Example 13.5: Ni2Si

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Data	Topic	Level
Electron diffraction	Complete procedure from data reduction to structure refinement using the dynamical approach.	beginner

Input data

Data

Electron diffraction data was measured on a transmission electron microscope Philips CM120 with a precession device DigiStar. Accelerating voltage 120 kV.

Input files

directory "dp" with the set of experimental data (difraction patterns)
Ni2Si.pts: input file for PETS2.0
Ni2Si_reference.cif_pets: result of the data processing. This file is provided so that this example can be solved without the data processing part, and to warrant reproducibility of the solution and refinement part. File Ni2Si.cif_pets will be the output of the data reduction.
Ni2Si_reference_dyn.cif_pets: result of the data processing for dynamical refinement. This file is provided so that the dynamical approach can be used without the processing part, and to warrant the reproducibility of the refinement.

Keywords

References

For further information about the data processing in PETS2.0 see:
- L. Palatinus et al. Specifics of the data processing of precession electron diffraction tomography data and their implementation in the program PETS2.0. Acta Cryst. B 75: 512-522 (2019).
-PETS2.0 manual pets.fzu.cz/download/

For further information about the dynamical refinement and associated parameters:
- L. Palatinus et al. Structure refinement using precession electron diffraction tomography and dynamical diffraction: theory and implementation. Acta Cryst. A71: 235–244 (2015).
- L. Palatinus et al. Structure refinement using precession electron diffraction tomography and dynamical diffraction: tests on experimental data. Acta Crystallogr B71: 740–751 (2015)

Instructions

PART 1 - Data treatment in PETS2.0

1. Prepare (check) input file for PETS2.0

File Ni2Si.pts is the input for PETS2.0. The file was prepared by the software used for the data collection. However, you can prepare easily such input file for your own data.
Open file Ni2Si.pts in any plain-text editor (Notepad, Wordpad, Notepad++, Vim, …)
The beginning of the file gives you the experimental conditions as comments (#). These are irrelevant to PETS2.0. The important part starts here:

```
lambda 0.0335
Aperpixel 0.002210
phi 1.67
omega 12.50
pixelsize 0.01
noiseparameters 3.5 38
reflectionsize ??
bin 2

imagelist
dp\001.tif -25.00   0.00
dp\002.tif -24.00   0.00
dp\003.tif -23.00   0.00
...
Endimagelist
```

lambda: relativistic wavelength of the incident electrons in Angstroms (100 kV = 0.0370 Å, 120 kV = 0.0335 Å, 200 kV = 0.0251 Å, 300 kV = 0.0197 Å)
Aperpixel: Scale of the images given as the size of one pixel in reciprocal angstroms
phi: Precession angle in degrees used during the data collection. Can be zero, of course.
omega: orientation of the tilt axis of the sample holder with respect to the positive horizontal axis of the image. Must be estimated either by calibration, or by visual inspection of the series of images. Should be close to the correct value within +/-20 deg. Can be refined later in PETS2.0.
bin: Binning of the images prior to any treatment. Saves time, makes nicer pictures, and seems to make the whole analysis somewhat more robust. bin 2 is a good choice from our experience for most data sets. Important: All parameters in the input file that are given in pixels refer to the original, unbinned images. This concerns Aperpixel, center, reflectionsize, mask.
reflectionsize: Diameter of the spots on the images in pixels. Used for peak search and integration, should be large enough to encompass also the strong spots, but small enough to avoid overlaps of neighboring spots.
noiseparameters: two parameters for the determination of σ(I). The first value is Gγ, the second is ψ. The uncertainty on each pixel γ is then calculated as:
$σ2(p)=Gγp+ψ$
For more information see Waterman, D. & Evans, G. (2010). J. Appl. Cryst. 43, 1356-1371.
imagelist – endimagelist: Multiline keyword. Each line contains one image. The format is: name, alpha, beta. Name is the file name of the image (including possible relative or absolute path), alpha is the tilt angle, and beta is the possible second tilt value of the double tilt holder.
Reconstruction – endreconstruction: Multiline keyword to generate reciprocal space sections. Each line contains a specification of one layer in the reciprocal space that should be

reconstructed by PETS2.0. The format is: filename, base vector 1, base vector 2, origin vector, where each vector is given by three reciprocal-space coordinates. The first two vectors define the orientation of the plane, and the third vector defines the origin of the plane. The standard set is hk0, hk1, hk2, hk3, h0l, h1l, h2l, h3l, 0kl, 1kl, 2kl, 3kl. You can determine a specific set of sections later in the graphical interface or directly in the input file using the keywords "reconstruction" and "endreconstruction".

Save and close Ni2Si.pts

2. *Run PETS2.0 and repare files for indexation*

Start PETS2.0 by double clicking the executable
Go to File->Open in the upper menu
Open the file Ni2Si.pts
In the Main panel (Image data tab) you can view individual diffraction frames of Ni2Si dataset stored in the directory "dp" using the arrows at the bottom of the panel. The contrast of the images can be adjusted by changing the value in the Cutoff-box (inside the red retangle on the picture). The parameters of the input files are visible in the console and in the menus of the tabs "Parameters" and "Peak search". The arrows to the left of "Frame" (at the bottom controls of the Image data panel in the blue retangle) and the value next to it control the image that is shown. The radio buttons "raw" and "processed" show you the raw diffraction pattern as recorded and the processed frame (some background correction etc.), respectively.
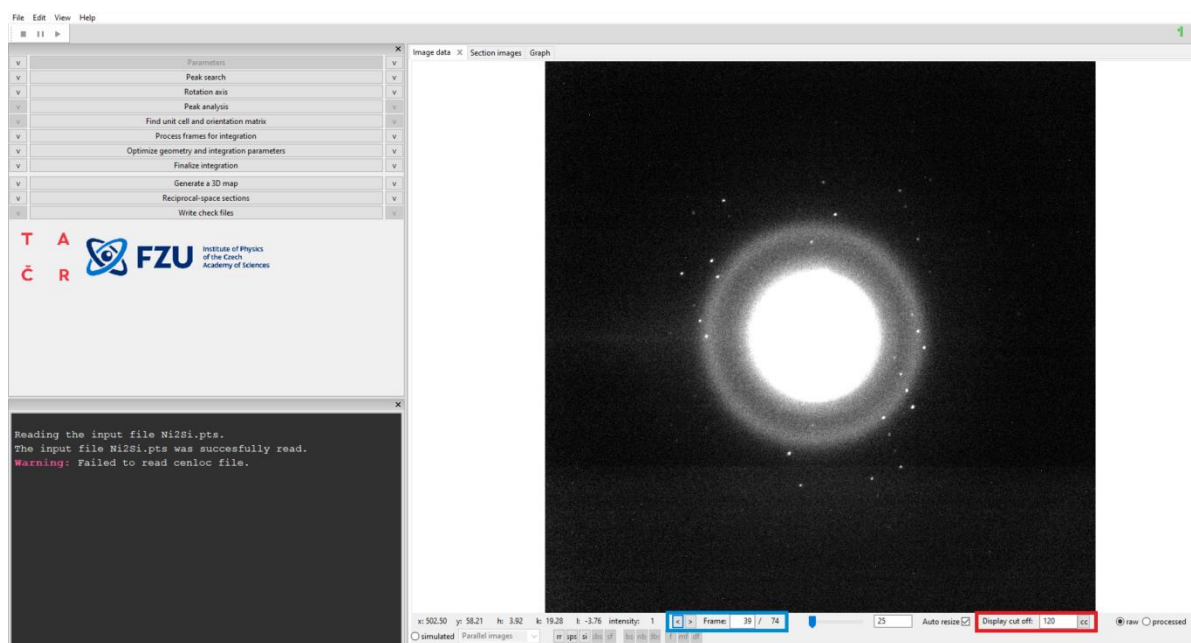
Data reduction steps are run by clicking the respective action button. The action options can be modified in the panel which opens after clicking the small arrow next to the action button. The "Parameters" action button (first in the list of action buttons) is gray because there is no associated action. Note that you can adapt the size of the different window components (e.g. make the plot window more narrow or wider) and decouple the action bar or the console from the main window.
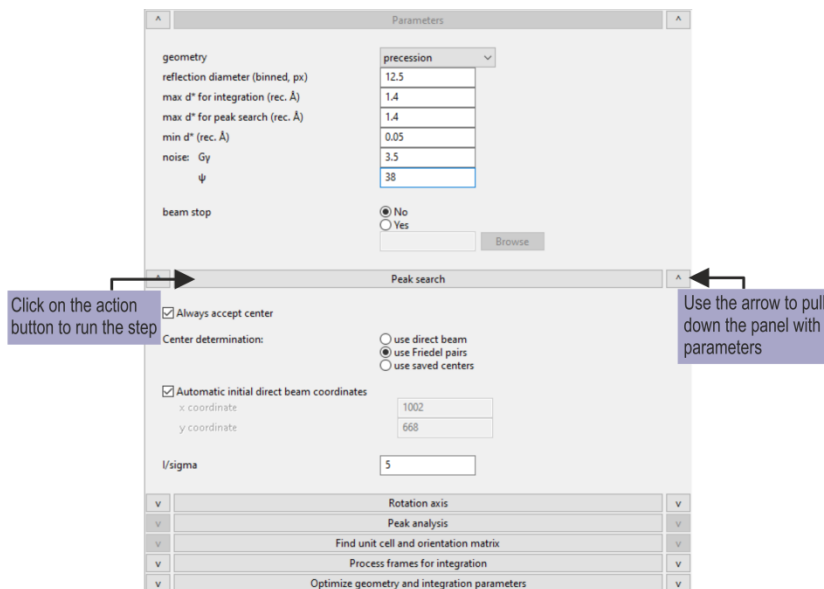
3. Peak search

Click on the arrows (v) next to "Parameters" and "Peak search" to check the following options
Parameters - reflection diameter 12.5
In our input file .pts, we defined "reflectionsize 25" and "bin 2", hence the binned reflection diameter is 12.5 pixel. The default parameters used for min d* of 0.05 Å-1 is OK. Here it means that any reflection/peak that is less than 0.05*Aperpixel pixel away from the center is ignored. beam stop No;
*in Peak search parameters - Always accept center checked, use Friedel pairs*
Click the action button "Peak search" to start the procedure. The progress of the Peak search can be followed in the Image data panel.
In the main panel, diffraction images are sequentially displayed with circles at points found by PETS2.0 as significant peaks (above I/sigma specified in the Peak search parameters). Crosses appear in the if the peak's Friedel pair was detected
At the end, the deviation of the x- (red) and y-coordinates (green) of the estimated direct-beam position is displayed in the Main panel in pixels (tab Graph):
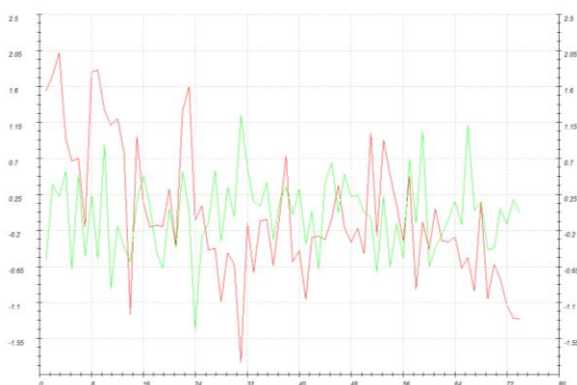
In the console, for each frame the estimated position of the primary beam, the number of Friedel pairs and the number of significant peaks are given. The process ends with the following message in the console:

```
Minimum, average and maximum position of the primary beam:  495.72   497.57
499.59 |    504.81  506.26  499.59
Finished reading 3403 peaks from the file Ni2Si.rpl.
```

4. Rotation axis

The next step after peak hunting is the refinement of the angle between the horizontal axis (x axis of the diffraction image) and the projection of the rotation axis. This angle depends on the experimental setup, and needs to be refined for each data set.

Click the action button "Rotation axis" to start the procedure.
Switch to the tab "Cylindric projection" in the main panel to see the progress of the refinement

In the main panel "Cylindric projection", a cylindrical projection of the difference space of the extracted peak positions is displayed. A correct azimuthal angle refinement results in the image containing sharp peaks aligned on sinusoidal curves. This step provides a first estimation of data quality. At the end of the refinement the omega angle is 12.9219 degree.
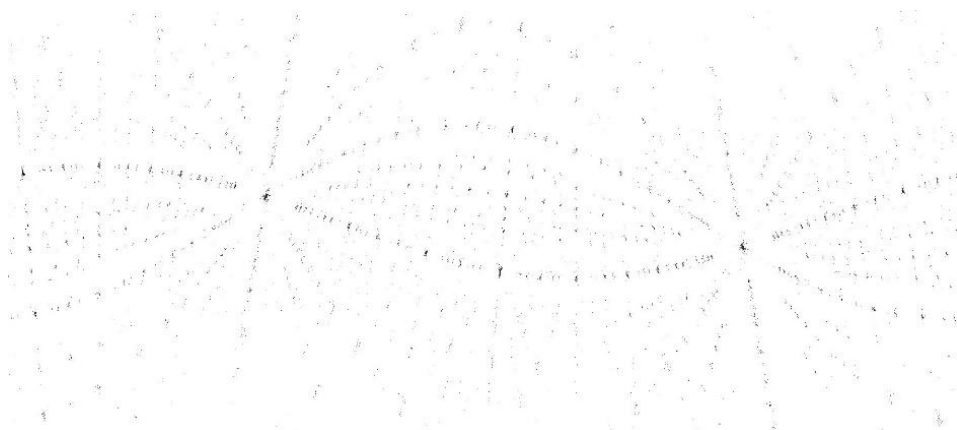
5. Peak *analysis*

Click the action button "Peak analysis".

PETS2.0 starts the analysis of the peak positions obtained in the peak hunting procedure. In the first step, distance distribution between peaks in the image plane is analyzed. A sorted plot of inter-peak distances is displayed in the Processing tab (red curve) with its derivative (green):
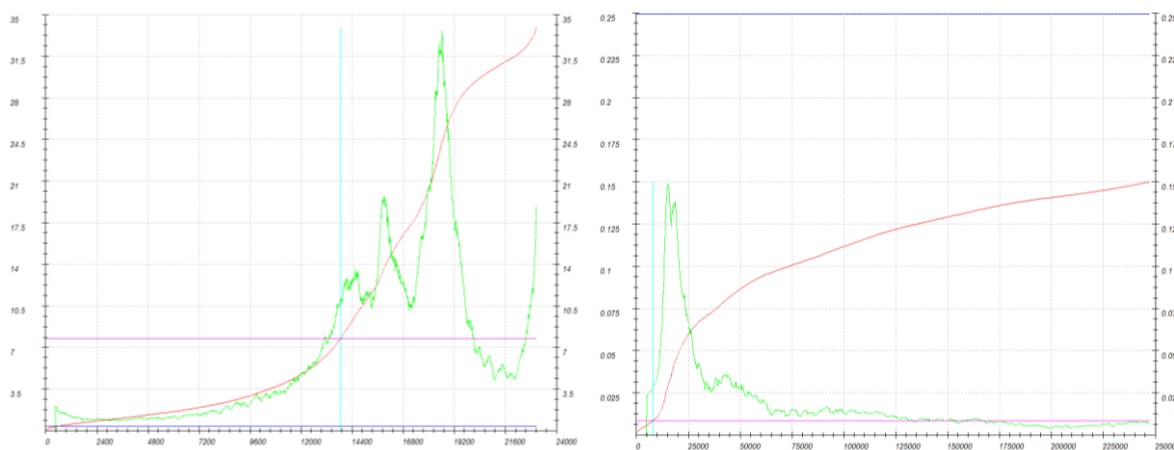
Click "Peak analysis (continue)"

For good quality data set, the red curve contains distinct steps, and, consequently, the green curve has distinct peaks.

Click "Peak analysis (continue)"

In the next step, an auto convolution of the diffraction pattern (difference space) is analyzed, and the groups of peaks in the auto convolution (clusters) are replaced by the cluster centers. Again, a distance distribution is displayed (right screenshot), and clear-cut steps on the curve indicate a well-defined lattice.

Now the first step is finished and PETS2.0 prepared the files needed for unit cell indexing.
*At this end of this step, you should get the following message in the console:*

```
Thresholds for considering two peaks the same: 0.0084 reciprocal angstroms.
 12844 peaks merged into 10314 clusters, 750 with more than one peak.
Peak analysis successfully finished.
```

6. *Find unit cell and orientation matrix*

Click "Find unit cell and orientation matrix"
New set of buttons with parameters opens as well as a new tab "3D panel" that displays the data used for indexing. The data can be enlarged by scrolling the wheel of your mouse, and rotated by holding the left mouse button.
Select "Data used for indexing: xyz" for the indexing
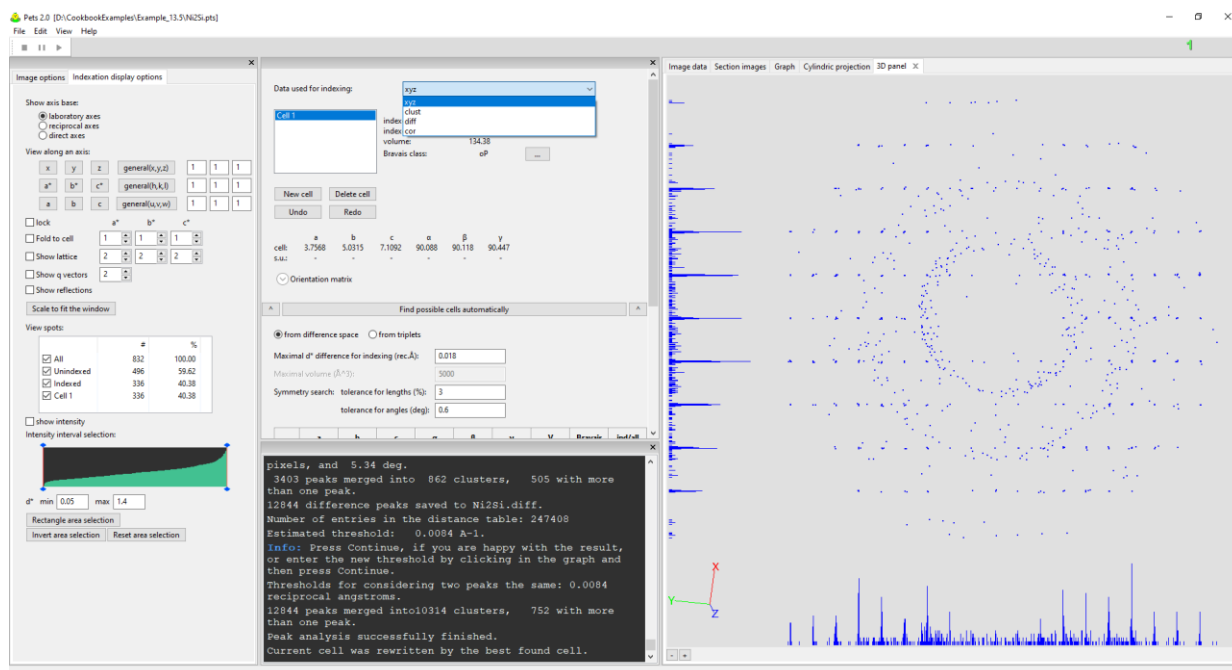
Open the menu below "Find possible cells automatically" by clicking on the arrow next to the action button.
You can see the parameters used in the procedure as wells as the possible unit cells found later by PETS2.0.
Check that the selected option is "from difference space" and keep the other parameters default.
Run "Find possible cells automatically"
Only one unit cell is found (see below) and the initial unit cell 10 10 10 90 90 90 is replaced by the new one a = 3.7569 Å, b = 5.0315 Å, c = 7.1091 Å, α = 90.089°, β = 90.119°, γ = 90.452°
Make sure that you use the Ni2Si.xyz file in "Data used for indexing" for the refinement
Click on the arrow next to "Refine cell" to access the options
Keep the default "Refine UB + cell",
 Select "orthorhombic" in symmetry
Click "Refine Cell"
The refined unit cell parameters are a = 3.7484 Å, b = 5.0173 Å, c = 7.1075 Å, α, β, and γ angles were set to 90.
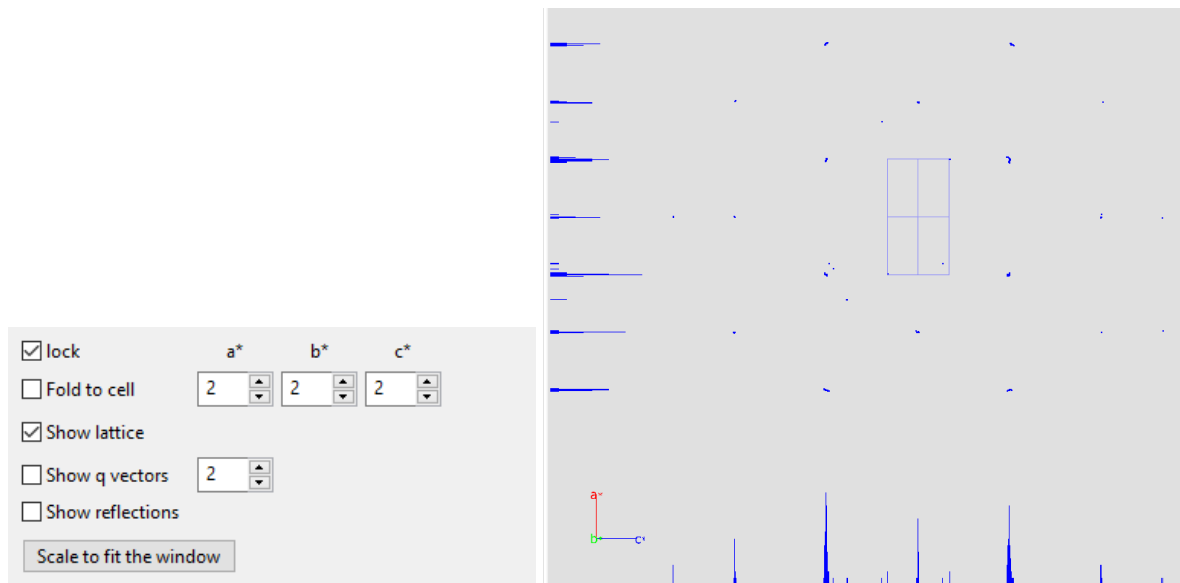*You might already guess the space group checking the extinctions here.*
Select "Data used for indexing: cor"

On the "Indexation display options", select "View along an axis: a"
Use "Rectangle area selection" to select the inner column in the "3D panel"
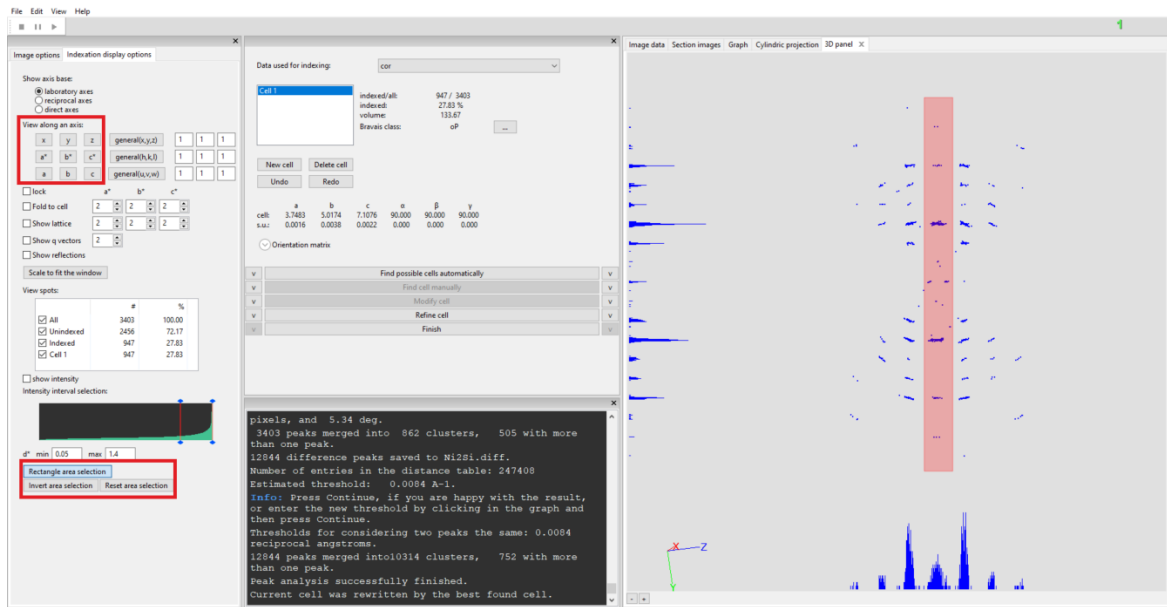
On the "Indexation display options", select "View along an axis: b"
Enable "Show lattice"and use 2x2x2

You will see that there are systematic extinctions h+l=2n+1 along h0l. If you reset the area selection and select the column corresponding to h1l, the absent reflections on h0l are present in h1l.This extinction condition corresponds to an n-glide plane perpendicular to the b-axis. If you reset the area selection and repeat the selection along the a-axis to see 0kl and 1kl, you will see that there are no further systematic absences. The space group symbol is Pmn-, which is non-standard Pnm- with our settings. The unit cell found by PETS2.0 might lead to a non-standard setting because it arranges the cell parameters based on the length. We will go back to the indexing and change the setting so we can have the standard one.

Select "Data used for indexing: xyz" for the indexing
In "Modify cell", use the "Transformation matrix" according to the image below:

Click on the arrow next to "Refine cell" to access the options
Keep the default "Refine UB + cell",
 Select "orthorhombic" in symmetry
Click "Refine Cell"
The refined unit cell parameters are a = 5.0173 Å, b = 3.7484 Å, c = 7.1075 Å, α, β, and γ angles are 90.
Leave the indexing panel by clicking "Finish"

7. Create reciprocal-space sections

PETS2.0 scans all images and reconstructs sections through reciprocal space at planes hk0, hk1, hk2, h0l, h1l, h2l, 0kl, 1kl, 2kl (default set). These sections are used as an aid to determine the space group. Sections are saved in the directory sections as TIF-files. In the parameters of the Reciprocal-space sections, you can change the size of pixels in the reconstruction images and the thickness of the slab that is projected into the section in Å^{-1}. Too small numbers may result in "holey" reconstructions. We will proceed with the default values.
Click "Reciprocal-space sections", OK (use the standard set)
You can visualize the sections in the tab "Section images" of the main panel and choose the section as shown on the picture below.
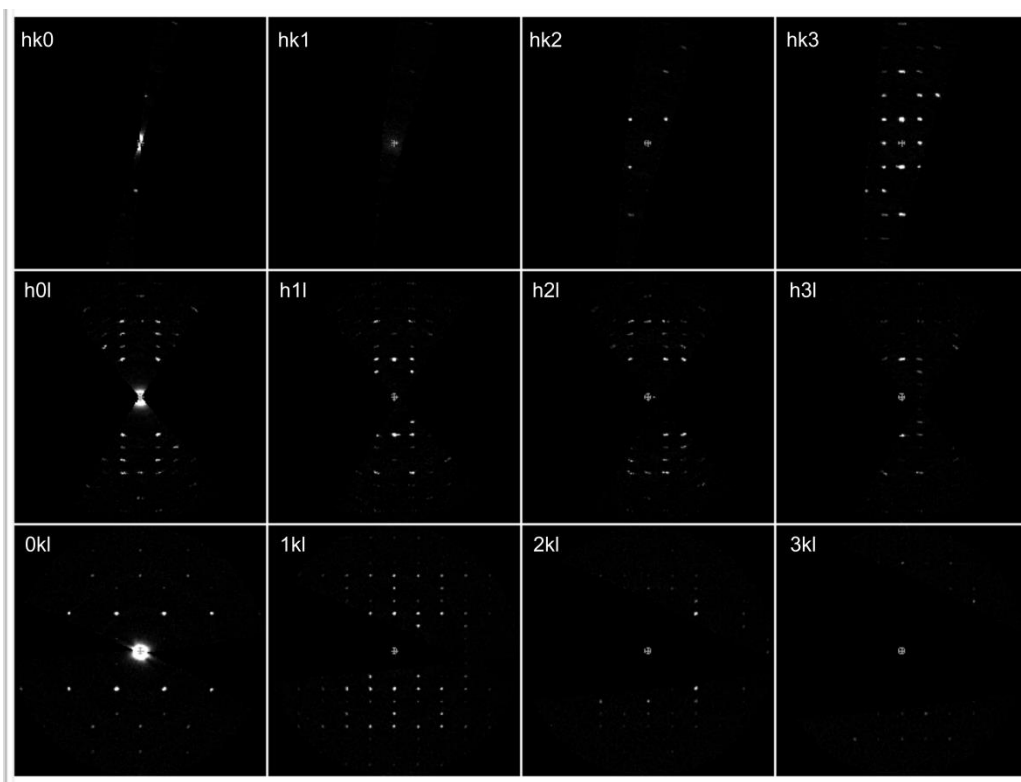
From electron diffraction, it is often difficult to select the correct space group. In particular, presence or absence of the glides and screw axes is hard to asses, knowing that kinematically absent reflections may have significant intensities due to dynamical diffraction effect. In this case the missing wedge leaves almost no information in the hk0 reciprocal-space section (see section hk0), and we cannot estimate the presence of absence of a glide plane in this direction. In the other directions the reconstructed reciprocal space sections can be very helpful in estimating the symmetry. In Section 6 (Find unit cell and orientation matrix), we found that the diffraction symbol is Pnm-, in the standard setting, and this can be confirmed in the reciprocal space sections. This corresponds to the space group Pnmm, Pnma, or their non-centrosymmetric subgroups.

8. Integrate intensities

(a) Process frames for integration

Start the procedure by clicking the action button "Process frames for integration" with all parameters of this section set by default: *the box "shift integration box maximum intensity" is selected and the parameters RC width and mosaicity stay at their default values.*
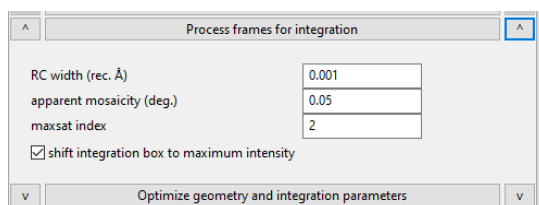

In the Image data tab, diffraction frames with circles drawn at positions predicted from the orientation matrix are displayed. A green circle means that a peak with I>3σ(I) was found. A brown diamond means that a reflection is expected on upcoming frame(s) or was present on

previous frame(s). To access the resolution of you data, you can also display the resolution rings by selecting the option "rr" in the Image data panel (see below).
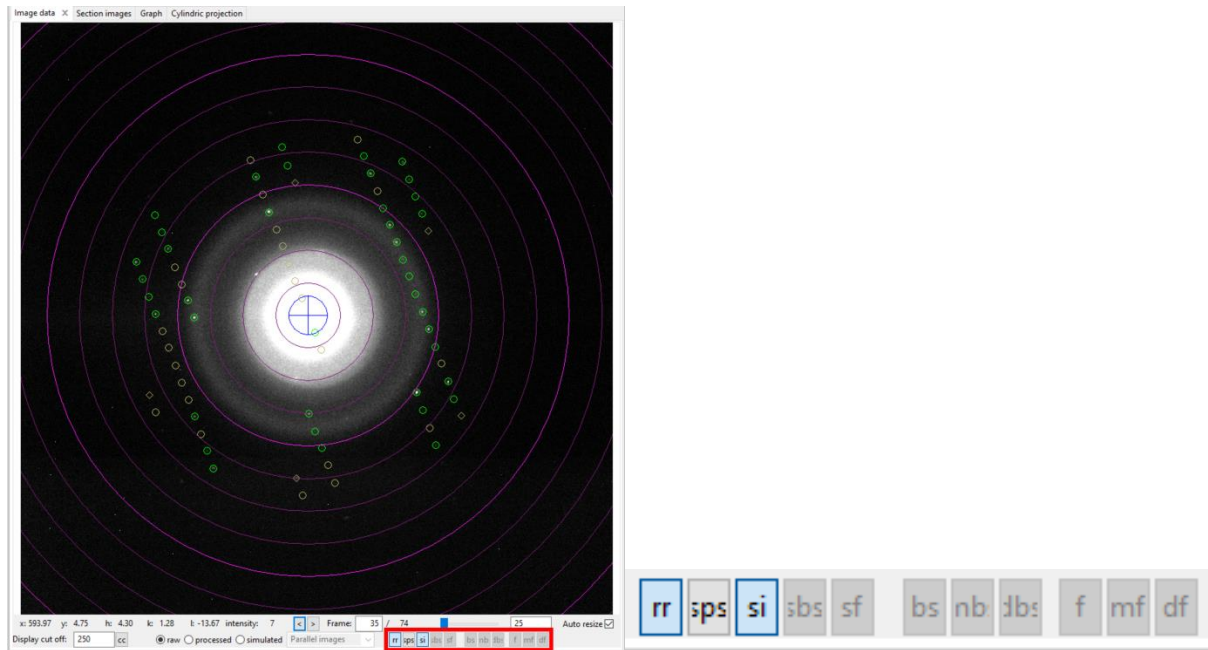
At the end of the integration process, in the Graph tab, the camel plots (rocking curves profiles) are displayed (data - blue, fit - red). On the horizontal axis of the plot there is the excitation error of reflections. Each curve shows an average rocking curve of reflections in one resolution shell, starting from 0.2-0.3 $Å^{-1}$ in steps of 0.1 $Å^{-1}$. Notice the splitting of the humps with increasing diffraction angle due to precession. By changing the parameters you can observe their effect on the curve. The reflections considered for the visualization of the camel plots are affected by the I/σ(I) threshold.
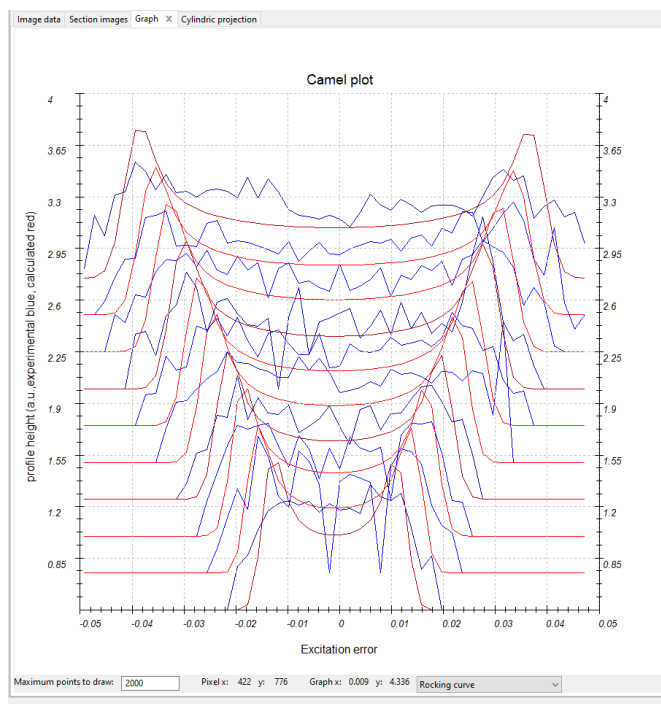
(b) Optimize geometry and integration parameters

Go to the options of "Optimize geometry and integration parameters"
Check that the refinement using rocking curve profile is checked as well as the RC width and mosaicity parameters.
Start the fitting procedure by clicking on the action button.
The fit results in the RC width of ~0.0018 Å^{-1}and mosaicity ~0.294 deg. The fit is slightly improved as visible in the plot on the Graph tab:
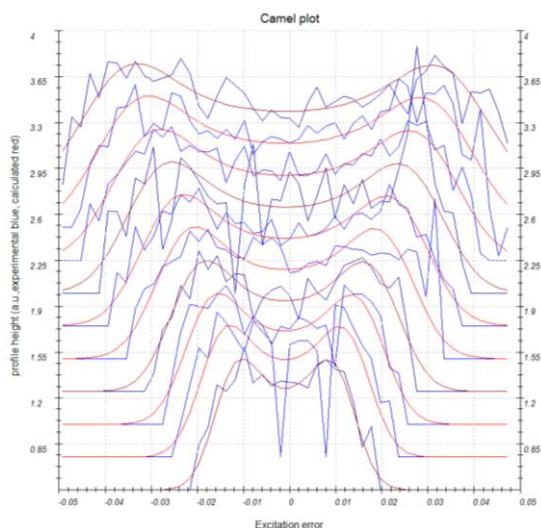
If the data or the fit would not seem to converge to the baseline at the x-limits on the graph it is necessary to rerun the previous step (Process frames for integration) with the newly refined parameters, otherwise it is possible to continue directly to next step. Here, we will run the Process frames for integration once more.
Run "Process frames for integration"
The resulting camel lot looks like this:
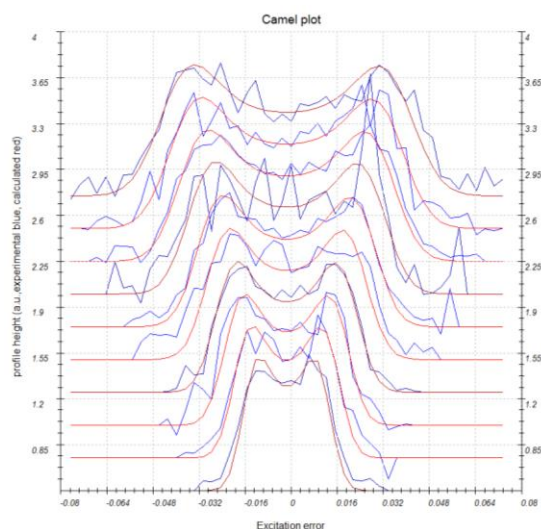
(c) Finalize integration

Open the parameters for "Finalize integration"
Check that both "kinematical" and "dynamical" integration are checked
Select the radio button "integrate profile" next to "intensity estimation".

Default value for intensity estimation is "integrate profile", which is the more robust technique and works well even for poorer data. However, for good data the "fit profile" option gives better results.

Run the procedure by clicking the action button.

After the integration, PETS2.0 shows the table of Rint values in the console for the different Laue classes. For the Laue class mmm the Rint of observed reflections is 12.36 % for 115/154 observed /all unique reflections.

Select the radio button "fit profile" next to "intensity estimation".

Run the procedure by clicking the action button "Finalize integration".

The table of Rint values in the console for the different Laue classes shows Rint of observed reflections 10.78 % for 109/147 observed /all unique reflections for the Laue class mmm.
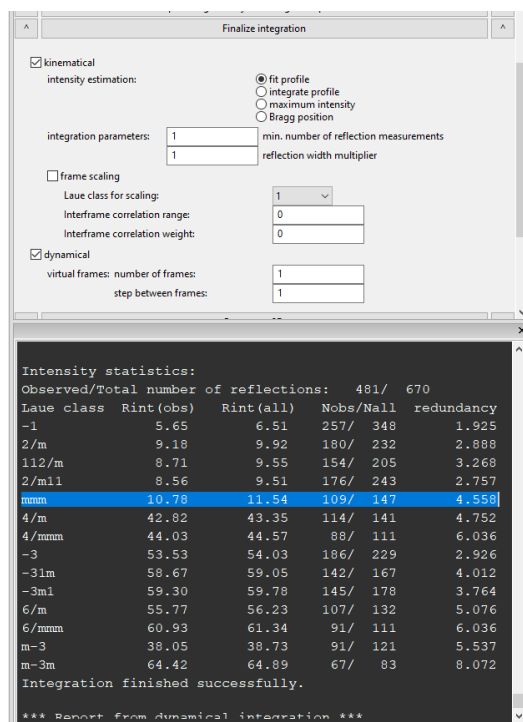
Optionally, it is possible to include frame scaling which optimizes frame scales to reach the best Rint for the given Laue class. In our case, we use Laue symmetry mmm.

Check the check box frame scaling, select Laue class mmm
Write 2 for "Interframe correlation range:"
Write 1 for "Interframe correlation weight:"
Run the procedure again by clicking the action button "Finalize integration".

The frame scaling may take some time. Rint lowers from 10.78% to 8.47% for Laue class mmm. It is possible to view the scale factors for individual frames on the tab Graph:
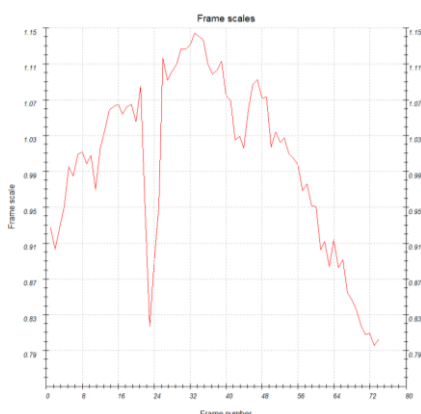
When the integration finishes, files Ni2Si.cif_pets and Ni2Si_dyn.cif_pets are created. Ni2Si.cif_pets is used to solve the structure and for kinematical refinement, while Ni2Si_dyn.cif_pets is used for dynamical refinement.

PART 2 - Structure solution and kinematical refinement in Jana2020

1. Create new structure

Important! The data-processing procedure is almost never perfectly reproducible. Small differences in the indexing and cell refinement procedure may result in small differences of integrated intensities. If you want to be sure that you can reproduce the following part of the tutorial, do not use the file Ni2Si.cif_pets that you have just created, but use the file Ni2Si_reference.cif_pets provided with the example files. Using your own cif_pets file is also possible, but your results may slightly differ from the results described in this tutorial.

Start Jana2020
In the Main menu bar, use "Structure → New" and open new structure "Ni2Si" in the directory of the Example 13.5

2. Import Wizard

[On the screen: Specify type of the file to be imported]
Select "Single crystal: known diffractometer formats"; NEXT
[On the screen: Data reduction file from]
Select "Pets electron diffractometer"
For "Input file", browse the file Ni2Si_reference.cif_pets (or your file)
This file produced by PETS2.0 contains all important information, including the list of intensities.
Check if "Make the reflection file for dynamical refinement" is disabled
NEXT
[On the screen: Complete/correct experimental parameters]
Leave all settings unchanged; NEXT
Leave all settings unchanged; NEXT
The program reads 670 reflections from the file
OK
For absorption correction select "None or done before importing" (default); NEXT
FINISH; OK to accept the data set
You just read in the cell parameters and the intensity list. If you do not have the data in the cif_pets format, you can read them in from a general hkl file using the option "reflection file

corrected for LP and absorption" at the beginning of the import wizard. You then have to input the radiation type, wavelength and cell parameters by hand.

3. Symmetry Wizard

NEXT to close the information window and start the symmetry wizard.
Symmetry Wizard can be started separately by expanding "Reflection file" in the Command tree and selecting "Make space group test". The default settings are prepared for x-ray diffraction. For electron diffraction we have to increase the tolerances.
Change the tolerances according to this screenshot:

Tolerances for crystal system recognition:

Original cell parameters: 5.017 3.748 7.108 90.00 90.00 90.00

Maximal deviation for cell lengths in Ang:	0.1
Maximal deviation for cell angles in degs:	0.2
Maximal deviation for modulation vector:	0.01

Tolerances for space group recognition:

Maximal ave(I/sig(I)) for systematic extinctions induced by cell centering:	5
Maximal ave(I/sig(I)) for systematic extinctions induced by other symmetry	25

☑ Search for higher symmetrical supercell (recommended)

☐ Introduce twin laws in case of subgroups

☑ Use old twin matrices in testing

NEXT
[On the screen: Select Laue point group]

Select Laue point group

Crystal system	Point group	Rint(obs/all)	#averaged(obs/all)	Redundancy
Triclinic	-1	5.59/6.49	258/348	1.925
Monoclinic-setting "a"	2/m	7.35/8.35	176/243	2.757
Monoclinic-setting "b"	2/m	7.14/7.89	178/232	2.888
Monoclinic-setting "c"	2/m	7.49/8.35	153/205	3.268
Orthorhombic	mmm	8.47/9.25	108/147	4.558

Select orthorhombic mmm; NEXT
The internal R-values would be rather high for x-rays, but are low for electron diffraction data.
[On the screen: Select cell centering]
Select primitive unit cell; NEXT
[On the screen: Select space group]
The window shows possible space groups.
The list of the strongest reflections contradicting the selected space group can be displayed by "Details" button.
In Section 7 in Part 1 (Create reciprocal-space sections), we saw that the diffraction symbol is Pnm-, corresponding to the space group Pnmm or Pnma. We will choose Pnma simply because it is a very frequent space group.

Select space group

Characteristics for systematically absent reflections

Space group	#obs/#all	ave(I/sig(I))	Figure of merit
P212121	0/10	--------/0.426	0.09855
P21221	0/10	--------/0.426	0.09855
P22121	0/10	--------/0.426	0.09855
P2221	0/10	--------/0.426	0.09855
Pnmm	15/69	4.300/1.511	0.30379
Pnm21	15/69	4.300/1.511	0.30379
Pn21m	15/69	4.300/1.511	0.30379
Pmmm	0/0	--------/--------	1.00000
Pm2m	0/0	--------/--------	1.00000
P2mm	0/0	--------/--------	1.00000
Pmm2	0/0	--------/--------	1.00000
P21212	0/0	--------/--------	1.00000
P2212	0/0	--------/--------	1.00000
P2122	0/0	--------/--------	1.00000
P222	0/0	--------/--------	1.00000
Pnma	20/75	6.455/2.252	1.08107
Pn21a	20/75	6.455/2.252	1.08107
Pnmn	18/73	10.586/3.155	4.34345
Pn2n	18/73	10.586/3.155	4.34345
Pnmb	21/75	10.836/3.564	5.19698
Pn2b	21/75	10.836/3.564	5.19698
Pnna	42/111	21.758/8.648	38.73916
Pnnm	37/105	22.952/8.527	44.40517
Pnn2	37/105	22.952/8.527	44.40517
Pnnb	43/111	23.541/9.535	50.32058
Pnnn	40/109	24.381/9.371	53.57935
Pm2a	5/6	12.923/10.769	202.65558
P21ma	5/6	12.923/10.769	202.65558
Pmma	5/6	12.923/10.769	202.65558

Important: Note that a wrong choice of the space group is not detrimental for the structure solution, because we will use Superflip, which will make additional check for the most probable space group.

Select Pnma; NEXT
Accept the space group transformed into the original cell; FINISH
Symmetry is saved in file Ni2Si.m50.

4. Creating refinement reflection file

In this step program creates file Ni2Si.m90 containing the data set merged by symmetry and with discarded forbidden reflections. Ni2Si.m90 will be used for refinement.
Proceed with the wizard, leaving all settings default. NEXT
The Import statistics-obs/all gives 479/670 reflections read from input file, 459/595 reflections written to output file and 20/75 reflections rejected as symmetrically extinct.
OK,
OK again; NEXT
The Rint(obs/all) = 8.37/8.87 for 99/122 independent reflections.
OK; FINISH

5. Structure Solution Wizard

OK to close the information window and start the solution wizard
[On the screen: window of Structure solution]
Structure solution wizard can be executed separately from the Command tree. Jana2020 does not contain solution procedures, it calls external programs. By default, Jana uses Superflip (using charge flipping as the solution method). Superflip is distributed with Jana2020.

In "Formula" textbox type just the list of chemical elements: Ni Si
Superflip does not need information about the chemical composition but it will be required for the peak assignment based on the electrostatic potential map calculated by Superflip. If

Jana2020 is used for peak assignment, a list of expected chemical elements is sufficient. If you know the expected formula, you may enter it and use EDMA for peak search (at the bottom of the window). If the formula is correct, EDMA often gives cleaner and more complete interpretations of the solution.

Check if CF (charge-flipping) is selected for the "iteration scheme".
Leave other settings default; "Run Superflip".

Window of Superflip appears with iteration running. After ten runs, a window with Superflip log file is displayed. The number will be close to the ones in the table below, but not identical, as Superlip starts each time from a different random starting point.

The space group proposed by Superflip is the same as our initial guess. The space-group selection is confirmed.

Click "Draw 3d map"
VESTA opens with a 3d electrostatic potential shown as isosurfaces, and an atomic model overlaid (in the default settings the shown isosurface level corresponds to the 2σ level based on the statistics of the 3D electrostatic potential map). Rotate the view to inspect the structure. Do you think it is complete?

You should come to the conclusion that the model is correct, correct elements were assigned to all maxima.

Close VESTA (do not save the file)

You may now check the structure to convince yourself that the structure is complete and chemically reasonable.

Click "Draw structure"

Now the structure is displayed using the plotting program of Jana2020 called JanaDraw

In the lower toolbar, press Fill unit cell

Close JanaDraw

Press "Accept last solution"

The program creates file Ni2Si.m40 with atoms of the structure model

6. Kinematical refinement

[On the screen: basic window of Jana]

Right-click the quick button "Run Refinement".

[On the screen: refinement options]

Change the number of cycles to 100;

OK

Choose "Yes+start"

Refinement converges with Robs value about 12.47%. At this moment you may check the refinement listing with a lot of information about the refinement procedure and results. As you can see, the refinement is already very good for electron diffraction. R values are low, all the ADPs were positive. Refinement of anisotropic atomic displacements would further decrease the R-value (Robs=11.59%), but the ratio reflections per parameter is already low.

The resulting structure

In the Command tree, expand "Draw structure" and start "Vesta"
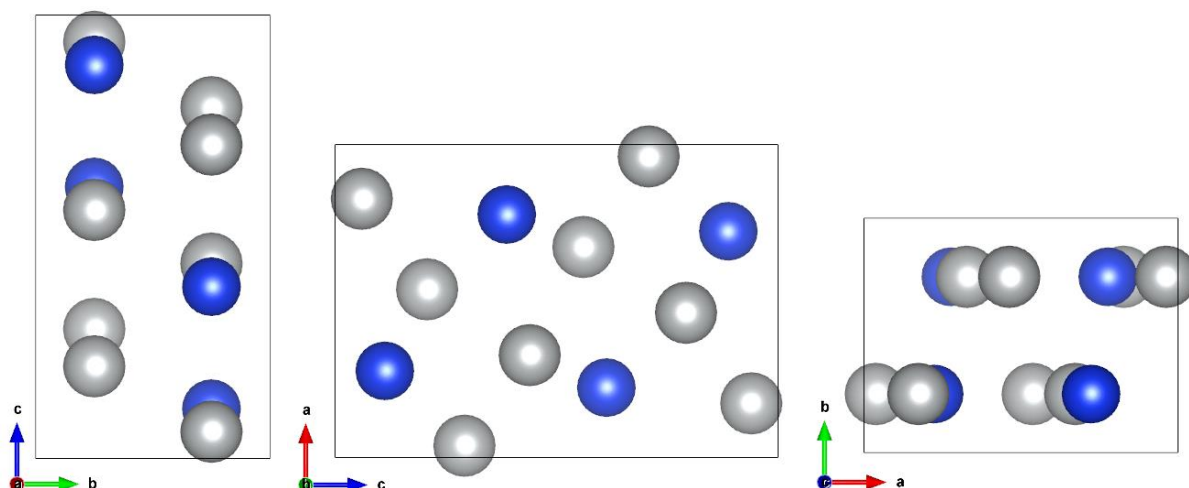
PART 3 - Dynamical refinement in Jana2020

1.   *Preparation of reflection file for the dynamical refinement*

During the integration procedure in PETS2.0, an input file for dynamical refinement was created. The integration for the dynamical refinement considers each diffraction image separately. The result is a list with the hkl indices, the measured intensity with the respective σ(I) and the number of the frame in which the intensity is present. PETS2.0 creates the file Ni2Si_dyn.cif_pets with all this information, which will be used in Jana2020. Note that the same hkl reflection might be present in several frames, with different intensity due to the multiple scattering relative to different crystal orientation and thickness at this condition.

In the main menu bar, use "Structure → Save as" and save a new structure Ni2Si_dyn for the dynamical refinement
YES to Continue with the new structure.
This step saves the structure you refined kinematically as a new job, which will be used for dynamical refinement.

In Command Tree, expand „Reflection file" and start „Import-modify reflection file"
Click „Reimport"
Important! Here again, if you want to be sure that you can exactly reproduce the following part of the tutorial, use the file Ni2Si_start_dyn.cif_pets provided with the example files. Rename this file to Ni2Si_dyn.cif_pets.
Select the Ni2Si_reference_dyn.cif_pets as the new reflection file
Enable "Make the reflection file for dynamical refinement"
NEXT, NEXT, NEXT.
Now, Jana reads 4166 reflections from all the frames.
OK, NEXT, FINISH, OK
[On the screen: Processing refinement reflection file…]
Check that "Make the reflection file for dynamical refinement" is enabled, NEXT
*Notice that 2334 reflections are observed out of 4166 reflections read. From these, 80 were rejected because they violate the systematic extinctions. You can find this information in the file *.rre (Edit/View→Reflection report).*
OK, OK, FINISH
[On the screen: basic window of Jana]
In Commads Tree, expand "Edit structure parameters" and start "Edit parameters for electron diffraction"
[On the screen: Parameters for electron diffraction data]
Check that "Use dynamic approach" is enabled

2. *Dynamical refinement parameters*

[On the screen: Parameters for electron diffraction data]
The default values for the selection of the intensities are: g(max)=1.6, maximal excitation error Sg(matrix)=0.01, maximal excitation error Sg(refine)=0.1 and Rsg=0.4. These values are "safe" to produce good quality refinement, but they are not optimized for speed. You can set the number of threads to the number of cores on your processor to speed up the calculation.

3. Sample thickness

The sample thickness is an important parameter of the dynamical refinement. The default value is 400 Å.

Enable "Except of scale, optimize also: Thickness"

Click "Run optimizations"

Dyngo calculates wR-factor for the thickness varying from 0 to 2000 Å.

"Show thickness plots". Check the variation of the thickness of the crystal for the frames. Quit.

Most frames have well defined wR-factor minima for one thickness. However, few frames either have poorly defined minimum or the minimum is at a very different thickness. You can check the wR(all) going through the zones in the main window of "parameters for electron diffraction data" or while viewing the graphs.
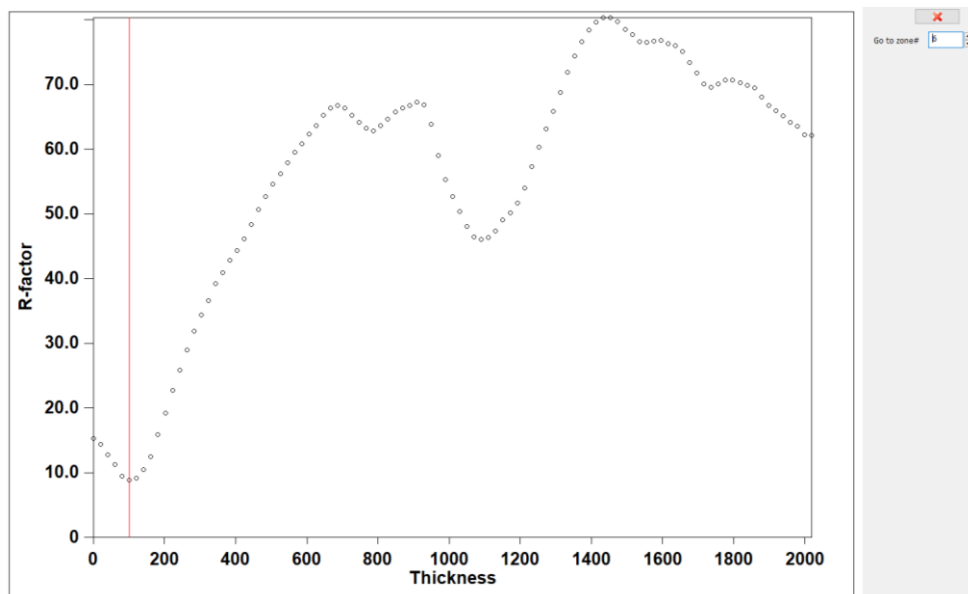
For example frames 13, 14, 25, 28, 33, 34 have the minimum at very low thickness (0 Å). Frames 31 and 65 have a plateau for thickness between 0 and 400 Å. Notice also that the maximum R-factor is around 55 % for the last frame. You can check the wR(all) going through the zones in the main window of "parameters for electron diffraction data" or while seeing the graphs. The different thickness is usually not a problem, but if you encounter a very aberrant frame (or a sequence of consecutive frames), it may be removed from the refinement by deselecting it in the list of zones for refinement.

4. Defining all the zones with the same thickness

 "Define zones of equal thickness". "Select all", OK

The crystal is a nanowire and the thickness is mostly constant along the sample. However, the thickness changes according to the tilt of the sample, because the nanowire lies roughly perpendicular to the tilting axis of the sample holder. Then, it is possible to define the same thickness for all the frames but still refine the variations of the thickness according to the tilting of the sample.

There are two ways of defining the same thickness for all the frames. You can either estimate an average thickness and type it for all frames, or run 0 refinement cycle and average the thickness. We will use the first option here, because it is faster and easier.

Looking at the thickness plots we have seen that the thickness is most of the times very low. 100Å seems to be a good estimate.

Click "Select zones for editing".

Click "Select all"

OK

Click in the field EDThick EDThick 60 and then click the yellow button "Unlock".

Type 100 in the field EDThick

Now we need to rerun the optimization of frame scales, as changing of the thickness also requires adapting the scale.

Click "Run optimizations"

In a few seconds the optimization is finished.

Click OK and YES to return to the main screen of Jana2020.

Right-click the quick button" Refine"

On the tab "Basic" check that the number of refinement cycles is set to 100.

OK; "Yes+Start".

The refinement runs 10 cycles, each cycle takes less than a minute (depending on your computer). You can see the details of the refinement in the file Ni2Si.ref (Command Tree → Refinement → View refinement listing). The refinement converges with R=9.51%. However, the ratio reflections per parameter refined is around 8 (689/84) and it is advisable to increase it. It can be done by increasing RSg (selecting more reflections for the refinement).
Another important thing to check is the number of observed reflections per zone. Also in the Ni2Si.ref file, you can see "R-factors for individual zones", where the list of zones with the respective R-factors and number of reflections is shown.

5. *Optimization of orientation*

Save a new structure "Structure→Save as→ Ni2Si_dyn_ori"; OK; YES
In Commads Tree, expand "Edit structure parameters" and start "Edit parameters for electron diffraction"
 [On the screen: Parameters for electron diffraction data]
Change the "Limit on RSg" to 0.75
Enable "Except of scale, optimize also: Orientation"
Click "Run optimizations"
[On the screen: Intensity calculation for optimizing of orientation].
The orientation of the crystal during the measurement plays an important role in the dynamical scatterings. EDTheta is the amplitude of the correction of the frame orientation. It should be a small number, a few tenths of a degree at most. Typically, a deviation of 0.5° is already considered high.
Using "Zone #" box, check the resulting EDTheta for the individual frames.
The frames with high misorientation are: 1, 2, 3, 8, 9, 10, 17, 18, 19, 22, 24, 28, 33, 34, 36, 43, 44, 52, 61, 63, 70, 72, 73, and 74. If you check these frames, most have few reflections (because the unit cell is small), the reflections are mostly weak (the nanowire is very thin) and some frames have 1 or 2 peaks visibly stronger than the rest. These stronger reflections dominate the optimization and hamper the correct estimative of orientation. Frames with EDTheta larger than 0.5° will be removed from the refinement.
Note that such a high number of misoriented frames is unusual and happens only in this case of very weak data on a structure with a small unit cell. Typically, only a very small number of frames, sometimes none, need to be removed in this step. In the present case, an alternative approach is not to apply frame orientation at all. In this example, the orientation is applied nevertheless for demonstration purposes.

"Select zones for refinement"
Disable the frames 1, 2, 3, 8, 9, 10, 17, 18, 19, 22, 24, 28, 33, 34, 36, 43, 44, 52, 61, 63, 70, 72, 73, and 74 (Ctrl + left mouse button).

Select zones for refinement

Zone#1	Zone#23	Zone#45	Zone#67
Zone#2	Zone#24	Zone#46	Zone#68
Zone#3	Zone#25	Zone#47	Zone#69
Zone#4	Zone#26	Zone#48	Zone#70
Zone#5	Zone#27	Zone#49	Zone#71
Zone#6	Zone#28	Zone#50	Zone#72
Zone#7	Zone#29	Zone#51	Zone#73
Zone#8	Zone#30	Zone#52	Zone#74
Zone#9	Zone#31	Zone#53	
Zone#10	Zone#32	Zone#54	
Zone#11	Zone#33	Zone#55	
Zone#12	Zone#34	Zone#56	
Zone#13	Zone#35	Zone#57	
Zone#14	Zone#36	Zone#58	
Zone#15	Zone#37	Zone#59	
Zone#16	Zone#38	Zone#60	
Zone#17	Zone#39	Zone#61	
Zone#18	Zone#40	Zone#62	
Zone#19	Zone#41	Zone#63	
Zone#20	Zone#42	Zone#64	
Zone#21	Zone#43	Zone#65	
Zone#22	Zone#44	Zone#66	

From the zone# [1] select each [3] rd zone

Select all   Refresh

Esc   OK

OK; OK; YES to save file M42
Click the quick button" Refine" to start refinement.
Start the refinement.

The refinement converges with R=8.29% after 3 cycles. Since there are more reflections now, it is possible to refine the anisotropic displacement parameters.

6. Refinement of anisotropic atomic displacement parameters

Save the structure as Ni2Si_dyn_ori_anis
Click the quick button "Edit atoms"; "select all→Define/Edit→ADP parameter(s): harmonic",
OK, OK, Yes
[On the screen: Mains window of Jana2020].
Click the quick button" Refine"

Now the refinement converges with R= 7.83%.

Final structure model: