

LANDYNE

User Manual

Simulation on Polycrystalline SAED pattern
& Phase Identification

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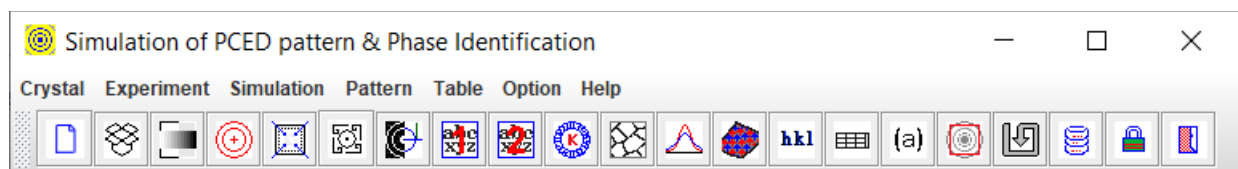


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1. Introduction

For polycrystalline or powder TEM specimens, electron diffraction ring patterns are extensively used for phase identification, in which the diffraction pattern of a known phase acts as a fingerprint.

Computer programs of powder X-ray diffraction may be used for phase identification in the analysis of polycrystalline electron diffraction patterns. Electron diffraction ring pattern needs to be transformed into the form of integral intensity as a function of scattering angle for comparison with a powder X-ray diffractogram. The other approach is to use computer programs that implement the formula for electron diffraction intensity to analyze raw or processed polycrystalline electron diffraction patterns. Integrated electron diffraction software packages often provide a module for simulating polycrystalline electron diffraction patterns, and they rarely include features for direct comparison with experimental data for phase identification. A specially designed computer program is needed to provide a fast and accurate method for accomplishing this task. We developed the first version of PolyCrystalline Electron Diffraction (JECPCED, Li 2004).

The next version is ProJECT/PCED2 (Li, 2010), which includes the following new features (i) Blackman's theory, an integral two-beam dynamical theory, for intensity calculation, (ii) Match model for out-of-plane and in-plane texture, (iii) pseudo-Voigt function for peak profile of diffraction ring and (iv) improvement on diffraction pattern indexing and matching to an experimental pattern. PCED includes a Miller-Bravais index dialog for a hexagonal system, a space group, a better GUI, and I/O compared to PCED2. PCED2 also incorporates with Landyne launcher, a desktop dock for JECPCED, and ProJECT package.

A drag and drop function was implemented, and the image operations, invert, alignment, resize, and rotation, were added in PCED3. The simulation of two crystalline phases was included, and an index system was applied in PCED4.

The current version is PCED5, which upgrades the manipulation of the experimental electron diffraction pattern and better index labels on the simulated pattern. The label tools were added to prepare figures for publication. The improvement provides a much easy way for operation, especially phase identification.

PCED was written and compiled with in JDK8. Further code optimization (including obfuscation) was carried out on the compiled class files. A license file is needed to unlock the program

(PCED.jar) for loading new input data files. Without the license file, the program works in demo mode using default input files (Al and Mg). License is available from LANDYNE computer software (jlandyne@gmail.com).

2. Theory background

The kinematic theory of electron diffraction is described in detail in any texture book on this topic (e.g., Peng *et al.* 2004). The electron atom scattering factor can be derived from the X-ray atom scatter factor using the Mott-Bethe relationship or directly obtained from a parameterized table of electron atom scatter factors (Peng *et al.* 1996). Here we use the second method.

Following the electron diffraction geometry, the radius of the diffraction ring, R , can be related to the length of the reciprocal vector $g(hkl)$ as

$$R = \frac{L\lambda}{d} \frac{\sqrt{1 - \left(\frac{\lambda}{2d}\right)^2}}{1 - 2\left(\frac{\lambda}{2d}\right)^2} \quad (1)$$

Where L is the camera length, $d=1/g(hkl)$ and λ is the wavelength of the incident electron beam.

The integrated intensity of reflections in the electron diffraction from the polycrystalline sample was derived in the book by Vainshtein (1964).

$$I = I_0 V \lambda^2 p \frac{F_{hkl}^2}{v_a^2} \frac{d_{hkl}}{2} \quad (2)$$

Here V is sample volume, λ is the wavelength, p is a multi-factor, F_{hkl} is diffraction amplitude, the v_a is the volume of unit-cell, d_{hkl} is the (hkl) spacing.

2.1 Blackman's theory on the intensity of polycrystalline electron diffraction

Blackman's theory (Blackman 1939) for polycrystalline electron diffraction intensity is based on Bloch wave dynamic theory initially formulated by Bethe. The formulas are given as integral intensity of two-beam dynamic diffraction. According to Peng *et al.* (2004), Blackman's theory for polycrystalline electron diffraction intensity is an advanced theory on this topic, although the theory was published decades ago. Readers should refer to the original paper for details derivation of the formulas. Some essential formulas are listed here.

The dynamical structure factor is given,

$$U_g = \frac{2meV_g}{h^2} \quad (3)$$

Where m is electron mass, e is the electron charge, h is Planck constant, V_g is Fourier coefficient of crystal potential.

The wave vector in a vacuum is given,

$$K_0^2 = \frac{2meE}{h^2} \quad (4)$$

Here E is the accelerated voltage of the electron microscope.

The wave vector inside the crystal is corrected with the mean inner potential,

$$K^2 = K_0^2 + U_0 \quad (5)$$

The diffraction intensity formulas can be given below in an integrated form considering there are angle distribution between the incident beam and the zone axis of the polycrystalline grains,

$$I_g^{integral} = I_0 \frac{U_g}{gK^2} \int_0^A J_0(2x) dx \quad (6)$$

Where I_0 is the intensity of the incident beam, U_g is dynamical structure factor, K is a wave vector and $J_0(x)$ the zero-order Bessel function and,

$$A = \frac{\pi U_g t}{K_n} \quad (7)$$

Here K_n is K normal component and $K^2 = K_n^2 + K_t^2$ (K_t is K component in the zero-order Laue zone).

The final intensity is an averaged integral intensity over K_n , considering the angle between the incident beam and the surface normal in a range from $-\frac{\pi}{4}$ to $\frac{\pi}{4}$.

2.2 Pseudo-Voigt function for peak profile

Although Voigt (V) function, as the convolution of a Gaussian (G) and a Lorentzian (L) functions, is regarded as the most suitable function to describe the profile of the cross-section of the diffraction rings, it is rather complicated. Pseudo-Voigt (pV) function is usually a good enough substitution for the job. the pseudo-Voigt function is a linear combination of Gaussian and Lorentzian function, as following (Li, 2007),

$$G(x) = h \exp(-4 \ln 2 (\frac{x - x_0}{FWHM})^2) \quad (8)$$

$$L(x) = \frac{h}{1 + 4(\frac{x - x_0}{FWHM})^2} \quad (9)$$

$$pV(x) = \eta G(x) + (1 - \eta)L(x) \quad (10)$$

Here h is peak height, x_0 is peak position, FWHM is full width at half maximum. Figure 1 shows the comparison of profiles of Gaussian and Lorentzian functions.

2.3 March Model for polycrystalline texture

For powder samples, axially symmetric platy or capillary specimens, composed of effective disk- or rod-shaped crystallites, can form textures due to the shape of crystallites. For thin film samples, polycrystalline microstructures with out-of-plane or in-plane preferred orientations often occur during the film growth. The diffracted intensities from preferred orientation can be corrected or measured with a single pole-density profile. The March model is used for correcting the powder X-ray diffraction with texture, which is a simple but effective method for both out-of-plane (platy) and in-plane (rode) textures.

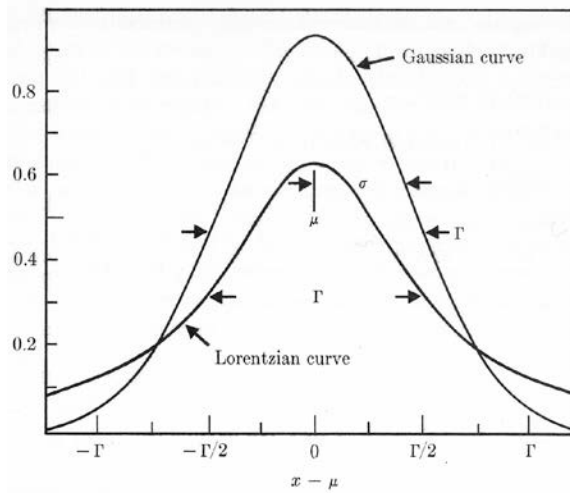


Figure 1. Comparison of profiles of Gaussian and Lorentzian functions.

The formalisms were firstly given for X-ray powder diffraction, which is reformulated for the diffraction geometry in polycrystalline electron diffraction considering the diffraction geometry of polycrystalline electron diffraction. Figure 2 shows the example curves of the March functions used in X-ray powder diffraction (Bragg-Brentano geometry) and in polycrystalline electron diffraction for platy (out-of-plane) texture. Referring to Figure 1 in the paper by Dollase (1986), the preferred zone axis density for polycrystalline electron diffraction can be formulated as follows.

For out-of-plane texture,

$$P_g(O) = P_G(\alpha) = (r^2 \sin^2 \alpha + r^{-1} \cos^2 \alpha)^{-\frac{3}{2}} \quad (11)$$

For in-plane texture,

$$P_g(O) = \tilde{P}_G(\alpha) = \frac{2}{\pi} \int_0^{\frac{\pi}{2}} P_G(\alpha, \beta) d\beta \quad (12)$$

If the axial pole-density profile, $P(\varphi)$ is known, $\tilde{P}_G(\alpha)$ maybe obtained by numerical integration making use of the relation among coordinates: $\varphi = \cos^{-1}(\sin \alpha \cos \beta)$.
 The intensity of polycrystalline electron diffraction including texture can be expressed as,

$$I_g^{ring} = s \sum_{i=1}^m P_G(\alpha_i) I_g^{integral} \quad (13)$$

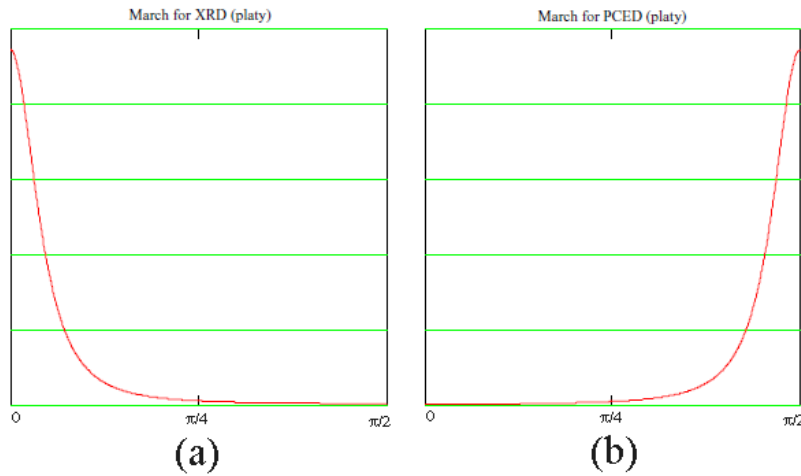


Figure 2. Example curves of the March functions used in X-ray powder diffraction (Bragg-Brentano geometry) and polycrystalline electron diffraction for platy (out-of-plane) texture. Angle away from the preferred orientation is in abscissa and the density (or weight) in ordinate.

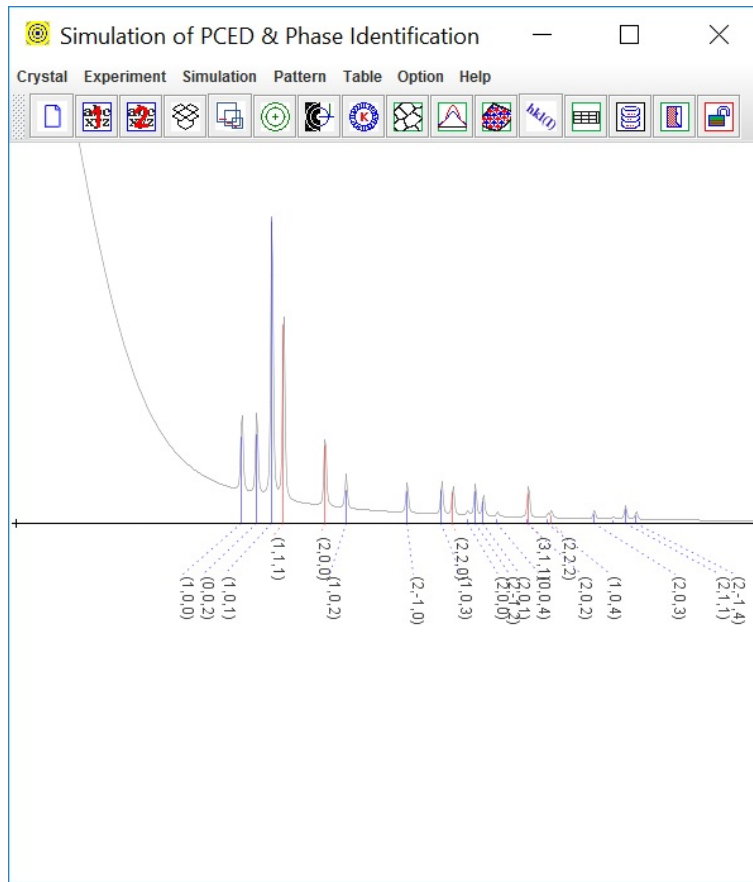


Figure 3. Snap-shot of the primary interfaces of PCED, a simulation of polycrystalline electron diffraction in a two-phase system, Al and Mg.

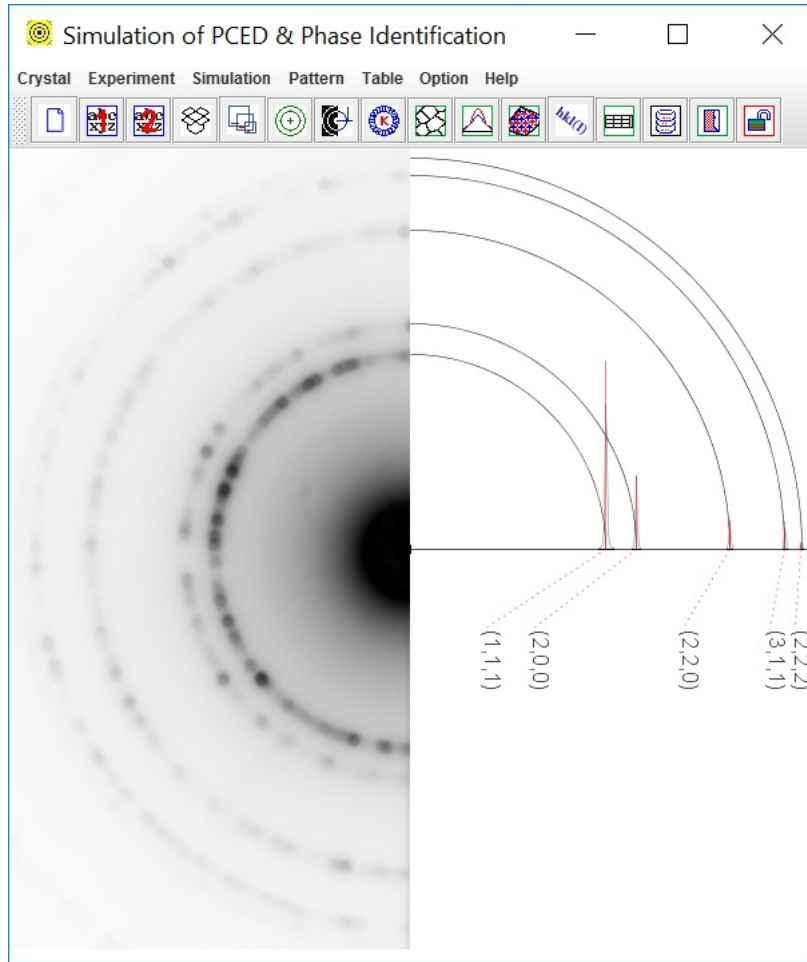


Figure 4. Snap-shot of the primary interfaces of PCED, a phase identification using calculated polycrystalline electron diffraction pattern. The experimental PCED pattern is Al.

Where s is the scale factor, $P_g(\alpha_i)$ is the density of $g(hkl)$ poles at the scattering vectors, and α_i refers to the angle between $G(HKL)$ and the i th member of the symmetry-equivalent set of m diffraction planes. The preferred orientation plane itself is a member of some set of symmetry-equivalent planes. Suppose the dominant morphological feature of the crystallites is re-indexed as some other member of this set. In that case, the individual values of α_i in the above will be permuted, but the sum of terms remains the same. The sum can be considered as a generalized multiplicity term.

3. The graphic user interface of PCED

3.1 Main interface

The main interface of PCED is shown in Figures 3 and 4, which includes a menu and a toolbar to take input parameters and a frame to show the output of the simulation. The usage of PCED for simulation needs to load structure data and to set up input parameters, then simply click the **Run** button in the **Calculation** dialog (shown later). The usage of PCED for phase identification

needs to load the experimental diffraction patterns first and then to match the calculated patterns. Two structure data can load at the same time for comparison or simulation of a two-phase system.

3.2 Menu and toolbar

Menu and toolbar can be used to pop up dialogs for data and parameters. The toolbar can also be hidden. Most functions of the menu and toolbar are the same; some functions are only provided either in the menu or in the toolbar, e.g., the drag-and-drop box for loading EDP in formats of JPEG and TIFF.

Crystal menu provides an interface for preparing the new crystal data for loading the crystal data for phase 1 and phase 2. The file name can be viewed on the Parameter table in a dialog.

Experiment menu provides an interface to load experimental polycrystalline electron diffraction (PCED) pattern in JPEG and TIFF format. The loaded PCED pattern can be inverted the grey color and can also be erased. The pattern can be resized and rotated for the best display. The pattern can be shifted to view the scale bar and make the calibration of the simulation using a G-spacing marker. The pattern can be shown in full or half mode. The file name can be displayed or hidden.

Simulation menu provides a submenu for the choice of electron diffraction theories and several dialogs (e.g., average grain size, peak profile, texture model) for input parameters in the simulation. Two theories are used for the calculation of electron diffraction intensity. The kinematical theory is selected by default, and the second choice is Blackman's theory, which is an integral two-beam dynamic theory. Intensities calculated by Blackman's theory are more accurate than those calculated by kinematical theory, but the calculation of dynamic theory will take a longer time. A dialog of an index label is used to adjust the position and font size of the index. A conversion tool is for Miller and Miller-Bravais index for a hexagonal system. A dialog of parameter-table is used for monitoring the parameters in the calculation.

Pattern menu provided the output of the simulated patterns, the hkl intensity list, the preference of the hkl-inten list file, and print to a printer or a PDF file when a PDF print driver was installed.

Options menu provides the look-and-feel provided by Java; an option to hide or show toolbar; freedom of customizing the appearance of simulated pattern; for example, the diffraction peak and ring color, the curve or solid profile; set the reciprocal spacing for a marker.

Help menu provides a tool to find the current driver and its serial number (S.N.), a table of space group numbers and symbols, version information.

4. Usage of PCED

The recommended way is to use Landyne launcher. The other ways are either double click PCED.jar. In the following, we show step by step from preparing structure data file, common routine usage for simulation, and to the last steps of saving and printing the results. More details on specific topics are left to the next section.

4.1 Prepare new crystal structure file

A structure data file can be prepared using the **New Crystal Structure File** dialogue window in Figure 5. The dialogue window provides an automatic assistant for the user and makes sure to meet the requirement of the file format. The template is embedded with the 230 space groups in the Hermann-Mauguin notation, which list in the international table for crystallography. However, only b unique axis will be used in a monoclinic system. Two origin choices can be accepted as input parameters, but choice-2 will be converted to choice-1. To save the data structure, click the **Save** button; or make a new one click the **New** button.

atom	elem #	x	y	z	occ.
		0.0	0.0	0.0	1.0

Figure 5. Snap-shot of the preparation of the new crystal structure file.

The crystal file can be also converted from a previous data for modification or from a crystallographic information file (CIF). If a data file in an alternative setting of space group for triclinic, monoclinic and orthorhombic systems is used, please click on the alternative settings (1~74). A tool for the transformation of files in the nonconventional notations to the standard notations is provided.

4.2 Simulation

The kinematical theory is used as a default selection in the simulation since it is fast and fewer input parameters are required. Integral dynamical two-beam theory (Blackman's theory) can be turned on using the **Theory** submenu in the **Simulation** menu.

Basic parameters for calculation and adjustment of the diffraction pattern can be filled in or changed in the **Calculation** dialog in Figure 6. To generate new diffraction pattern needs, click the **Run** button. The pattern is automatically updated, corresponding to the change of other parameters.

Mass ratio defines relative masses of the two phases when the fields of phase1 and phase2 are checked.

Volume ratio defines relative volumes of the two phases when the fields of phase1 and phase2 are checked.

g-spacing zoom and **Intensity scale** simulated the camera length and the exposure time in the experiment, which is used to make a better appearance of the calculated pattern.

Peak type can be chosen from the integral intensity on a ring or intensity on unit-arc-length.

The diffraction diagram can be viewed as **peak** and **profile** separately or together.

Diffraction ring can be viewed as either a ring to match the experiment pattern or only the top half in order to clearly show the index without intervening or without a ring at all.

The diffraction ring's appearance is also controlled by the profile intensity (**intensity threshold**).

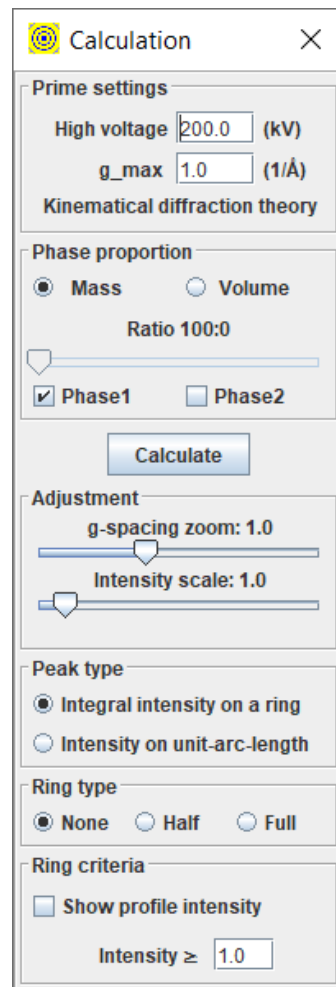


Figure 6. Snap-shot of the calculation dialog.

4.3 Index and intensity label

There are cases that diffraction peaks are quite dense; the index labels may be overlapped when they are shown by the side of the diffraction diagram, especially for the diffraction diagram of the two-phase system. It is essential to allow users to adjust the positions of the index (and intensity) labels.

In order to avoid the situation, PCED is allowed to select the location of an index label and to spread it when necessary. In the **Index Label dialog**, a top point can be viewed when the checkbox of the **Top point** is selected and moved around with mouse drag-and-drop. Dash

line can be viewed when the **Baseline's** checkbox is selected and moved up/down by the mouse wheel or the arrow keys (after clicking the panel).

Figure 7 shows the example for adjusting the positions of index labels. The Label dialog provides i) remove indexes of the weak diffraction peaks; ii) position adjustment; iii) the font size for phase 1 and phase 2. To use the position adjustments of the index labels, i) select phase 1 or phase 2; ii) tick the checkbox for index; tick the checkbox for the guidelines; iii) select Top point or Baseline; iv) move the mouse pointer to adjust the positions of index labels by a drag-and-drop operation; v) remove the indexes of weak diffraction peaks by increasing the intensity level (arbitrary unit); vi) select the link lines on or off; vii) choose the proper font size.

4.4 Phase Identification

It is fairly easy to make a phase identification using PCED. An experimental PCED pattern and the crystal data of the possible phases are needed. The PCED pattern is in JPEG (.jpg) or TIFF (.tif) formats, which can be quickly loaded using the window file system in a menu or the drag-and-drop box in the graphic menu bar. The PCED pattern can be processed, such as resize, rotate, and center, as shown in Figure 8. The scale-bar on the PCED pattern can be used for calibration of the G-spacing zoom in Figure 9. The PCED pattern can be displayed in full or half size. The simulation of the possible phases is then calculated on the right side of the pattern panel if the positions and intensities of the diffraction rings are matched in agreement with the experimental PCED pattern.

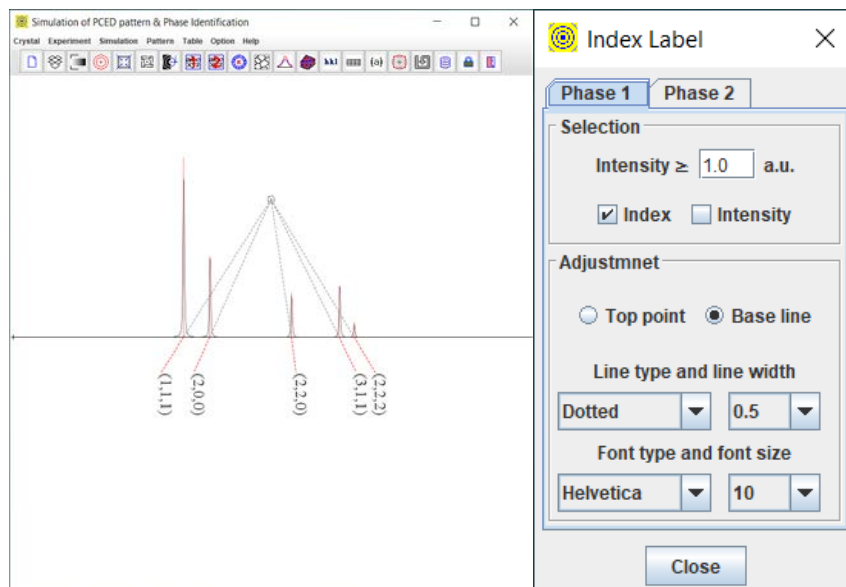


Figure 7. Example for the Label adjustment. Snapshot of the Label adjustment dialog.

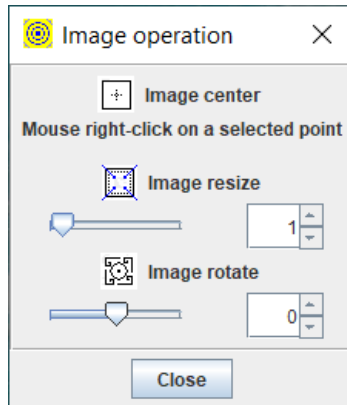


Figure 8. Snap-shot of the image operation, such as alignment, resize, and rotation.

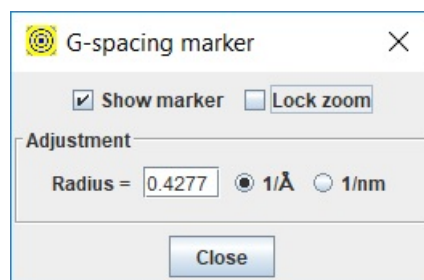


Figure 9. Snap-shot of the G-spacing marker.

The phase identification is made; otherwise, the next possible phase is loaded for a check until all possible phases are run out.

For the hexagonal system, the index can be the three-index Miller notation or the four-index Miller-Bravais notation. Three-index Miller notation is used in the PCED. A tool for converting the Miller notation and Miller-Bravais notation is also provided, as shown in Figure 10.

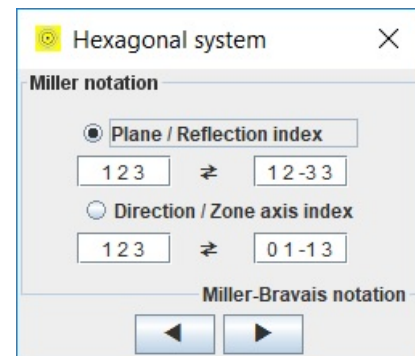


Figure 10. Snap-shot of the Hexagonal system index.

4.5 Output for publication

Calculated diffraction data can be saved into a file using **Save** in **File** menu, in which it includes hkl and intensity (per unit-length) together with the parameters used in the simulation as a default option. More info, such as length of a reciprocal vector (g), crystalline plane spacing (d), intensity (per unit angle), can be chosen to be saved by using **selection in hkl-inten** dialog in the options menu.

Simulated diffraction patterns and preloaded experimental patterns can be saved as output files in TIFF and JPEG format. The output size can be selected through the region of interest (ROI) and the point per inch (PPI). A tool for figure label is available, so the figure in one-step to be used for publication. An example figure and the tool for the figure label are shown in Figure 11.

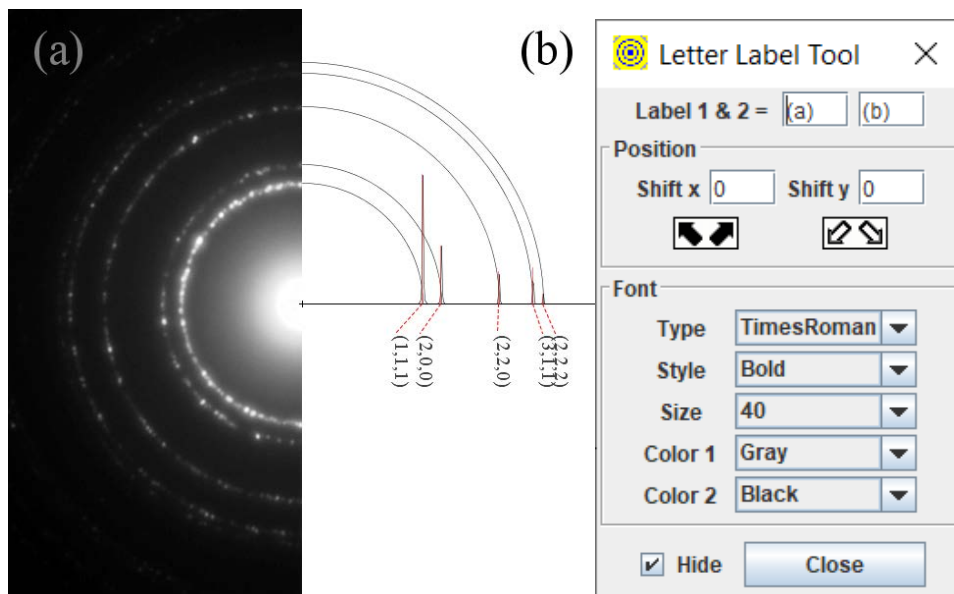


Figure 11. Snap-shot of output and a tool for figure label.

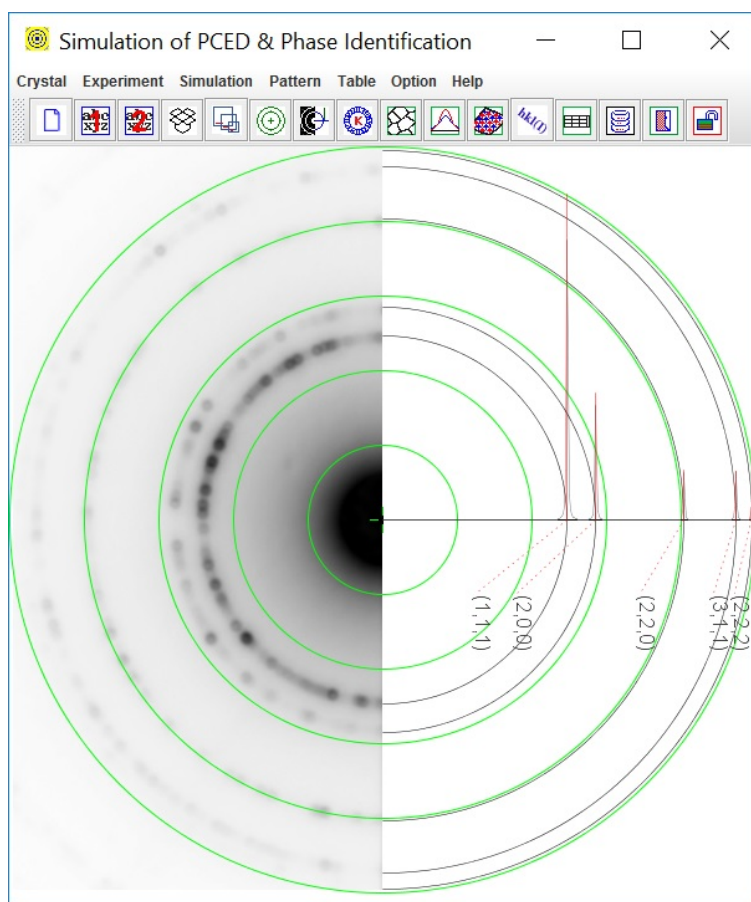


Figure 12. Snap-shot of experimental pattern processing, resize, rotation, and center.
(Snapshot of PCED)

5. Special topics on PCED

5.1 Partial occupancy factor and isotropic temperature factor

Some atom coordinates maybe not in full occupancy in a crystalline structure. In this case, the occupancy factor (default value 1.0) should be changed to the values according to the crystalline structure in preparation for the structure data file for polycrystalline electron diffraction simulation.

The partial occupancy factor can also be used to simulate a certain level of chemical order in structure. For example, the chemical ordered FePt L1₀ phase, see section 6. In this case, a different type of atoms may be assigned to the same atomic coordinates with different occupancy according to the chemical ratio. However, the sum of the two atoms' occupancy factors is 1.0.

Isotropic temperature factor is used here to simulate the effect of lattice vibration (Debye model). Although the isotropic temperature factor is a rough model, it can be used to simulate the decrease of the diffraction intensity with the variation of the $|g|$ value. The higher the value of $|g|$, the more decrease of the diffraction intensity.

5.2 Load and center EDP

For phase identification, the experimental polycrystalline electron diffraction pattern is needed to load up and compare with simulated patterns, which were calculated from the related phase files. The experimental pattern should be in JPEG and TIFF formats. The experimental pattern can be loaded using the **Load** in the **Experiment** menu or drag-and-drop box and then resized / rotated / centered. Once **Shift & Center** is clicked, concentric circles will appear in the main panel. Select the center of the pattern using a mouse and hold on the left button of the mouse; the pattern can be dragged and drop into the center of the panel, see Figure 12. Click the **Shift & Center** again; the PCED pattern is locked up in the position, and concentric circles disappear. More adjustments in small steps may be needed to find the accurate position. The number of concentric circles can be changed using the **Number of Reference Circle**.

5.3 Peak profile, incident beam, and texture

Profile of the cross-section of diffraction ring is referred to as peak profile, which can be simulated using a pseudo-Voigt function, together with grain size (diameter) and constant K. The ratio of Gaussian and Lorentzian functions in pseudo-Voigt function can be adjusted using **Peak Profile** in the **Simulation** menu (default value is 0.5). The grain size (diameter) and constant K can be adjusted using the **Average Grain Size** dialog in the **Simulation** menu.

The Incident beam in polycrystalline electron crystalline is very strong comparing to the diffraction ring; it may submerge the relative weak rings near the incident beam. The incident beam can be included in the simulated pattern by check on the **Included** box in the **Peak Profile** dialog and adjust the shape by changing the corresponding values of height and fwhm. Peak and profile can be selected individually, and the style of profile can be chosen between a solid shadow or curve.

5.4 Parameter table

In **texture dialog**, the axis of texture and March parameter (default value 1.0 for full random case) can be input for simulation. These values can be set up separately for phase1 and phase2. March parameter (r) is suitable for describing the slight deviation of the randomness of polycrystalline/or powder sample. Thus the higher the value, the better approaching the randomness.

To describe the status of preferred orientation, we suggest using the reversed March parameter $r' = 1.0 - r$. Thus, full randomness $r' = 0$, the higher the preferred orientation, the higher the value of r' and for the perfect prefer orientation $r' = 1.0$.

For convenience to track down the simulation parameters, a **Parameter Table** can be pop up using **Parameter Table** in **the Simulation** menu, which lists the essential parameters of phase1 and phase 2.

5.5 Determine the weight ratio of a two-phase system

PCED provides a way to simulate the composite diagram of the two-phase system with a given weight ratio. The formula used in the program is to convert the weight ratio into the numbers of unit-cell in diffraction using atomic weight per unit-cell for each phase for simplicity. Experimentally determining the two-phase system's weight ratio should be calibrated by using a standard sample with a known weight ratio.

5.6 Grain size and the FWHM of diffraction ring

PCED provides a way to simulate the FWHM of diffraction rings for a given average grain diameter and K factor. The values of FWHM for each ring in the calculated diagram are consistent with the same pseudo-Voigt function used in the simulation. The diffraction rings in the experimental pattern are often composed of separately individual spots from grains with different grain size; the peak profiles are obtained by using data sum up to around each ring. In such a case, the peaks may need to fit different pseudo-Voigt functions for each ring. The grain size is not related to the peak profiles of polycrystalline electron diffraction ring pattern in a simple way. The grain size should be measured by using a bright field or dark field imaging method.

6. Examples

6.1 Phase identification of the Cr-N thin films

According to the phase diagram of the Cr-N system (Venkatraman & Neumann, 1990), the Cr₂N phase is stable with a nitrogen composition range of about 27.5–33 at.%, and the CrN phase has a very narrow equilibrium range (47.5–50 at.% N). CrN has a face-centered cubic (f.c.c.) structure ($a = 0.414$ nm) while Cr₂N has a hexagonal structure ($a = 0.4752$ nm and $c = 0.4429$ nm) (Villars & Calvert, 1985).

It is well known that Cr–N thin films have been studied continuously as important coating materials. On the other hand, our research on Cr–N films originated from studying Cr-doped AlN (AlCrN), a ferromagnetic semiconductor system. To clarify the origin of the ferromagnetism, we must exclude the contributions from any possible second phases in AlCrN, including CrN and Cr₂N. Therefore, the structure and properties of Cr–N films were studied (Li, et al. 2004).

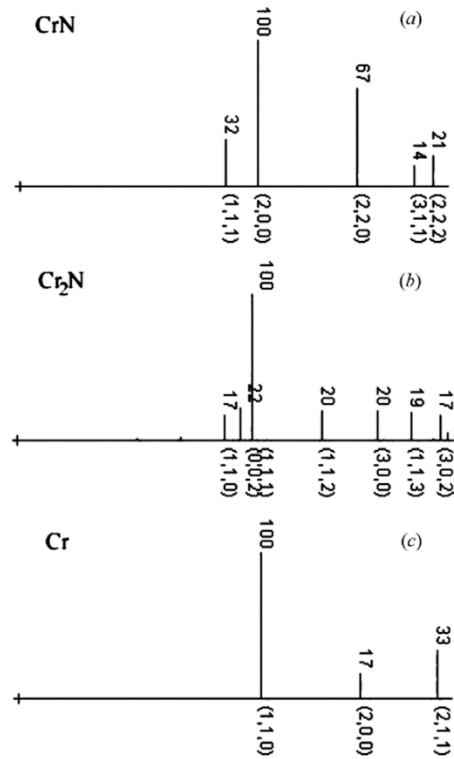


Figure 13. Comparison of calculated polycrystalline electron diffraction patterns of (a) f.c.c. CrN, (b) hexagonal Cr₂N and (c) b.c.c. Cr, with intensities (b) on top and indices at the bottom of each peak.

Cr–N films were grown on Si (001) substrates by reactive magnetron sputtering under an N₂/Ar atmosphere at room temperature. The composition of the films, expressed as Cr_{1-x}N_x, can be varied by changing the N₂/Ar pressure ratio during the synthesis process. Two batches of thin films were prepared with different N₂ partial pressures, sample 1, P(N₂) = 5 x 10⁻¹ Pa and sample 2, P(N₂) = 5 x 10⁻³ Pa.

Simulation of the polycrystalline electron diffraction patterns from CrN and Cr₂N and Cr

Fig. 13 shows the calculated polycrystalline electron diffraction patterns of the structures of (a) the f.c.c. CrN phase (Villars & Calvert, 1985), (b) the hexagonal Cr₂N phase (Kim et al., 1990) and (c) b.c.c. Cr. The calculated patterns show that it should be possible to distinguish the CrN, Cr₂N and Cr phases by their polycrystalline electron diffraction patterns.

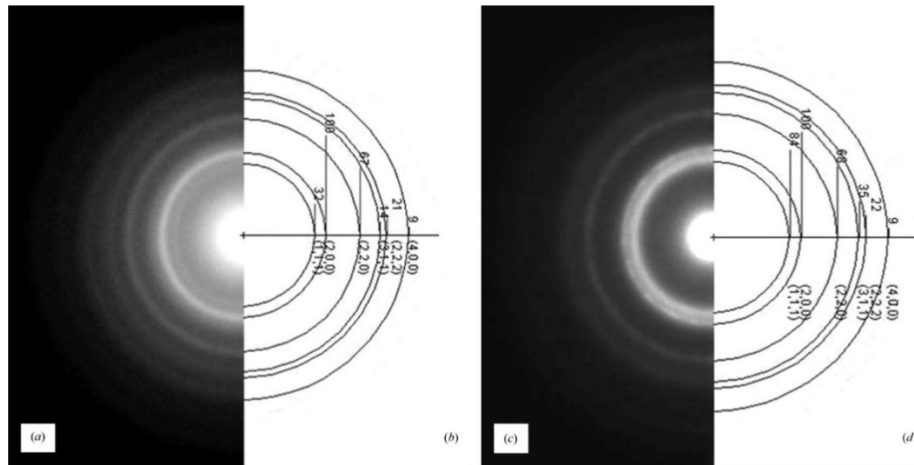


Figure 14. Experimental polycrystalline electron diffraction patterns of thin films produced (a) with N_2 partial pressure $P(N_2) = 5 \times 10^{-1}$ Pa and (c) with N_2 partial pressure $P(N_2) = 3 \times 10^{-3}$ Pa. Simulated patterns (b) based on the cubic CrN structure and (d) based on the cubic CrN structure with half occupancy of N sites.

Thin films produced with different N_2 partial pressure

Fig. 14 shows (a) a polycrystalline electron diffraction pattern taken from sample 1 and (b) a simulated pattern calculated on the basis of the structure of the f.c.c. CrN phase. Both the radii of the rings and their relative intensities are in good agreement, confirming that the microcrystalline phase in sample 1 is the f.c.c. CrN phase. Fig. 14(c) shows a polycrystalline electron diffraction pattern taken from sample 2. The simulated patterns (see Figs. 13b and 13c) of the hexagonal Cr_2N phase and Cr do not fit the experimental pattern. The radii of the rings in the experimental pattern are the same as those in Fig. 14(a), although the relative intensities of corresponding rings are different. Therefore, the microcrystalline phase in sample 2 should have the same or a very similar type of unit-cell and lattice parameters. Considering the fact that sample 2 was synthesized under a low N_2 partial pressure compared with that used for sample 1, we tried to use the structure of the cubic CrN phase with vacancies to interpret Fig. 14(c). A simulated pattern is shown in Fig. 14(d), which is calculated using the structure of the cubic CrN phase with half occupancy at N sites. This figure shows that the intensity of the inner most ring dramatically increased while the intensities of other rings do not change very much. The simulated pattern in Fig. 14(d) can be used to match the experimental one in Fig. 14(c); thus, the microcrystalline phase in sample 2 can be interpreted as the cubic CrN phase with vacancies at the N sites.

6.2 Analysis of thin films with FePt $L1_0$ phase

In order to increase the areal density of magnetic recording media, the grain sizes of the magnetic films must be reduced. A high-anisotropy material is thus essential for the new media to retain thermal stability. The high anisotropy energy of the FePt $L1_0$ phase makes the FePt alloy system one of the most promising materials candidates. As-deposited FePt films typically consist of the disordered fcc phase, which is a magnetically soft phase and can be transformed into the $L1_0$ phase upon annealing treatment. The chemical order of the $L1_0$ phase depends on the composition and the heat treatment condition of the film.

For data-storage applications, the [001] axis of the $L1_0$ phase should be perpendicular to the film plane since the [001] axis of the $L1_0$ phase is an easy magnetic axis. Hence, the films in a (001) texture have an advantage over randomly or other (e.g. (111)) oriented films. One way to achieve the (001) texture has been developed by depositing a multilayer of Fe and Pt film and annealing at elevated temperature. The formation of the (001) texture for various ranges of film thicknesses was a focus point in the research work.

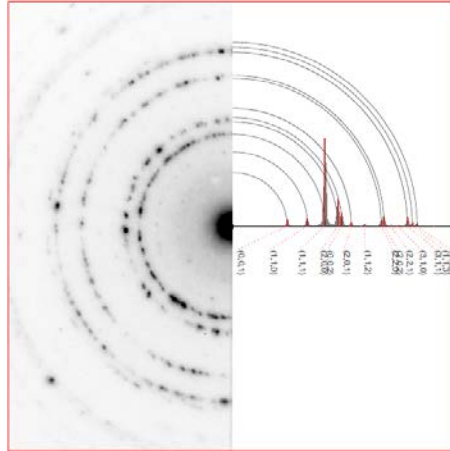


Figure 15. SAED pattern of the FePt $L1_0$ phase in a film with a thickness of 20 nm annealed at 600 °C for 30 seconds and calculated pattern for phase identification.

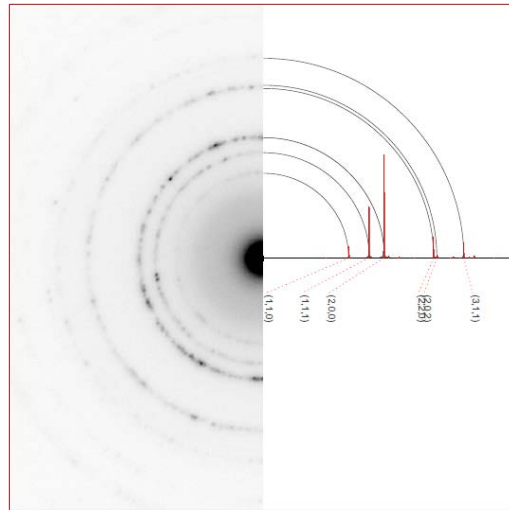


Figure 16. SAED pattern of the FePt $L1_0$ phase in a film with a thickness of 4 nm annealed at 600 °C for 30 seconds and calculated pattern with (001) texture.

FePt film with a thickness of 20 nm

Figure 15 shows the SAED pattern of FePt film with a thickness of 20 nm annealed at 600 °C for 30 seconds and the calculated pattern of the FePt $L1_0$ phase-in for phase identification. The ring radiuses and corresponding intensities in the calculated pattern are considered to match well with the experimental pattern. On the other hand, the ring radiuses and corresponding intensities in the

calculated pattern with FePt fcc polycrystals (not shown here) do not match the experimental pattern. Thus the phase is confirmed to be the chemical ordered FePt L₁₀ phase.

FePt film with thickness 4 nm

It is clear that the radius step of the diffraction patterns in Figure 15 is the same as those in Figure 16; however, the intensity distributions of the diffraction patterns are dramatically different. Now we estimate the (001) texture of the film with a thickness of 4 nm semi-quantitatively.

Fig.17 is a processed digital diagram of Fig.16 using a program QPCED (Li, 2007). Using March parameters of 0.45 and a given chemical order (Pt sites with 80%Pt+20%Fe; Fe sites with 80%Fe+20%Pt), a calculated diagram in Fig.18 is best matching with Figure 17 is obtained. As discussed in section 5.3, if we use revised March (0.0 for full random and 1.0 for perfect texture), the texture can be described as 55% of the (001) texture.

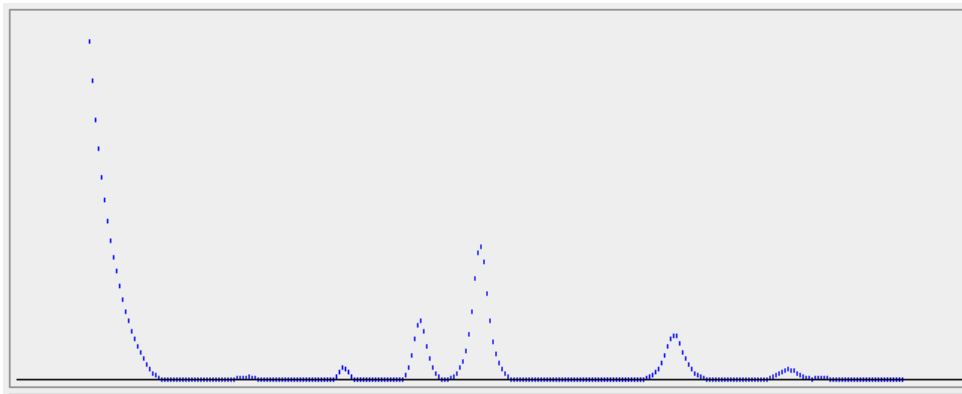


Figure 17. Digital processed diagram of the experimental electron diffraction pattern in Figure 17.

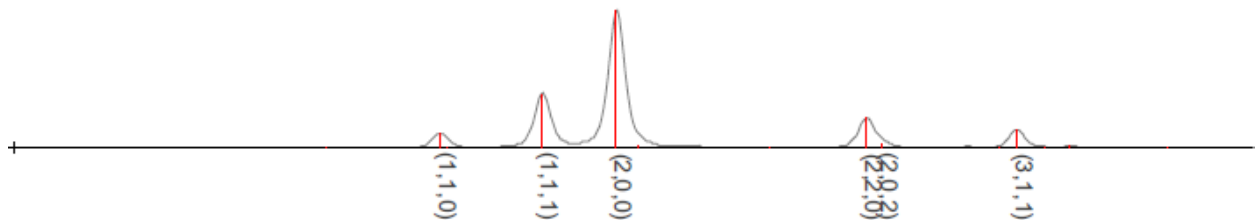


Figure 18. A simulated diffraction diagram fitting to Figure 18 reveals the degrees of chemical order and (001) texture of film with a thickness of 4nm.

7. Installation of PCED

7.1 Installation

Download and install the most recent JRE from the Java official site. Download and install the JAI, which is also available at <https://www.unl.edu/ncmn-enif/xzli/download>.

Download (<https://landyne.com>) and decompress the Landyne5 package. Copy the license to the programs\ subfolder.

7.2 PCED in Landyne suite

Click the PCED in Landyne launcher to start the software.

7.3 License

A license file is needed to unlock the program (PCED.jar) for simulating PCED patterns. The program may run in demo mode, which works in full functions but limits to the given crystal files. A license is available from LANDYNE (computer software and graphic design).

8. References

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