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Electron Beam Precession System
- Users Manual -

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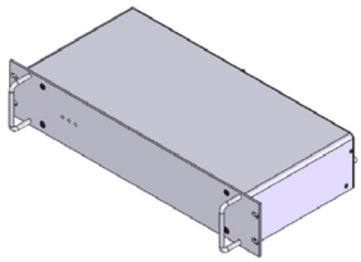

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1. Overview

The Ack! Industries Electron Beam Precession System is used to obtain high quality electron diffraction patterns in a transmission electron microscope (TEM). The system provides a TEM with the appropriate signals to set up the illumination and projection conditions for obtaining Vincent-Midgley precession electron diffraction (PED) patterns.

The PED technique produces electron diffraction pattern intensities that are closer to kinematical than conventional diffraction intensities, giving higher quality data for precision crystallographic studies. PED patterns contain more information about higher order Laue zones (HOLZ) than conventional diffraction patterns, improving crystal symmetry identification. Further, the quasi-kinematical data is potentially free enough of dynamical scattering defects to be used for *ab initio* crystal structure solution.

This manual covers the theory behind formation of the precession probe (section 2-3) and the operating procedures for obtaining precessed diffraction patterns (section 6). The software and hardware are described in 4 and 5. A list of references related to electron beam precession is included at in section 7. This list is by no means exhaustive, but may serve as a starting point for the beginning precession diffractionist to learn more about the technique.

2. Theory of operation

PED is a powerful technique that can improve the quality of electron diffraction data from bulk crystals [1]. The improved data is useful for symmetry determination, and for phase recovery algorithms that can restore the structure from diffraction intensities alone.

In PED, the beam is deflected prior to interaction with the specimen to form a tilted illumination condition. After interaction with the sample, the diffracted beams are deflected using a complementary signal to restore the beam to its original (pre-tilted) location on the viewing screen. Applying this tilt/de-tilt serially around the optic axis forms a hollow cone of illumination at the image plane and a diffraction pattern at the back focal plane as seen in Fig. 1.

The scheme is equivalent to rocking the crystal as is done in the Buerger X-ray precession method. However, the acquired data differs because the small dimensions of the crystal in the TEM give rise to an elongated shape function in reciprocal space (the reciprocal lattice rods, or relrods). As a result, the scattered intensity is modified from the kinematical sinc function depending on the number of simultaneously excited beams and the thickness of the specimen. In reciprocal space, the Ewald sphere is swept through the relrods within a finite range of excitation errors $s_{\mathbf{g}}$ defined by the constraints imposed by precession angle ϕ and incident electron wavelength. The collected intensity is thus an integral of the scattered intensity within that range of excitation errors.

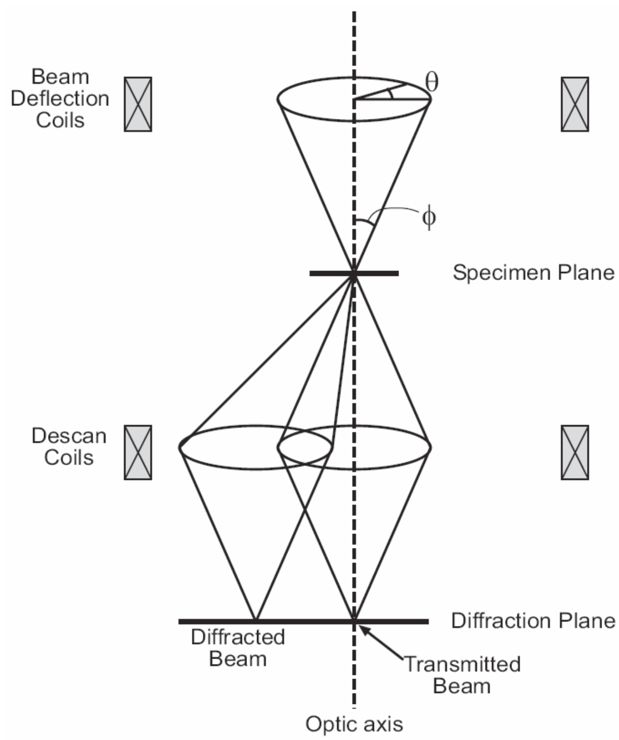


Figure 1. A schematic diagram of precession electron diffraction. The beam is tilted off zone by angle ϕ using the beam tilt coils and serially precessed through an angle $\theta = 2\pi$. A complementary de-tilt is provided below the specimen by descan coils to restore the zone axis pattern.

Fig. 2 shows a snapshot of this integration. In Fig. 2a, the reflection ***g*** is intersected by the Ewald sphere with positive excitation error s_g. The beam may have an angular spread of α, denoted by the solid curves, not exceeding the value for overlapping discs in the diffraction pattern. The dimension $2R_0$ describes the zeroth order Laue zone (ZOLZ), where half that dimension is the radius of the ZOLZ. Reflections in the middle of the 'bowl' are weakly excited, and reflections close to the edges of the bowl where it intersects the *x*-*y* plane are strongly excited. In Fig. 2b, a cross-section of the bowl at the *x*-*y* plane is shown demonstrating how the ZOLZ precesses about the *z*-axis. A complete precession occurs when θ traverses 2π, and the integrated spots represent the incoherent summation of the constituent tilts.

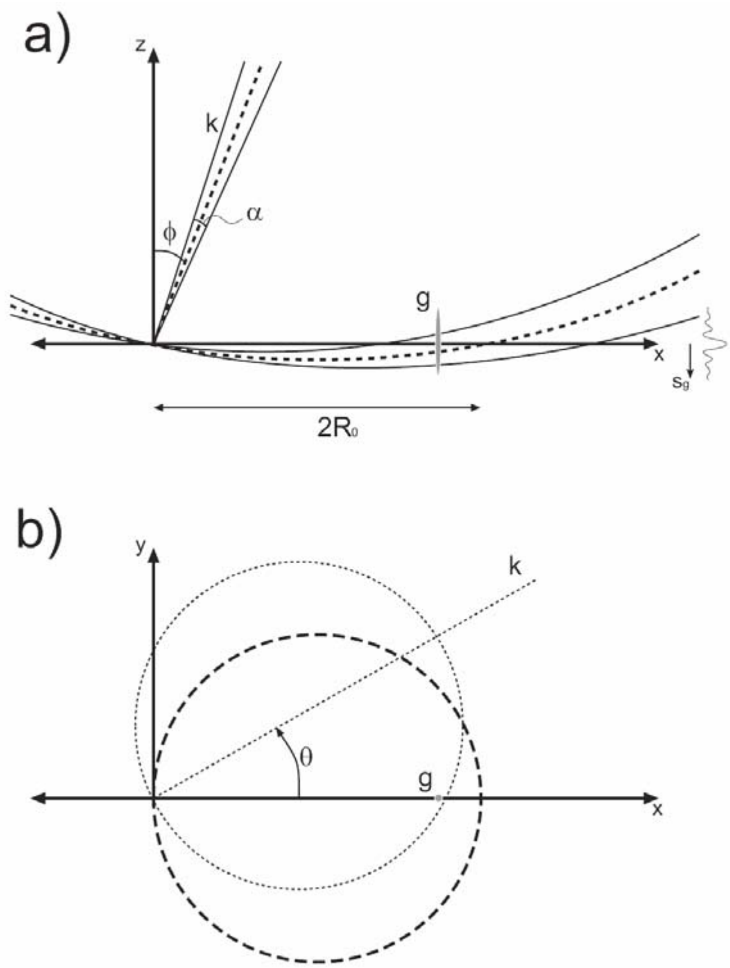
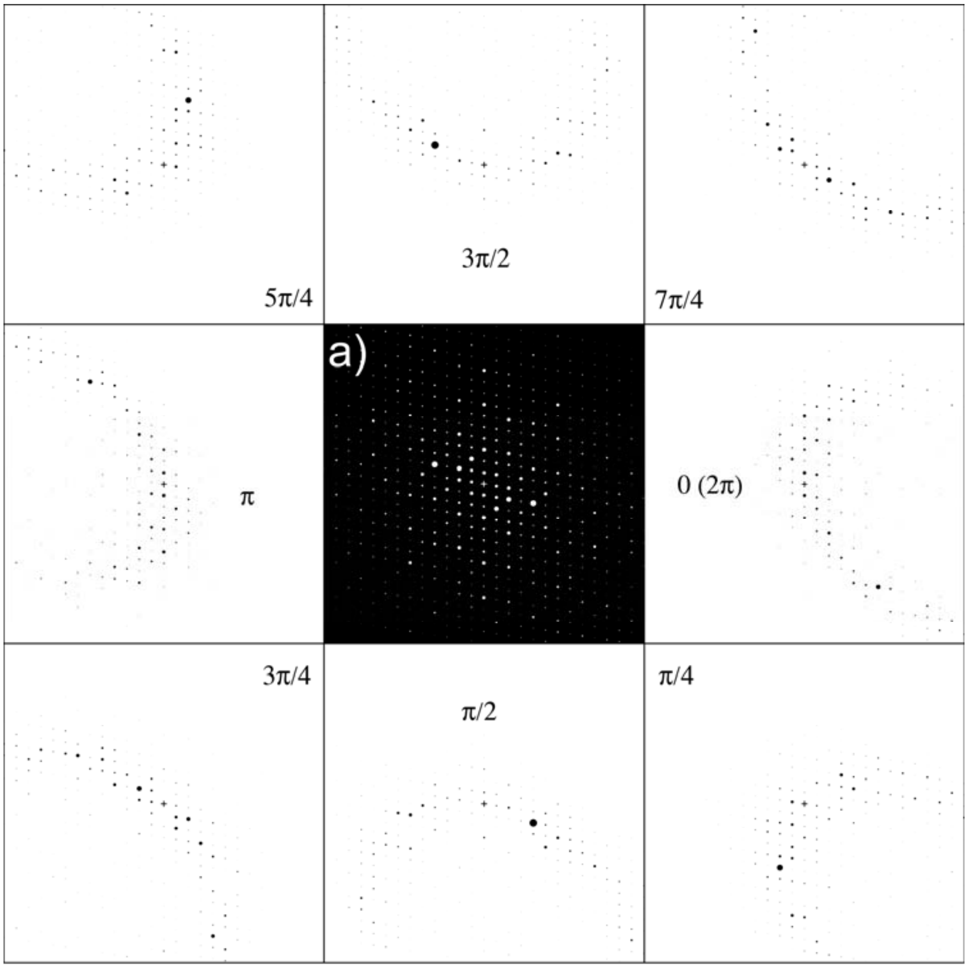


Figure 2. Reciprocal space geometry for precession in (a) *x*-*y* plane and (b) *x*-*z* plane. The beam precesses about the *z*-axis maintaining constant ϕ. In (b), the ZOLZ (bold dashed circle) precesses about the z-axis.

An example of the summation of diffraction intensities from constituent tilts is shown in Fig. 3 for a real crystal. This is simulated data for a 41 nm thick $(Ga,In)_2SnO_4$ M6 crystal phase. In Fig. 3a, a PED pattern with half-angle of 24 mrad comprises all tilts about the central axis, of which eight are shown. A much smaller number of simultaneously-excited reflections are present in each off-zone condition, reducing the dynamical mixing of intensities. Fig. 3b is the unprecessed zone axis pattern for the same crystal, which at this crystal thickness is highly dynamical. When comparing the two patterns, the striking difference in the distribution of intensity between precessed an unprecessed electron diffraction patterns is readily seen.



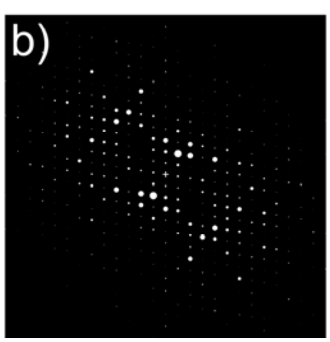


Figure 3. Center precession pattern (a) is an incoherent integration of the simulated tilt series (contrast inverted) that surrounds it. (b) is the non-precessed pattern. Thickness is 41 nm, ϕ = 24 mrad, patterns represent structure factor amplitudes. [001] zone of $(Ga,In)_2SO_4$.

When a large precession angle is used, the first-order Laue zone (FOLZ) will spread from a ring of reflections into an annulus that overlaps the rest of the pattern. Depending on the structure, many FOLZ spots may project directly onto ZOLZ spots, obscuring the true intensity of either. This is demonstrated in Fig. 4, where the FOLZ reflection at point **F** which is scattered at an angle γ may obscure a ZOLZ reflection when it is precessed by π radians to the opposite point **F'**. This type of overlap is especially likely for structures with large unit cell dimension along the beam direction. It may be necessary to reduce the precession angle in order to avoid obscuring important ZOLZ spots that might otherwise be overlapped by the FOLZ.

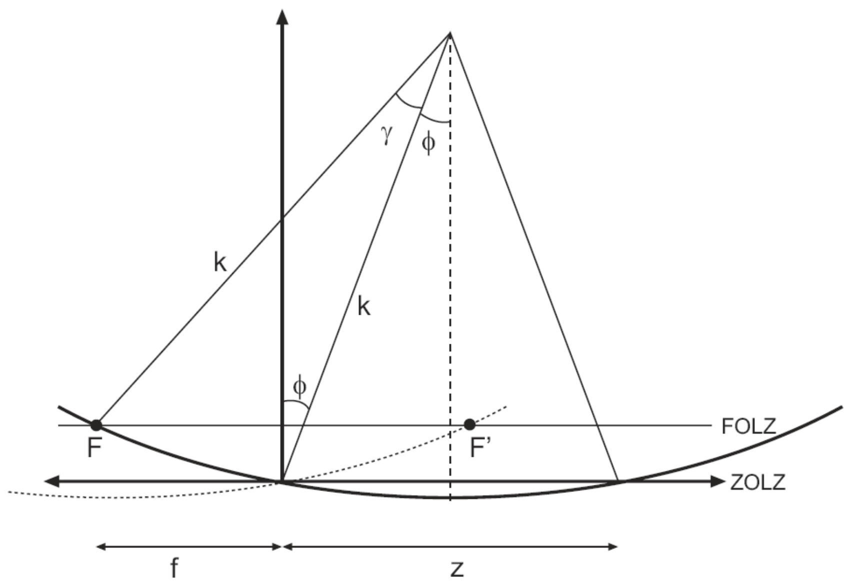


Figure 4. Schematic of precession geometry showing the relationship between ZOLZ and FOLZ excitations. The distance *z* corresponds to the zeroth-order zone radius; *f* corresponds to the usable diffraction radius, subtended by angle γ.

Some of the key features of electron beam precession are summarized below:

- The pattern may be indexed as a conventional diffraction pattern while the intensities have actually been obtained from off-zone reflection conditions.
- Non-systematic dynamical effects such as Kikuchi lines and intensity variations in CBED spots are reduced by averaging over incident beam directions.
- Since the beam is entering the sample from an off-axis direction, much of the dynamical scattering that is particularly strong at the exact Bragg condition (or zone axis channeling condition) is avoided. Typically only one strong beam is simultaneously excited with the transmitted beam (two-beam condition). In some cases three beams may be strongly excited, but in most cases no clear systematic dynamical path exists so the interaction between beams is not strong.

- Many more ZOLZ reflections are excited, under more kinematical conditions, by the Ewald sphere allowing acquisition of an increased number of intensities for use in symmetry identification and structure solution techniques.
- HOLZ reflections are excited, yielding data sets expanded into the third dimension provided that spots from separate Laue zones do not overlap.
- Studies have shown that dynamical mixing is dramatically reduced for most crystals above about 20 mrad half-angle. Improvements to the data continue for higher angles, but yield diminishing returns up until the point at which optical aberrations limit probe localization on the sample.

For more information on PED and guidelines for how to interpret the intensities from PED patterns, please refer to the thesis of C.S. Own and the references provided in section 7 of this document.

3. Electron-optical Considerations

In the usual configuration for PED, the probe is scanned and descanned symmetrically about the sample plane located at the eucentric height (defined by the optimum current setting of the objective lens). This geometry is shown in Fig. 5, which traces the path of the transmitted beam through the objective lens area of the TEM. The scan is purified such that the scanned probe is rocked about the sample plane in a symmetric fashion about the zone axis. The descan is purified such that the precessed diffraction pattern passes through the imaging/projection system on axis. In a well-aligned system, the beam passes through each lens element in the column close to its central axis in order to avoid aberrations. The beam deviates from the central axis only in the objective lens and in the scan coil layers that are directly adjacent.

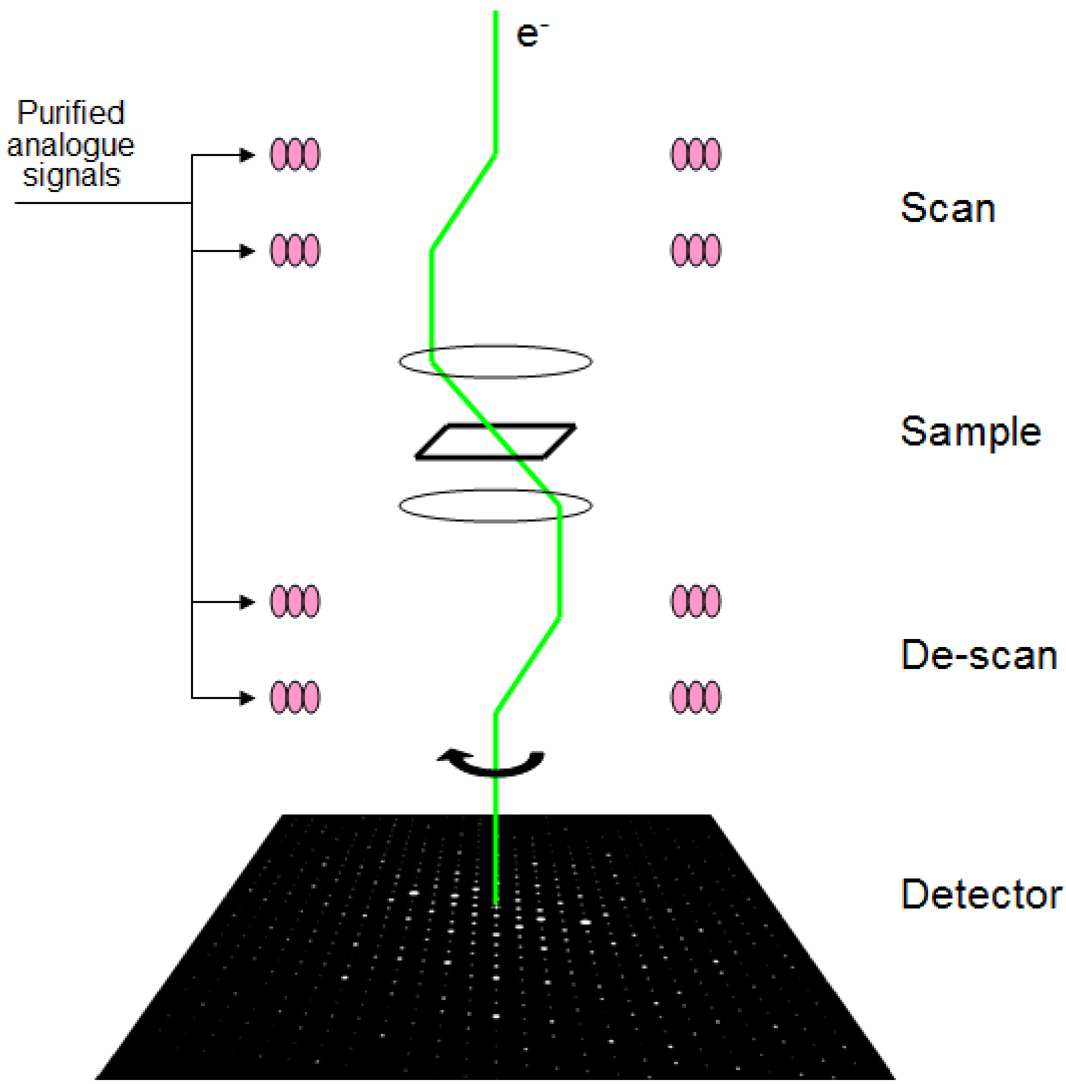


Figure 5. The configuration of the objective lens and deflectors in precession. Ray path for one tilt is shown demonstrating the symmetric scan/descan geometry.

Dynamical diffraction is minimized when the precession has a large cone half-angle. At the same time, an illuminating probe of minimal dimensions is desired on the sample. Because of aberrations in the optical system, these two are competing factors. Fortunately, since large half-angles are typically used in PED – much larger than the crystal mistilt – and all reciprocal lattice rods are typically sufficiently sampled in PED, minor unevenness of the hollow cone can be readily tolerated (up to a few percent error). On the other hand, too broad a probe in image space may illuminate incorrect regions of a multi-phase material and contaminate the diffraction pattern with spots scattered from the wrong phase. Therefore the probe localization is a more stringent constraint than tilt accuracy. To achieve a fine probe, tilt purity must be carefully calibrated to provide a precise tilting center in the sample plane. This is the primary challenge when aligning the electron beam precession system. Indeed, precession electron diffraction is aberration correction by another name.

Fig. 6 demonstrates graphically the interplay between the aberrations and the scanning system. In the theory of image formation, it is known that as the aberration function $\chi(\phi)$ changes with angle ϕ, the phase shift imparted by the aberration function causes beams of higher angle to deviate from the Gaussian focal point at the sample plane, causing a diffraction-limited convergent probe to blur and/or change shape at the sample. In electron beam precession, the beam is not convergent but ranges from near-parallel to mildly convergent (e.g., non-overlapping discs in the diffraction pattern) and thus for a given tilt illuminates the objective lens with only a thin pencil of illumination – a small angular range $\Delta\phi$ – away from the optic axis. When precessed, this thin pencil then becomes a thin annulus of illumination.

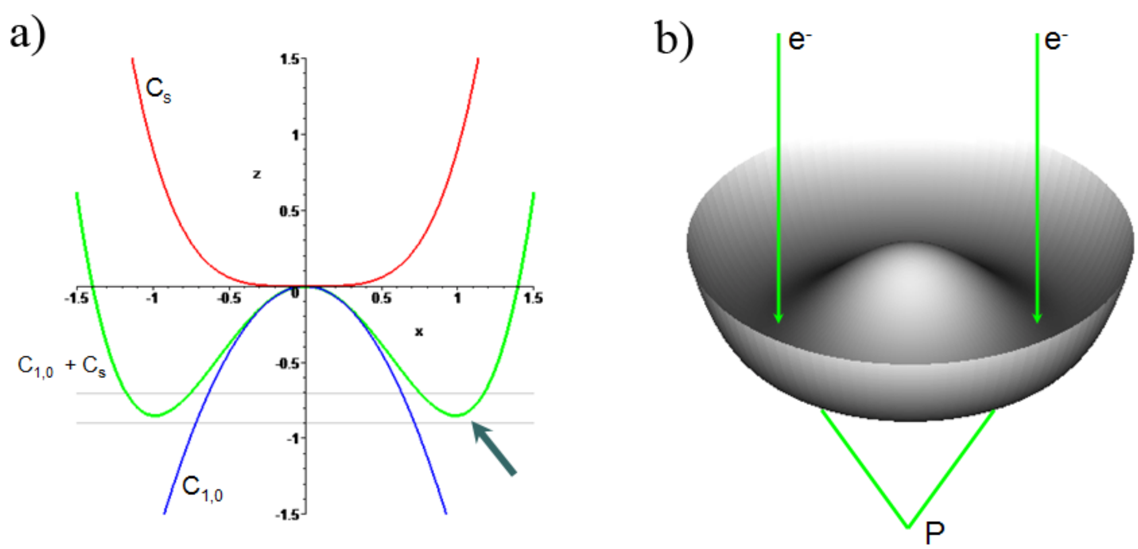


Figure 6. (a) Two-dimensional sections of aberration functions for spherical aberration (C_s), defocus ($C_{1,0}$), and their combination. The grey lines mark a region of constant phase. (b) The combination aberration in three-dimensions showing how two separate pencils of illumination entering the objective lens are focused to a point P at the Gaussian focal point.

In Fig. 6a, a two-dimensional section of the aberration functions for C_s (spherical aberration) and $C_{1,0}$ (defocus) is shown. In PED these aberrations are balanced against each other to create a combined aberration function that forms an annulus of flat phase as pointed out by the arrow in the figure. It is analogous to the Scherzer defocus known in high resolution imaging, except it is an extreme version that compromises the flat phase in the center of the lens in order to achieve high angles at the sample. Because the center region is not filled with electrons, central rays that would otherwise deviate from the Gaussian focal point are not present and do not blur the probe.

To illustrate this point further, in Fig. 6b thin pencils of illumination are shown entering the combined aberration function (now shown in 3-D) at opposite regions of the flat phase annulus. Because the phase shift is constant around the annulus, both rays are focused equally strongly by the objective lens, therefore both reach the sample at the same point **P** in the sample plane and have the same half-angle. Also, astigmatism and coma are naturally introduced because of the curvature of the aberration function. They cause the beam to become slightly elliptical and egg-shaped as it precesses around the optic axis. The direction of astigmatism and astigmatism is aligned with the tilt direction, and rotates as the beam precesses.

It should now be apparent that changing the height of the rocking point is equivalent to changing defocus. In practice, it is preferable to set the tilt purity so that rocking point is in eucentric plane, thereby setting the condition where objective lens aberrations will be lowest.   Refer to the TEM manual for the lens current for focusing the objective lens at the eucentric plane.

Since no physical electron optical element is perfect, the aberration function contains several types of aberrations that influence the quality of the hollow cone with increasing severity. If the aberration is not cylindrically symmetric, the precessing beam will no longer be focused to the Gaussian focal point P and will instead wander around the sample as it is precessed. Additionally, the scanning system imperfections must also be considered. With the large tilts inherent in precession, the electron beam passes far from the central axis of the scanning coils directly adjacent to the objective lens. Depending on physical construction, many dipole deflectors generate considerable sextupole moment in this condition, causing the conical scan to have a three-fold component.

The following table summarizes the aberrations that affect precession, in order of increasing relative severity. As the alignment converges, aberrations lower down become prominent.

Aberration	**Cause**	**Effect**
Defocus	Sample not at eucentric height or scan rocking point incorrect	Circular probe on sample
Spherical aberration	Scan rocking point incorrect	Circular probe on sample
Astigmatism	Tilted illumination	Elliptical shape of probe
Coma	Tilted illumination	Egg shape of probe
Scanning astigmatism	Difference in deflector strength along orthogonal axes	Elliptical scan circle
Scanning three-fold astigmatism	Sextupole moment from dipole misprojection	Three-lobed scan circle
Higher order effects	Interplay between lens and scanning aberrations	Squiggly probe shape
Temporal aberrations	Power supply instabilities and 60 Hz	Pulsating or shaking probe, sometimes frequency dependent

The dark field ronchigram, or tilt-scanned dark field image, best illustrates the combined effect of the aberrations in the table above (Fig. 7). The sample is a combined test specimen with ~5 nm diameter gold balls interspersed on a holey carbon grid. In Fig. 7, the central patch has a large defocus, as does the region outside the annulus of flat phase. As angle increases, the central patch transitions to an annulus of infinite radial magnification, then to an annulus of infinite azimuthal magnification. A small amount of scanning astigmatism (ellipticity of the annulus) and three-fold is present.

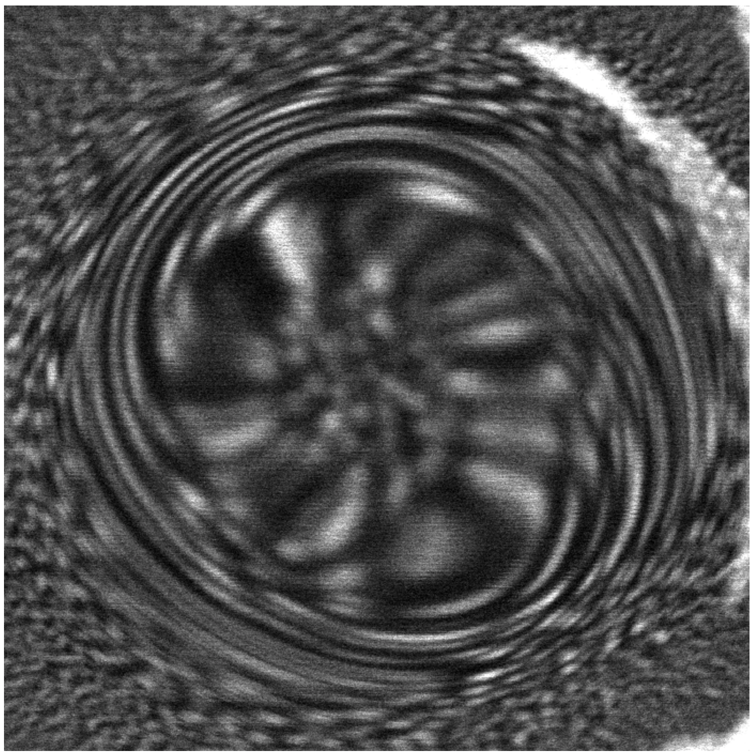


Fig. 7. Dark field ronchigram taken on a Nion SuperSTEM in uncorrected mode. with an objective lens with C_s of 1.2 mm, and the annulus of infinite magnification is about 40 mrad in this case.

4. Hardware

4.1. Signal generation and flow

Fig. 8 shows the flow of the electrical signals in the precession system. The heart of the system is a 16-bit signal generator that provides voltage waveforms that are distributed to the deflector coils inside the TEM. Parameters entered in the software interface are used to calculate the waveforms. Once generated, they are sent to the distribution hardware, buffered, and sent down differential data lines to receiver boards located close to the TEM electronics. The receiver boards condition and buffer the signals and they are finally sent to the TEM's deflector driver circuitry for controlling the electron beam. Multiple levels of voltage regulation, differential signal lines, and careful grounding are used to avoid introduction of external noise to the TEM.

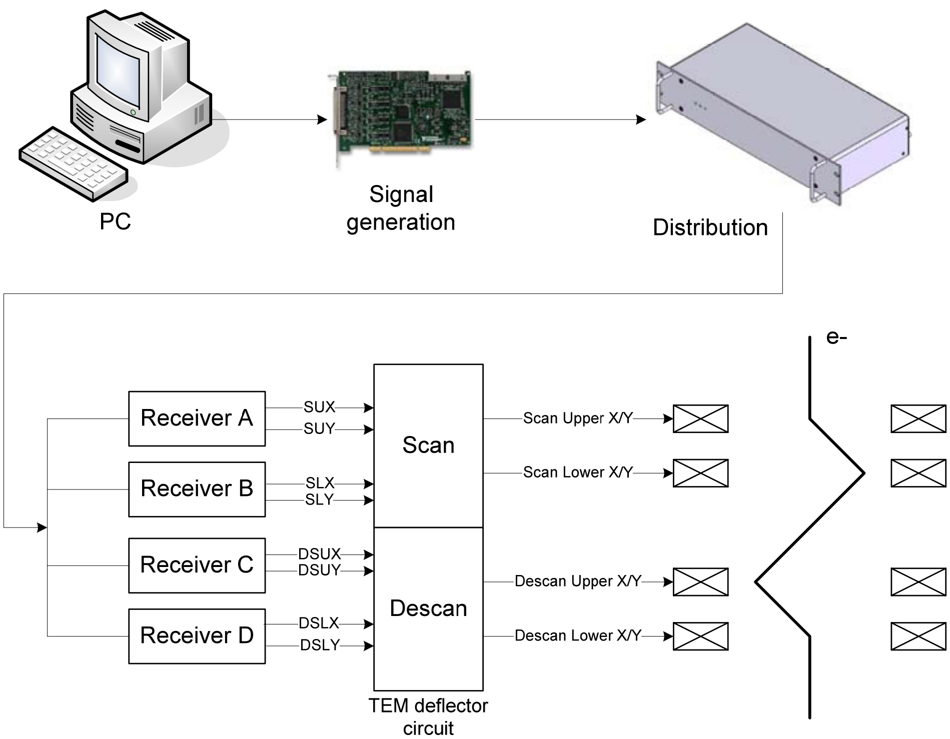


Figure 8. Routing of the electrical signals in the precession system.

The following table shows the designations for the eight available analog output channels and how they are distributed to the column deflectors. The convention is for the electron source to be at the top of the column and the detector(s) at the bottom.

Layer	X	Y
Scan Upper	SUX	SUY
Scan Lower	SLX	SLY
Descan Upper	DSUX	DSUY
Descan Lower	DSUY	DSUX

Relays controlled by software disconnect the precession system from the TEM when it is not in use. This aids in troubleshooting and separates precession from the native functions of the microscope. Quick disconnects are included at the output of the relay boards to completely detach the precession system from the TEM deflector circuits. The output signal has a hardware limit that saturates the signal at ±5.1 V.

4.2. Physical Connections

The precession unit back panel has the following connections:

Connection	Description
120VAC	Mains power input. Receives IEC 15A socket.
Chassis GND	The ground connection for the chassis, shorted to mains ground. If unit is plugged into an outlet with lifted ground, use this terminal to ground the chassis.
Scope GND	The ground connection to the microscope column. This connection must be made in order for the power supplies to regulate.
Data/Control:	Connection with the PC via 68-pin HD connector.
AO_A…AO_D	Signal outputs for each layer. Two analog channels, power, and relay control signals are included in each. .
UPD*	BNC output signal that can be configured for either scan start/stop or next tilt. 50Ω impedance.
EXT SYN	BNC input signal that can be configured for triggering either scan start/stop or next tilt. 50Ω impedance.

The front panel has the following indicators:

Blue: Digital power (always on when connected to PC)
Green: Main power
Amber: Data out update

5. Software

The precession software is a Microsoft .Net program executable that resides in its own directory: C:\Program Files\Precession. The program is started by clicking on the shortcut in the start menu or on the desktop. The software is also available as a Gatan DigitalMicrograph plugin. Plugin functionality including scripting functions is covered in section 5.3.

All data files are stored in the precession folder. The data formats are .ppf and .cal, corresponding to profiles and calibrations.

5.1. Main Interface

The main interface window has controls for starting and stopping precession, controlling the scan and descan, and accessing the setup window for configuring precession parameters. A status bar indicates the state of the system.

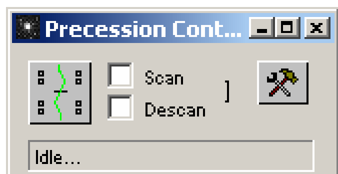
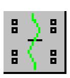


Figure 9. Main Interface

 Precession button – starts and stops precession waveform generation.

 Tools button – launches the Precession Setup window.

The scan and descan buttons are used to select whether waveforms are generated for the scan and descan, respectively.

5.2. Precession Setup

The setup window is used to configure precession parameters. A set of configured parameters is called a precession profile. The program automatically loads the default profile from file at startup, and saves the current profile as the default when it is closed. Profiles can be saved and loaded manually using the menu items in the “File” menu. The setup window contains four tabs: Prec Setup, Dipole Alignments, Waveforms, and AO Setup. Their contents are described in the following sections.

The Tools menuitems allow access to the hardware diagnostics panel (see section 8.2). The menuitem “Bridge PC/Scope Grounds” is used to bridge the TEM and PC grounds inside the distribution unit, which can improve noise performance of the precession system. This menuitem should be checked if the PC is on a lifted ground and is not directly connected to the microscope ground with a stiff connection.

Any numeric control is easily adjusted by either entering a number in the field and pressing enter, or double-clicking it to access the scrolling display. The scrolling display (Fig. 10) allows the user to efficiently change a numeric value by moving the mouse horizontally. It displays the last value of the control, the desired new control value, and the decimal place affected by the mouse motion. The up/down arrow keys or the mouse scroll wheel change the strength. Pressing esc cancels and restores the last value. Pressing enter or clicking the left mouse button stops the scrolling display and saves the new value to the numeric control.

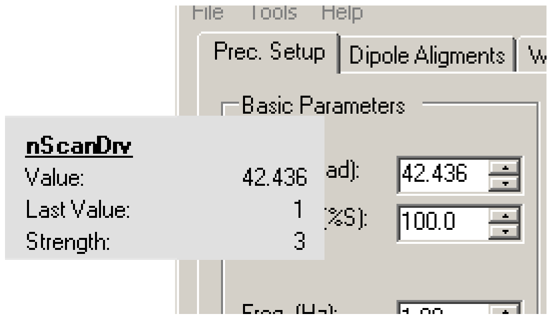


Figure 10. Numeric scrolling display.

5.2.1. Prec Setup Tab

Basic parameters for setting up the precession and tuning the aberration components are located in the Prec. Setup tab.

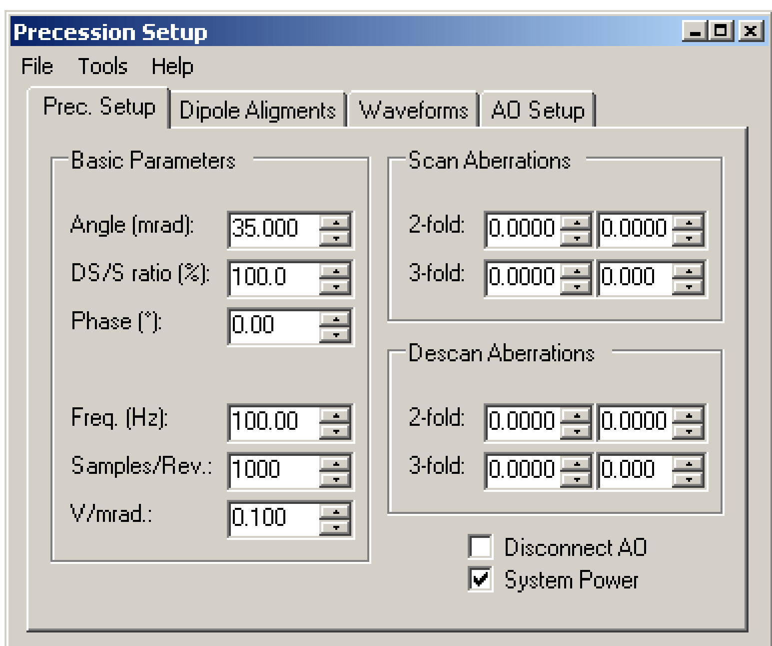


Figure 11. Prec. Setup tab in the Precession Setup window.

Control	Description
Basic parameters	
Angle (mrad)	Amplitude of the scan signal. This determines the precession semi-angle.
DS/S ratio (%)	Amplitude of the descan signal in percentage of scan.
Scan/Descan phase shift (°)	The phase difference between the scan and descan in degrees.
Freq (Hz)	Scan frequency (precession revolutions per second).
Samples/Rev	Number of discrete tilts per revolution. For large precession angles, a larger number of tilts is desired in order to sufficiently sample the relrods. Typically, 1000 tilts is sufficient.
V/mrad	The factor for calibrating the half-angle in mrad units.
Scan Aberrations	
2-fold	X and Y components of the two-fold aberration compensation.
3-fold	Amplitude and phase components of the three-fold aberration compensation.
Descan Aberrations	Same as above, except for descan.
System Power	Turns off power to the unit. This also disconnects the analog outputs.
Disconnect AO	Signals the relays on the differential receiver boards to disconnect the precession unit from the microscope.

5.2.2. Dipole Alignments Tab

The Dipole Alignments tab contains the purity controls for scan and descan and provides wobblers for checking the strengths and directions of the dipole layers. In the alignments section of this document, the dipole strength components will have the following nomenclature matching the layout in this interface (example is for scan-upper):

SUX	SUYx
SUXy	SUY

For the cross terms, the major acting axis is capitalized and the signal from the orthogonal axis that is cross-fed into the major axis is lower case.

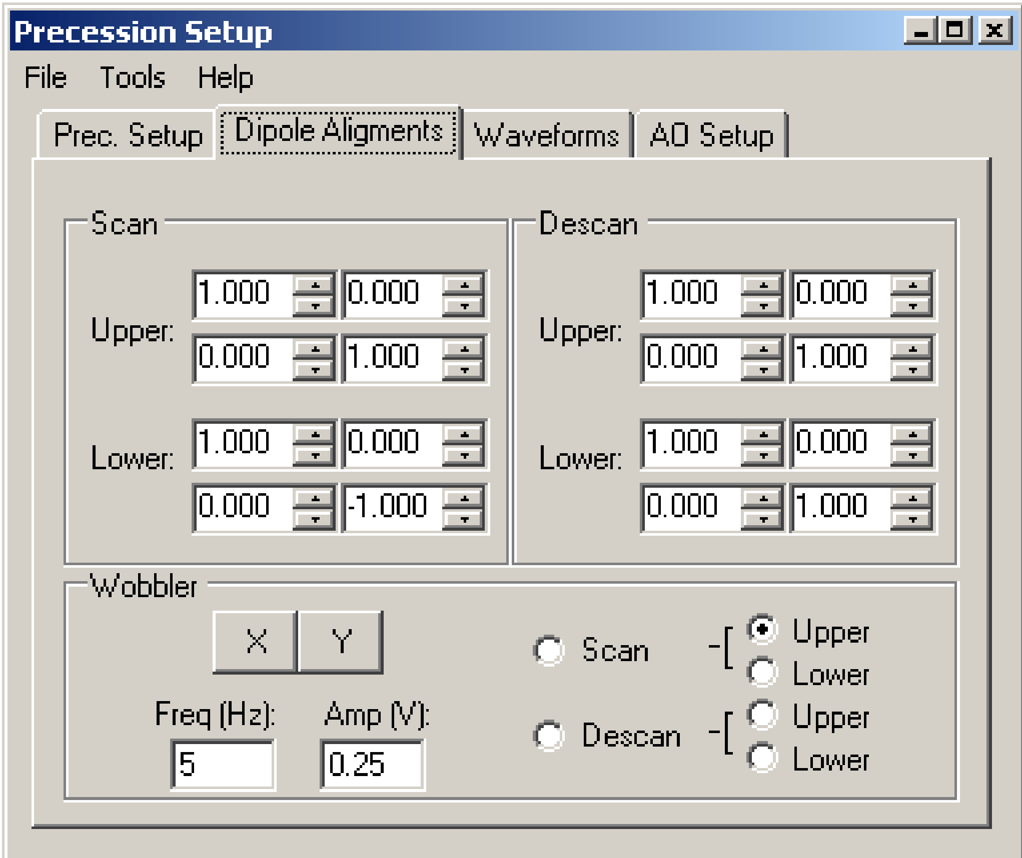


Figure 12. Dipole Alignments tab in the Precession Setup window.

Control	Description
Scan group	Dipole strengths are set using these controls. The top left and bottom right controls are the major axes (X and Y, respectively). The top right and bottom left controls are the amounts of X signal cross-fed into Y, and Y signal cross-fed into X, respectively.
Descan group	Amplitude of the descan signal in percentage of scan units.
Wobbler	Click X or Y buttons to start the wobbler of the nominal axis direction for a selected layer. Click the Scan or Descan radio buttons to select combined layers. Frequency and amplitude controls are provided.

5.2.3. Waveforms Tab

The Waveforms tab can be used to examine the numeric output of the signal generator or to bypass the signal generator with custom waveforms. Specialized non-conical scans such as square or spiral scans can be entered with this feature. The file formatting is tab-delimited.

Reference waveforms can be created by generating a waveform internally, starting and stopping precession in the main interface, and saving the waveform to file.

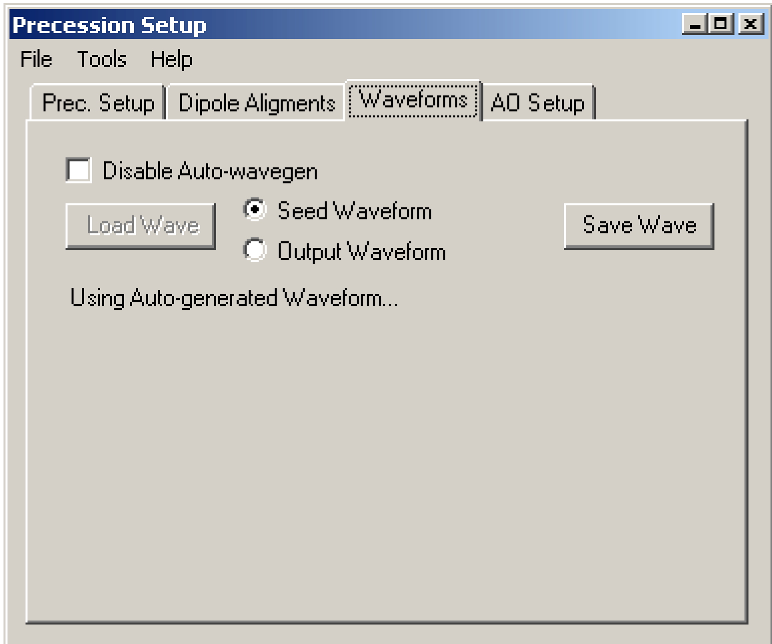


Figure 13. Waveforms tab in the Precession Setup window.

Control	Description
Disable Auto-wavegen	Disables the internal waveform generator. When the 'Start Precession' button is clicked on the main panel, the software will use the loaded waveform instead of the one generated from the precession parameters.
Load/Save Wave	These buttons load/save waveforms from/to file. The type of waveform is specified using the Seed Waveform and Output Waveform radio buttons.
Seed Waveform	When selected, the seed waveform type is used. This waveform type represents the X-Y source scan before the signals are mixed according to the tilt purification parameters. When selected, the software will use the loaded seed waveform to generate an output waveform according to the tilt purities in the Dipole Alignments tab. The Angle and Scan/Descan Aberrations controls in the Prec. Setup tab are bypassed.
Output Waveform	When selected, the output waveform type is used. This type represents the signals that are directly outputted by the signal generator, in volts. All parameters in the Prec. Setup and Dipole Alignments tabs are bypassed.

5.2.4. AO Setup Tab

The low level parameters of signal routing, voltage offsets, and software saturation limits are accessed in the AO Setup Tab.

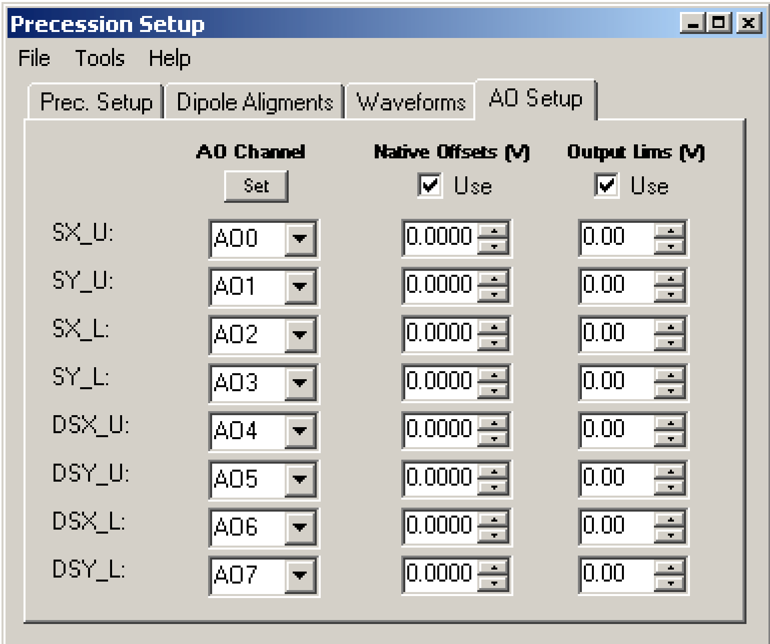


Figure 14. Prec. Setup tab in the Precession Setup window.

Control	Description
AO Channel	Changes the signal routing for the channels. Click “Set” to save the displayed configuration.
Native Offsets (V)	The voltage amplifiers in the system can accumulate a small error in the final output signal sent to the TEM deflector circuit. These controls apply a permanent offset signal to cancel the cumulative error so that the beam does not shift when the precession system is connected to the TEM. “Use” must be checked in order for the offsets to take effect. Entered values are saved regardless of whether the offsets are used.
Output Lims (V)	These controls impose a software limit on the voltage outputs to prevent overdriving the microscope circuits. The waveform will saturate at ± the value for each channel. “Use” must be checked in order for the limits to take effect. Entered values are saved regardless of whether the limits are used. The hardware limit in most installations is ±5.1 V.

5.3 DigitalMicrograph Plugin Functionality

The DigitalMicrograph version of the precession software is a .dll file that resides in the <program path>\DigitalMicrograph\Plugins folder. The precession interface is started from the Precession menu in DigitalMicrograph. Profile data is stored in the DigitalMicrograph root folder.

The following script functions are available:

Function	Description
Prec_OpenIface()	Start precession interface from script function call.
Prec_Start()	Start precession with currently loaded profile.
Prec_Stop()	Stop precession.
Prec_LoadProfile(string name)	Loads profile "name" from file. Profile files name.ppf and name.cal must be present in the DigitalMicrograph root directory.

6. Operating Procedures

The precession system is best aligned using a fast scanning CCD camera with anti-blooming feature in conjunction with software that can mark features on the screen (such as Gatan DigitalMicrograph). When taking images, use an exposure time that is an integer multiple of 1/Freq. This will prevent taking exposures containing incomplete precession cycles.

Rough alignment (section 6.2) and scan angle calibration (section 6.3) must be completed prior to using the system for crystal studies. Refer to section 5.2.2 for the nomenclature for the dipole strength controls used in this section.

During alignment, the TEM will be switched between image and diffraction modes and a variety of magnifications and camera lengths may be used to monitor the effects of each alignment (typically 40kx-120kx mag and 1cm-1m CL). Note that some microscopes may change lens modes as these parameters are adjusted, so it is important to check that the effect of controls is consistent between TEM adjustments during precession alignment.

6.1. Precession Alignment

Begin with a fully aligned column in diffraction mode with precession off. The beam should be voltage- and current-centered through the objective lens and appear round on the sample in image (zoom) mode. The projection system should display round spots in the back focal (diffraction) plane. The beam current should be low enough so that the transmitted beam does not bloom substantially on the detector.

Typically, the smallest condenser aperture is used to prevent saturation of the CCD and provide a small spot size on the sample. Further, the beam may be converged to increase the angular spread of each diffracted spot and reduce probe size, to the limit of overlapping diffracted spots. If the acquired data will be used quantitatively, take note of the convergence angle because it affects the integration of the relrods in numerical calculations.

Set the projection system for a short camera length (~1 cm) to allow for large deflections of the beam during this alignment. The sample should be at eucentric height.

1. Align sample to within +/- 1 mrad of the zone axis.

2. Move to an area of the sample adjacent to the region of interest (ROI) to avoid beam damage. This area should be at the same sample height as the ROI.

3. Set up the basic parameters. Typical starting parameters are:
    a. Angle = 35 mrad
    b. DS/S ratio = 75%
    c. Freq. = 50 Hz
    d. Samples/Rev. = 1000

4. Start precession with Scan on and Descan off. The diffraction pattern will become a set of circles, each corresponding to a diffracted spot. The brightest circle is the precessed transmitted beam.

5. Check that the pattern is circular. It is helpful to use a reference circle as shown in fig. 15a to check for roundness. If the circle is considerably out of round (>5% difference between dimensions), the scanning purity must be corrected (see section 6.2). If the scan circle is close to round, use the Scan Aberrations controls in the Prec. Setup tab to make the beam as round as possible.

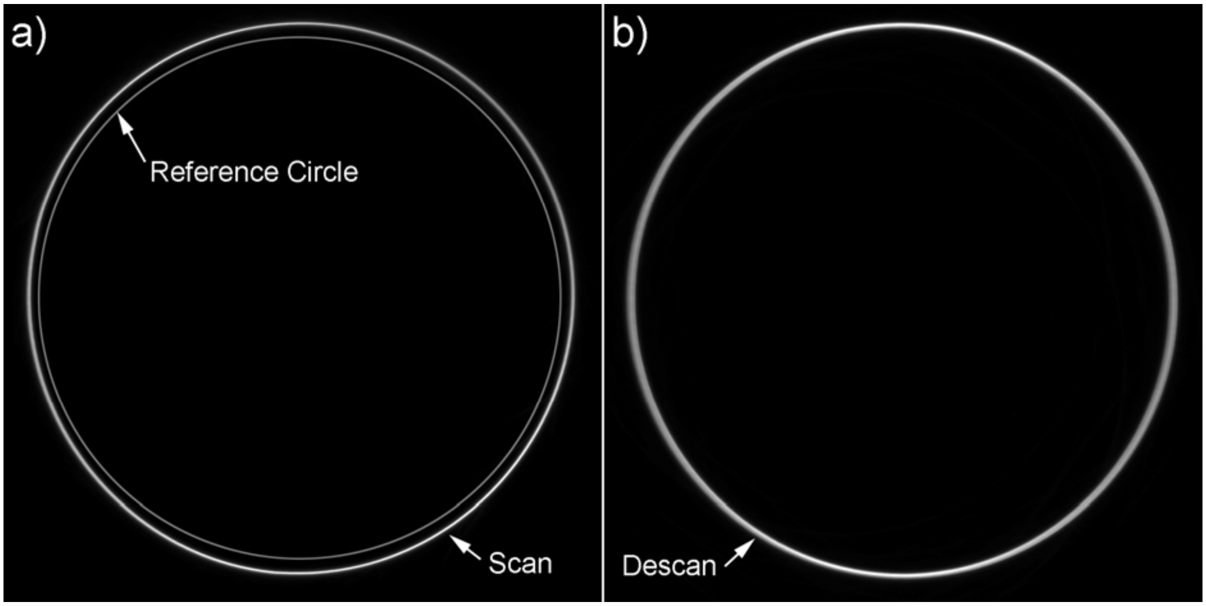


Figure 15. a) Scanned transmitted beam. The scanned beam has been aligned using the Scan Aberrations controls to trace a round path. b) Descanned transmitted beam aligned to match the scan exactly in amplitude, roundness, and antiphase. When combined, the scan and descan collapse the beam to a point.

6. Increase Angle to the desired value. Optionally, if a sample with known unit cell dimensions is used, it is worth checking that the radius of the circle matches the desired half-angle as measured against known diffraction spacings (see section 6.3).

7. Switch to image mode and converge the beam. The beam will be tracing a circle (possibly distorted) on the sample. Use the Scan Lower parameters in the Dipole Alignments tab to make the probe as small as possible on the sample. If the TEM is well-aligned and rough precession alignment is good, it should be possible to obtain a probe under 100 nm with careful alignment.

8. Iterate steps 5-7 until the probe is as small as possible. When the probe wandering is minimized, spread the illumination to obtain the desired probe size and diffraction spot size.

9. Switch to diffraction mode, with scan off and descan on. Using the reference circle from step 5 as a guide, increase DS/S Ratio so that the descan circle matches the size of the scan circle. Tune the descan in a similar way to how scan was tuned in steps 5-7 (Fig. 15b).

Note: In the next steps, the alignment can be made easier if the diffraction spots are temporarily sharpened by using a less convergent beam. When the alignment is complete, return to the conditions of step 8 (image mode, scan on, descan off) to restore the illumination to the desired probe size and diffraction spot size.

10. In diffraction mode, turn on both scan and descan. The composite circle will likely be larger than the scan or descan circles.
    a. Adjust the Phase control until the circle size is minimized. Avoid crossing over (collapsing the circle until it inverts and gets larger).
    b. Adjust DS/S Ratio to further minimize circle size, as in the previous step.
    c. Iterate until the diffraction pattern collapses to as small a point as possible.

11. At this point, the spots are likely fine squiggles of varying shape. Switch to larger camera length (0.5m-1m) and observe the low order spots. Adjust the Descan Lower controls in the Dipole Alignments tab so that the precessed spots are as sharp as possible (see Fig. 16 for an example).

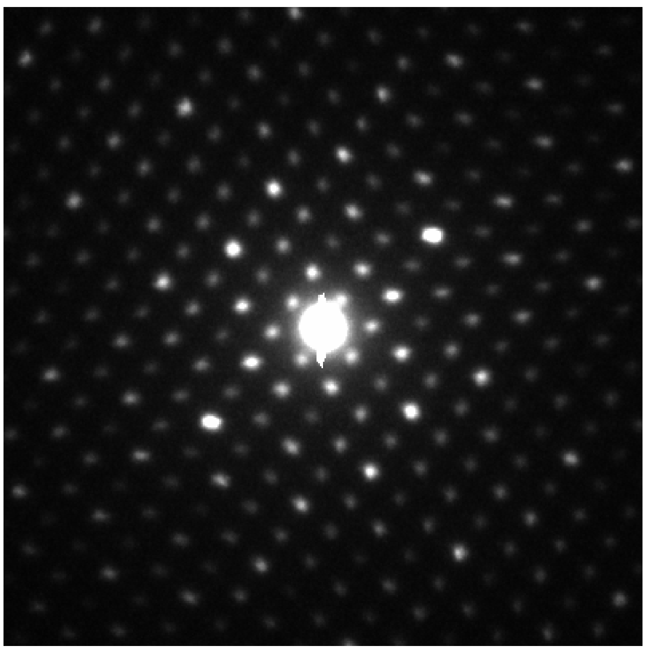


Figure 16. Precession diffraction pattern from $(Ga,In)_2SO_4$ [001].

12. Switch to image mode and turn off descan. Move the sample to the ROI. If the beam was made more convergent, restore the beam to the desired probe size and diffraction spot. If the probe is larger at the ROI, adjust the sample height to restore the probe size on the sample.

13. Switch to diffraction mode and reactivate descan. Take exposure.

14. Optional: Turn off descan and take exposure of the scanned diffraction pattern to save a reference of the precession angle.

6.2. Initial Setup / Rough Alignments

Begin with a fully aligned column in image (zoom) mode with precession off. The beam should be voltage- and current-centered through the objective lens and appear round on the sample, and the projection system should display round spots in the back focal (diffraction) plane. The beam current should be low enough so that the transmitted beam does not bloom on the detector. Set the projection system for a short camera length (~1 cm) to allow for large deflections of the beam during this alignment. The sample should be at eucentric height.

All dipole strengths in the Dipole Alignment tab should be set to 0.00.

1. Finding the reference axis (scan upper layer)
    a. Activate the X wobbler with Scan Upper selected. Set the amplitude to be fairly high with a scan frequency of 5 Hz. If during this procedure the beam is deflected off the screen or does not trace a straight line, reduce the amplitude.
    b. In the Dipole Alignments tab, set SUX and SUY to 1.00. The beam should be scanning in the +X direction.
    c. Mark the direction and amplitude of +X. This axis will serve as the reference direction and amplitude for the remainder of this alignment, including for descan.
    d. Switch to the Y wobbler. The beam should scan in the raw +Y direction.
    e. Adjust SUY and SUYx so that the Y-scan is orthogonal to and has the same amplitude as the X-scan on the detector.

2. Aligning the scan lower axes (combined scan upper and lower)
    a. Switch back to the X wobbler and select the combined scan wobbler.
    b. In the Dipole Alignments tab, set SLX and SLY to -1.00. The beam should be scanning in the raw -X direction, analogous to step 1b, but with different amplitude and possibly rotated.
    c. Adjust SLX and SLXy such that the beam does not move on the sample.
    d. Switch to the Y wobbler.
    e. Adjust SLY and SLYx such that the beam does not move on the sample.

3. Aligning the tilt (combined scan upper and lower)
    a. Switch the column to diffraction mode.
    b. Activate the X wobbler, with combined scan selected. Mark the amplitude and direction. This direction may be rotated from the X-axis in image mode, depending on whether the column has been calibrated to match image and diffraction rotation.

c. If the beam does not trace a straight line, the voltage centering may be off, or the amplitude is too high and the tilt is hitting aberrations at high angles in the objective lens. Reduce the amplitude in this case and/or improve the voltage centering for the microscope.
d. Switch to the Y wobbler and check the amplitude and direction. The direction should be orthogonal to X, and ideally the amplitude will be within a few percent. If not, adjust the entire column of Y parameters (SUYx, SUY, SLYx, SLY) by a fixed multiplier to match X and Y amplitudes. Step 2 should be repeated to correct the scan lower alignment in image mode for the Y-axis. This step may need to be iterated to achieve good tilt purity and probe localization.
e. Flip the sign of the amplitude, and check that both X and Y wobblers have the same amplitude in the opposite directions. If not, redo the voltage centering alignment for the microscope and repeat this section.

4. Finding the descan reference axis
    a. Switch the column to diffraction mode.
    b. Activate the X wobbler
    c. In the Dipole Alignments tab, set DSUX and DSUY to 1.00. The beam should be scanning in the +X direction.
    d. Match the direction and amplitude of SUX found in step 1c using DSUX and DSUXy.
    e. Switch to the Y wobbler. The beam should scan in the raw +Y direction.
    f. Adjust DSUY and DSUYx so that the Y-scan is orthogonal to and has the same amplitude as the X-scan on the detector.

5. Aligning the descan lower axes
    a. Switch back to the X wobbler and select the combined descan wobbler.
    b. In the Dipole Alignments tab, set DSLX and DSLY to -1.00. The beam should be scanning in the raw -X direction, analogous to step 1b above, but with different amplitude and possibly rotated.
    c. Adjust DSLX and DSLXy such that the beam does not move on the sample.
    d. Switch to the Y wobbler.
    e. Adjust DSLY and DSLYx such that the beam does not move on the sample.

6. Aligning the de-tilt (combined descan upper and lower)
    a. Switch the column to diffraction mode.
    b. Activate the X wobbler, with combined descan selected. Mark the amplitude and direction. This direction may be rotated from the X-axis in image mode, depending on whether the column has been calibrated to match image and diffraction rotation.
    c. Switch to the Y wobbler and check the amplitude and direction. The direction should be orthogonal to X, and ideally the amplitude will be within a few percent. If not, adjust the entire column of Y parameters (SUYx, SUY, SLYx, SLY) by a fixed multiplier to bring the tilt into round. Step 2 should be repeated to correct the scan lower alignment in image mode for the Y-axis.

   d. Flip the sign of the amplitude, and check that both X and Y wobblers have the same amplitude in the opposite directions. If not, check the centering of the projection system and repeat this section

7. In the file menu, click "Save Profile" to save the new settings.

6.3. Calibrating Scan Angle

The V/mrad value in the Prec Setup tab is used to calibrate the scan angle. To calibrate it, a sample with known lattice spacing is required, and steps 1-3 of the rough calibration in section 6.2 must already be completed. In this procedure, the beam will be precessed without descan and the circle size in diffraction space will be compared with diffraction spots of known lattice spacing.

Begin with a fully aligned column in diffraction mode with precession off. The beam should be voltage- and current-centered through the objective lens and appear round on the sample in image (zoom) mode. The projection system should display round spots in the back focal (diffraction) plane. The beam current should be low enough so that the transmitted beam does not bloom on the detector. Set the projection system for a short camera length (~1 cm) to allow for large deflections of the beam during this alignment. The sample should be at eucentric height.

1. Set parameters in the Prec. Setup tab as follows:
   a. Angle = 1
   b. Freq. = 50 Hz (or a frequency typically used for precession)
   c. V/mrad = 1

2. Tilt sample to zone axis and obtain a diffraction pattern.

3. Mark the distance between the transmitted beam and a spot in the diffraction pattern. A higher index spot is preferred. Calculate this distance in units of milliradians using the crystal dimensions, the operating camera length, and electron wavelength.

4. Start precession with scan on and descan off.

5. Increase the Angle control such that the circle radius is the same as the distance marked in step 3.

6. Switch to image mode and ensure that the beam is highly localized on the sample. Use the Scan Lower controls in the Dipole Alignments tab to localize the beam if it is wandering as it is scanned.

7. Iterate steps 5-6 until the circle radius matches the reference spot in diffraction space and the beam is localized in real space.

8. Adjust V/mrad until the value in the Angle field corresponds to the angle calculated in step 3.

9. In the file menu, click “Save Profile” to save the new V/mrad setting.

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8. Appendix

8.1. System Specifications and Requirements

Specifications:

- Digitally-controlled hollow cone scanning and descanning to 50mrad half-angle.
- Probe localization of better than 100nm, assuming column properly aligned by manufacturer.
- Hardware specs:
    - Digital generation of signals to 16 bits resolution.
    - Update rate of 500,000 samples/sec for all channels active.
    - Output for tilt position update for synchronizing a framegrabber.
    - Input for tilt position update for synchronizing with an external source.
    - AC mains synchronization to eliminate 60 cycles noise.
    - Dimensions: 19.00" x 10.61" x 3.66".
- Software specs:
    - Manual tilt purity correction.
    - Dynamic aberration compensation to 3-fold.
    - Precession profile loading and saving.
    - Custom waveform loading and saving.

Requirements:

- Electronics:
    - The coils + driver system should have flat frequency response to about 100Hz.
    - The column may have an external interface for controlling beam tilt and image shift coils. Typically this interface does not allow access to individual coil layers, in which case the internal shift-tilt purity controls must be able to compensate non-orthogonality within the physical coil axes (e.g., cross-feed X into Y and Y into X).
- Recording media:
    1. CCD camera:
        - High bit depth (12 bits or greater preferred)
        - Ability to capture an entire diffraction pattern (~200 mrad field of view, or about 10°)
        - Ability to capture diffraction pattern without saturation of all but central spots
            a. Anti-bleed feature (ability to bleed off excess current without affecting neighboring pixels)
            b. If low current modes and spreading the beam are not enough to prevent saturation, fast exposures down to about 20 ms may be required(either using beam blanking shutter or electronic shutter)
            c. If using beam blanker for shuttering, the diffraction pattern must not be swept across the camera during precession. A pre-sample blanker is preferred, and aperture-limited blanking is ideal because the effective blanking speed is reduced.

    2. Image plates (>16 bit native dynamic range)

3. Film:
    - Suggest using software that can combine multiple exposures and obtain high dynamic range (1 in 10^4)

8.2 Troubleshooting

For the precession system to function, drivers for the National Instruments PCI-6733 card and NI-DAQmx must be installed. The precession software requires the card be named "Dev1" in the NI Measurement and Automation explorer.

8.2.1. Hardware Diagnostics Interface

The software includes diagnostics for troubleshooting the hardware in the "Tools > Diagnostics" menu of the Precession Setup interface. It includes controls for testing the digital and analog signals within the system. The Diagnostics interface

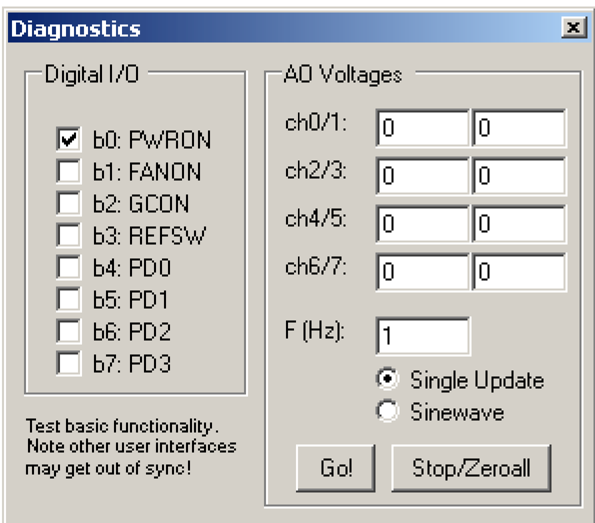

Figure B1. Hardware diagnostics interface.

Control	Description
Digital I/O	Tests the software-controlled relays.
PWRON	±15V power supplies for signal routing and powering the relay boards.
FANON	Controls the internal fan. If unchecked, fan activates if internal temperature exceeds 160°F.
GCON	Connects PC ground to analog ground internally.
REFSW	Reserved.

PD0..3	Controls the signal relays on the daughterboards.
A/O Voltages	Sets the voltage amplitude of the output channels. Set the frequency using the “F (Hz)” field, and the type of waveform using the radio buttons. Clicking “Go!” will output the voltages. Clicking Stop/Zeroall will set them to absolute zero values. These values do not use the voltage offsets or output limits in the AO Setup tab of the Precession Setup interface.