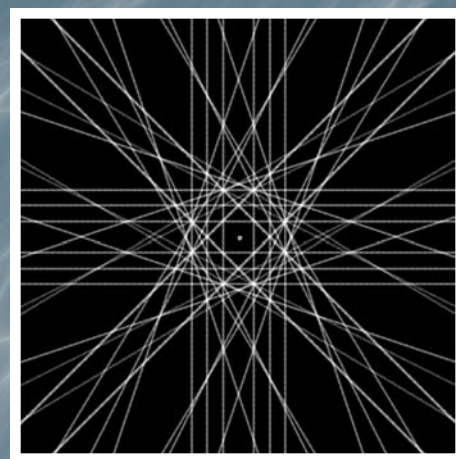
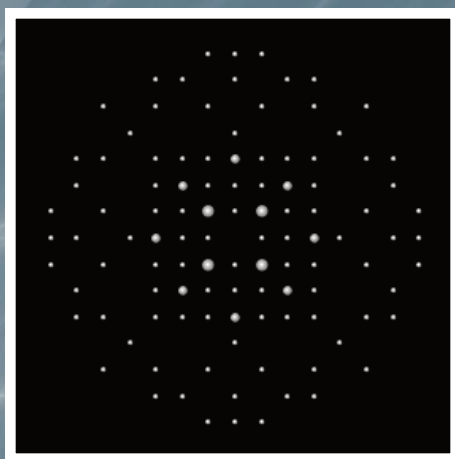
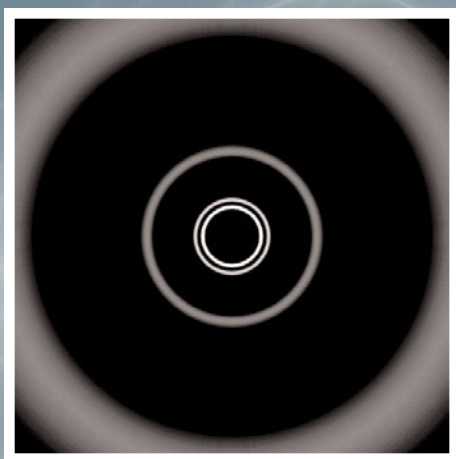


# Electron Diffraction



## International Centre for Diffraction Data



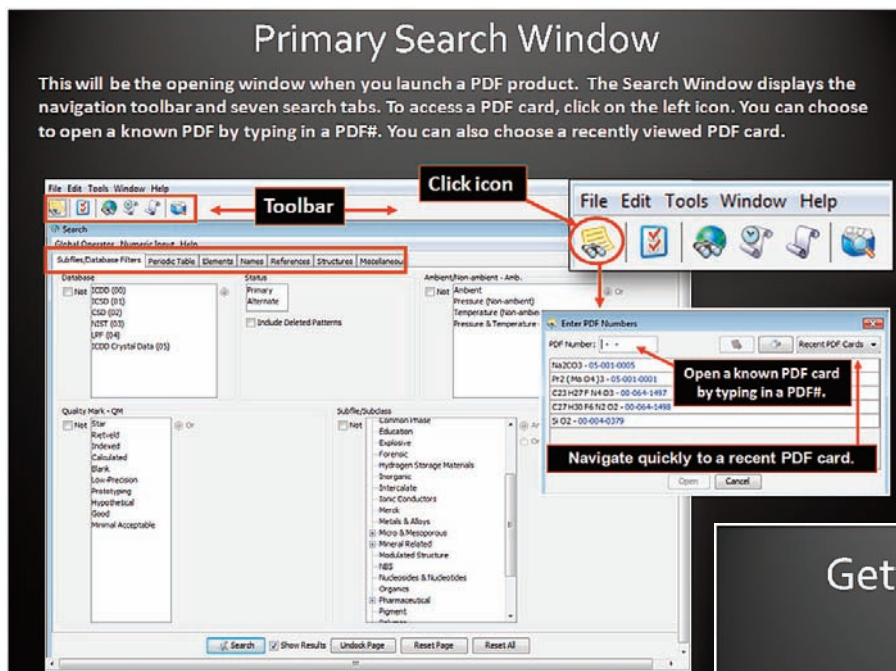
# Getting Started

**ALL** ICDD Powder Diffraction File databases can be opened as a stand-alone program. Our integrated software offers users flexible searches, data mining, and graphics capabilities.

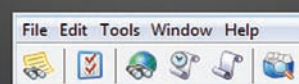
## To access the PDF Database:

Choose the PDF database icon on your desktop OR

Start menu → All programs → ICDD PDF Product (folder) → ICDD PDF Product



## Getting Started: Tool Bar



### Quick Navigation Icons

- Select a PDF Card
- Select Preferences Module
- Open Search Window
- Open History Window
- Open Results Window
- Open Sieve or Sieve+

### Quick Icon Tips

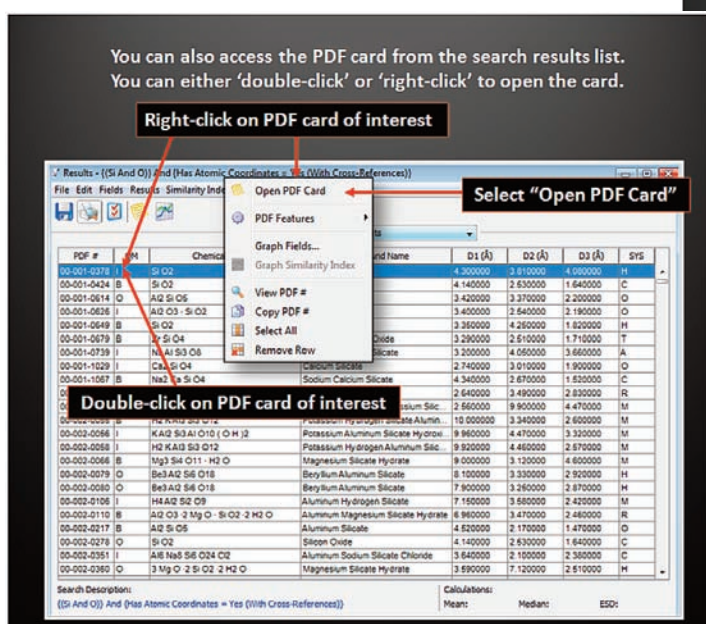
- Text Criteria Icon – select button to display a list containing lookup values.
- List Criteria Icon – select button to display a single-level or multi-level listing of search criteria.

You can also access the PDF card from the search results list. You can either 'double-click' or 'right-click' to open the card.

**Right-click on PDF card of interest**

**Select "Open PDF Card"**

**Double-click on PDF card of interest**



For additional details on using PDF products, please access **USE PDF-4 DATABASE** or **USE PDF-2 DATABASE** tutorials from the ICDD website ([www.icdd.com/resources/tutorials](http://www.icdd.com/resources/tutorials)).



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The **Powder Diffraction File™** (PDF®) is a comprehensive materials database that is the key source of reference powder diffraction data for identification and analysis of materials of all types; from natural minerals and high-tech ceramics, to metals and alloys, and pharmaceuticals. New developments in X-ray analysis, and advances in scientific research, have dramatically influenced the range of data present in the PDF. ICDD's premier database, PDF-4+, contains d-spacings, intensity, and hkl data used for phase identification, as well as atomic coordinates for more than 223,000 phases. In addition to being a reference database used for the analysis of traditional single phase X-ray diffraction data, the PDF-4+ supports electron diffraction (ED) based analyses as well. ICDD has continuously developed tools for identifying materials by electron diffraction methods since the 1950's. Most recently, a suite of simulation tools, interfaced to a relational database, has been under development. These tools have been designed to allow users to identify materials via filtering the database by elemental composition and physical properties combined with the diffraction pattern.

Often in materials characterization diverse forms of diffraction are used to complement one another. For decades, research scientists have used electron diffraction as a method for obtaining diffraction intensities from nanoscale quantities of a material, when X-ray scattering is limited. The increased intensity of an electron beam versus an X-ray beam, allows the user to investigate smaller samples, and utilize a more focused beam to determine the atomic arrangement of crystals. However, the high energy beam can often promote multiple scattering interactions; making structure solution by electron diffraction alone relatively difficult compared with X-ray diffraction structure analysis. In support of the diffraction community, the ICDD has continued to develop tools that enable users to specify the form of diffraction they choose to reference. Given the complimentary character of these two techniques, it seemed only logical to compile this data into one comprehensive database, PDF-4+.

Over the last several years, we have been enhancing the suite of electron diffraction tools, which currently consists of simulation modules that generate electron diffraction spot patterns, 2-D powder patterns, and electron back-scattered diffraction patterns. These patterns can be used to verify the presence of a phase in an unknown material, and may also provide vital information about the crystal symmetry, crystallinity, and orientation of the material.

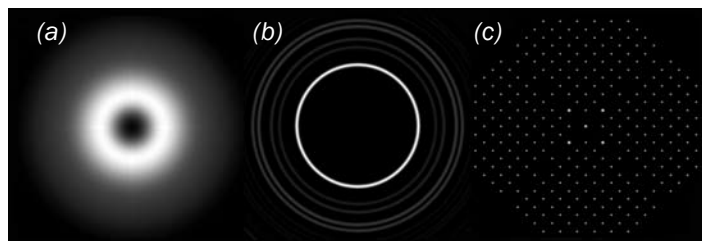


Figure 1: Electron diffraction simulation of (a) an amorphous, (b) a polycrystalline, or (c) a single-crystal material showing the variation of ED patterns due to crystallinity.

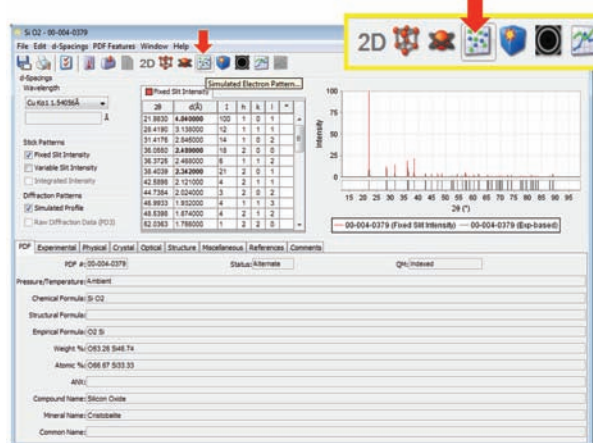
The simulations are derived using calculations based on the atomic parameters, electron scattering factors, and/or X-ray scattering factors specified in the PDF entry of interest. Each simulation is interactive and allows the user to perform instantaneous alterations to the pattern by adjusting dynamic parameters such as zone axes, camera length, electron voltage, etc.

## SPOT PATTERNS

The simulation of selected area electron diffraction (SAED) patterns was introduced to the PDF in 2006, based on programming originally developed and kindly provided by R.P. Goehner. At that time, only the spot positions were simulated from the crystallographic information in the database with no intensity estimation. Subsequent software releases approximated, and later calculated, intensities for the spots taking into account both reflection multiplicity and systematic absences. The features available in the PDF-4+ electron diffraction suite centered on spot patterns include (i) spot pattern simulations, and (ii) importing of a users' experimental spot pattern data to construct graphical overlays that can be utilized during phase comparisons/identification. The simulated spot pattern features in the PDF-4+ software can be accessed using two options (Figure 2).

### Option A: TOOLBAR ICONS

Open PDF card of interest, and then click on the "Simulated Electron Pattern" icon located in the tool bar.



### Option B: RIGHT-CLICK MENU

Right-click on the entry of interest in a PDF-4+ search results list, then select "PDF Features" and choose the "Simulated Electron Pattern" option.

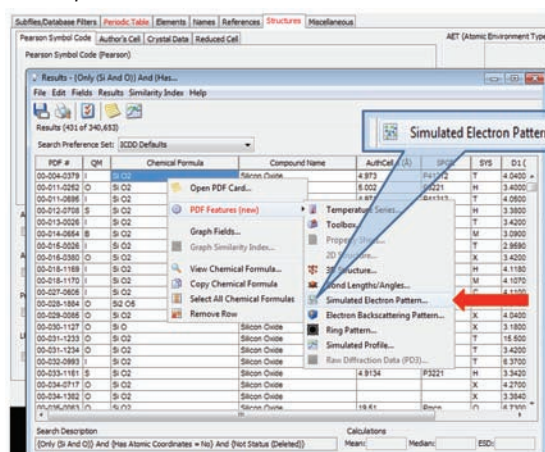


Figure 2: Options for accessing PDF-4+ simulations.

Both options will open the spot pattern simulation screen shown in Figure 3. The top portion of the screen displays the materials' crystallographic information, and the interactive simulation is located in the center of the panel (with a zoom scrollbar directly at its left). On the right, are various dynamic parameters used to modify the simulation according to the user's preference(s). As mentioned above, there are several methods for simulating the electron spot pattern intensities, and the user can verify the source used to calculate the intensities associated with their simulation by viewing the sentence located directly below the simulation pattern. In Figure 3, we observe a simulation where the ***"Intensities have been calculated using atomic coordinates"***.

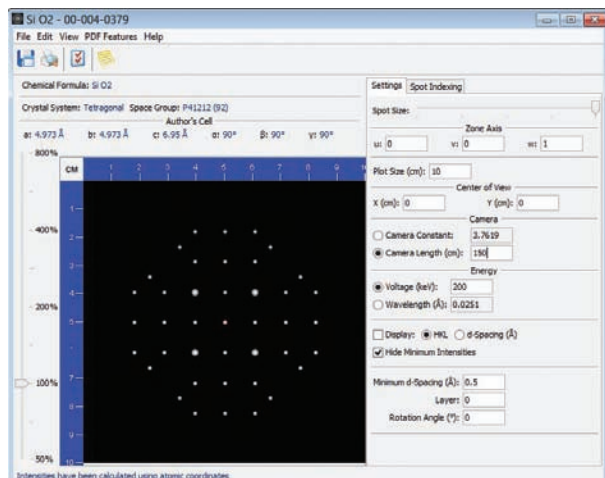


Figure 3: Display screen of spot pattern simulation for  $\text{SiO}_2$  (PDF 00-004-0379).

To construct graphical overlays for comparison to ED simulated spot patterns, the user can import their experimental data by selecting FILE → Import Image (on the simulated spot pattern screen: Figure 4). The software currently supports \*.jpg, \*.gif, and \*.png file formats. When the experimental data are present, the user can then manipulate the simulated pattern using the ED dynamic parameters to determine whether their experimental data are comparable to the PDF reference given proper crystal and experimental conditions.

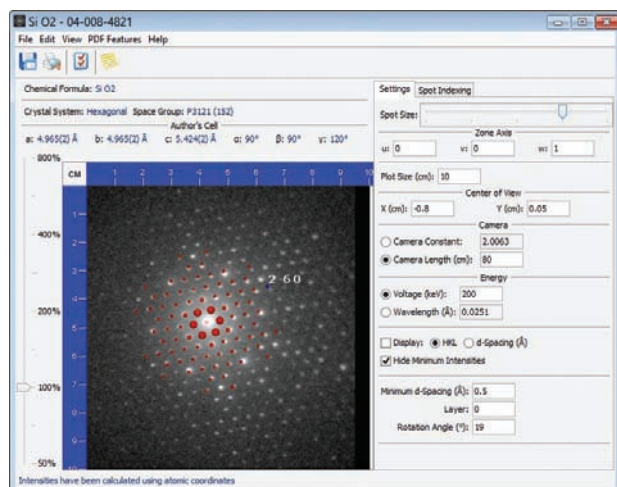


Figure 4: Graphical overlay of user's imported data (black spots) with spot pattern simulation (red spots).

## POWDER (1-D) and RING (2-D) PATTERNS

Powder (1-dimensional) and ring (2-dimensional) pattern simulations can be accessed by the same methods explained previously for spot patterns; by choosing the "Ring Pattern" or "Simulated Profile" option from the icon toolbar (on the PDF card) or using the right-click drop down menu (from the results list). Both options will bring you to the appropriate simulation screen (Figure 5).

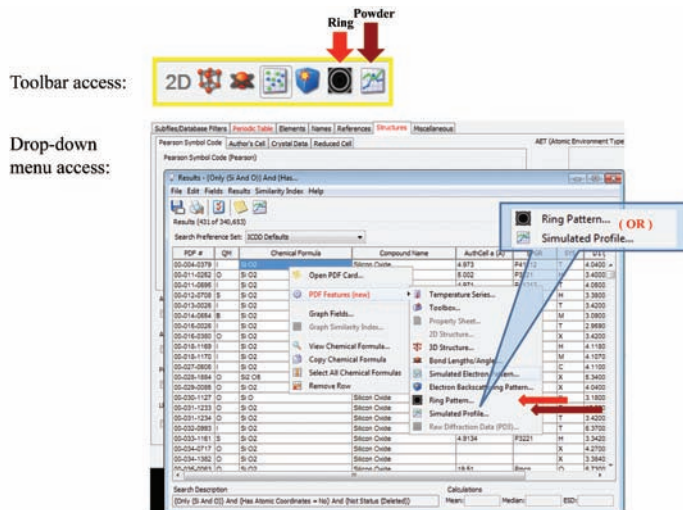


Figure 5: Methods to access simulations by toolbar icons (option A) or right-click menu (option B).

When viewing the ring pattern simulation, the user will encounter an interactive pattern (center) bordered by a scale bar, a zoom scrollbar (on the right), and a group of dynamic parameters to modify the simulation (on the left). To simulate a ring pattern based on electron diffraction, the user must select the radial button for "Electron Diffraction" from the group of dynamic parameters. Other variables can be modified to simulate experimental results under certain user-specified conditions. As with the other interactive simulations, the pattern will update instantaneously.

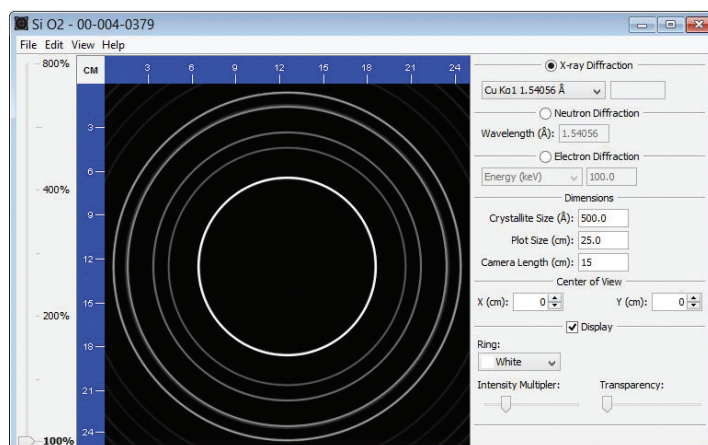


Figure 6: Display screen of ring pattern simulation for  $\text{SiO}_2$  (PDF 00-004-0379).

The simulated ring patterns provide the user with a 2-D representation of the data as it would appear on a flat 2-D detector (without taking into account contributions from



preferred orientation, axial positioning, or inconsistent crystallite sizes). The ring intensities are calculated from atomic coordinates where available, or from structure factors based on 200 KeV electrons if atomic coordinates are not available, or may be approximated from X-ray intensities if only cell parameters are available.

The 1-D powder pattern simulation is also interactive; however, it can be a bit more complex to locate the dynamic parameters. The dynamic parameters for the 1-D powder simulations are only accessible through the PREFERENCES menu or by right-clicking on the simulated pattern once it has been opened (Figure 7). They are not displayed the same as previously discussed simulations. Unlike the 2-D ring patterns, the 1-D powder pattern simulation allows the user to account for orientation contributions.

By providing PDF-4+ users with both 1-D and 2-D simulation options, we are enabling them to verify identification of unknown components using a variety of experimental data forms.

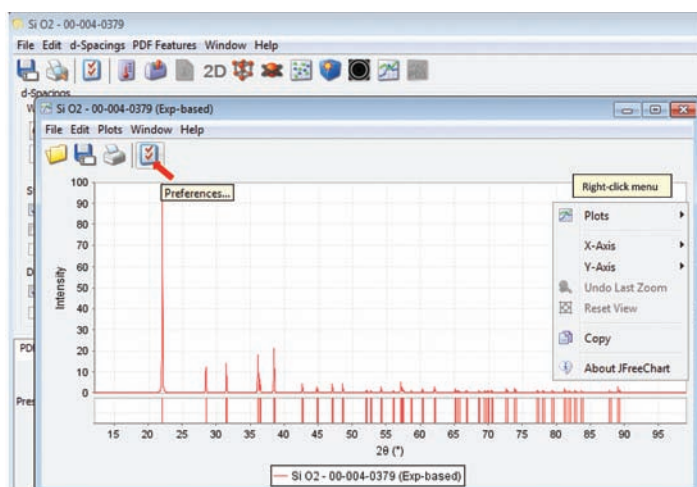


Figure 7: Method for accessing dynamic parameters for simulated powder diffraction profiles through the PREFERENCES icon or the right-click menu.

## ELECTRON BACK-SCATTER DIFFRACTION (EBSD)

Electron backscatter diffraction simulations are also available in the PDF-4+ database. Electron backscatter diffraction pattern simulations were introduced to the PDF in 2006 at the same time as the SAED pattern simulations. The EBSD pattern simulations are based on programming provided by R.P. Goehner and J.R. Michael. The line positions are determined kinematically using unit cell parameters and space group extinction conditions associated with the PDF entry, and the EBSD patterns are generated accordingly. Similar to all previously discussed simulations, these patterns can be accessed by two methods: (i) toolbar icon on the PDF card or (ii) right-click menu (Figure 5); however, one must choose the option for “Electron Backscattering Pattern”. This option will take you to the display shown in Figure 8, which has been modeled after the previously described spot pattern simulation display. The

dynamic parameters are used to manipulate the simulation and generate a pattern under user-specified conditions. The intensities used for EBSD simulations can be generated by various methods. The user can verify the source used to calculate the intensities associated with their simulation by viewing the sentence located directly below the simulation. For this particular entry we observe a simulation where the **“Intensities have been calculated using atomic coordinates”** (Figure 8). Various interactions occur in EBSD that can affect the line intensities. However, the PDF-4+ EBSD simulation tool was not designed to account for all interactions as a user may be unable to define parameters needed for calculating the dynamic interactions for these simulations. Therefore, the currently available EBSD simulations only take into account kinematic contribution to the diffraction intensities. Nonetheless, this simulation provides the user a primary idea of where the EBSD lines would be observed in the pattern.

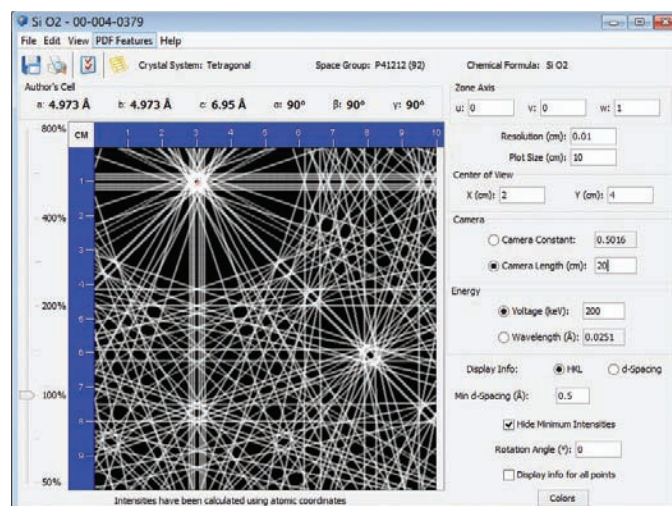


Figure 8: Display screen of electron backscattered diffraction (EBSD) pattern simulation for SiO<sub>2</sub> (PDF 00-004-0379).

## SIEVE+ SEARCHES

Since the PDF-4+ is commonly used as a reference database for phase identification, additional electron diffraction based search/match capabilities have been incorporated into the SIEVE+ search/match software. SIEVE+ was designed to replace traditional manual search methods used with printed manuals. The SIEVE+ program utilizes various search algorithms (i.e Hanawalt, Fink, Long8), including an electron diffraction based algorithm designed to work with 2-D (spot pattern) data. To utilize this option, the user must select the “Electron Diffraction” radial button from the SIEVE+ tab, in the PREFERENCES menu (Figure 9). Once the user has extracted a d-I list from their electron data, the list can be imported into SIEVE+ where an ED-based search/match comparison can be carried out. This type of search (using an ED-based algorithm) takes more time to complete than typical search/match processes because all diffraction spacings present in the d-I list are taken into account, not just a few most intense spacings.

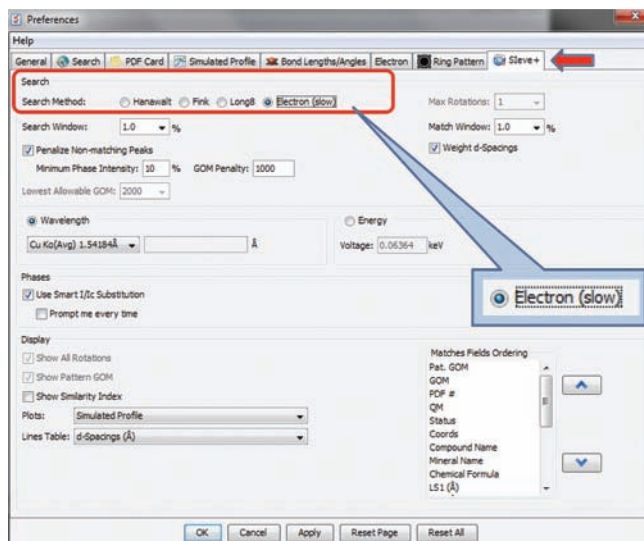


Figure 9: Display screen (in PREFERENCES) where 1-D electron based powder patterns can be simulated.

## ZONE AXIS AUTO-INDEXING

Recently, an algorithm to identify the zone axis in a user's electron spot pattern has been under development. This new electron diffraction tool extracts spot positions (hkl's) from the ED spot pattern, and performs automated indexing to determine the zone axis (Figure 10). The zone axis provides the user with information about the orientation of a material/crystal. Once the zone axis is defined, the user can simulate new patterns for other zone axes. The user may also import data to perform comparisons between possible phase matches. This new auto-indexing feature has been added into the suite of electron diffraction tools for the PDF-4+ 2014 release, with development continuing for future enhancement of electron diffraction based identification and characterization.

PDF-4+ offers a variety of algorithms and simulation options that allow users to analyze electron diffraction data. Currently, its data mining capabilities provide extensive filtering options to enhance the identification process, and the PDF-4+ search/match engine, Sieve+, enables PDF users to carry out vital search/match analyses using ED data. PDF-4+ electron diffraction tools streamline comparisons and contrasts of X-ray and electron diffraction data in one place. In many cases, this provides a unique capability to analyze the most difficult problems.

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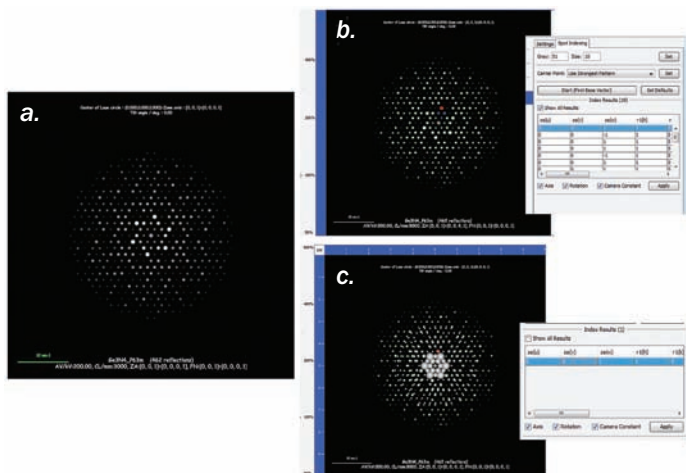


Figure 10: Various displays as seen when performing zone axis auto-indexing. Screen (a) shows the user's imported data, (b) shows the location of the central spot for analysis (red) and the list of possible choices for zone indexing (box on the right), and (c) shows the simulated data points (based on the indexed zone axis; box on the right) compared to the user's original data.

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