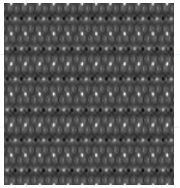
†Gjonnes, K. has provided geometrical corrections for the CBED precession case. ((1997) Ultramicroscopy. **69**, 1.)

## $La_4Cu_3MoO_{12}$ Potential Maps:

Intensity from DP, Phase from known structure

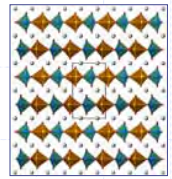
Kinematical amplitude + kinematical phase

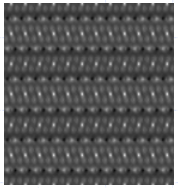




Precession amplitude + kinematical phase

SADP amplitude + kinematical phase





Sharper & more detailed than SADP-derived map.

*Precession intensities yield better definition.*