Zones - A simple program for fast tilting electron-beam sensitive crystals to zone axes

User Manual

1. Introduction

The tilting of crystals to proper zone axes is necessary but a tedious work in taking selected-area electron diffraction patterns and high-resolution images using TEM. In practice, it may take time by manually adjusting the crystal orientation, which is unacceptable to electron beam sensitive materials. This computer program, named *Zones*, is developed to solve this problem.

Zones has two main functions: (A) indexing the electron diffraction pattern taken along different directions and calculate the tilting angles of a double-tilt sample holder to a wanted zone axis. (B) tilting a crystal to the exact zone axis with the help of Laue ring. The combination of them will effectively rotate one crystal to the desired zone axis very precisely.

The main functions of *Zones* are simple, so the operating procedure of the program is linear (that is, no complicated user interface). Users just need to follow the instructions displayed on the window to operate.

Zones is developed in Python3.6 and can run on the Windows system. To start with, just download and run the executable file (Zones.exe) in a Windows system. No other module is required.

2. General Steps and required inputs

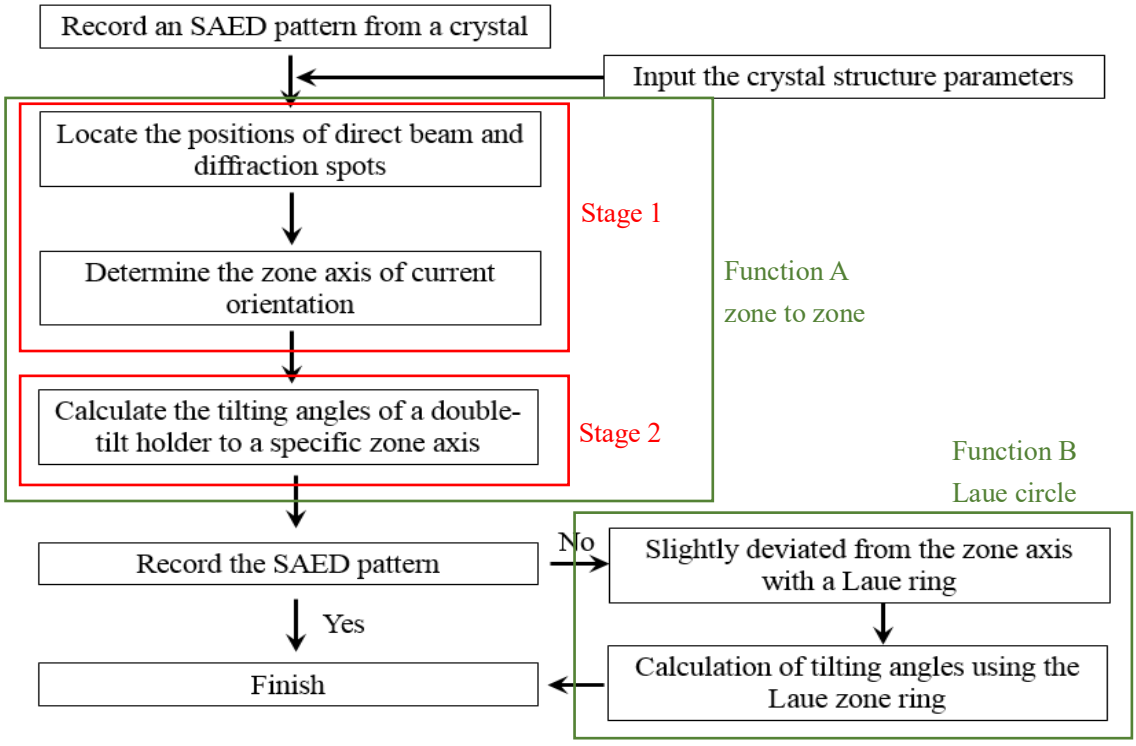


Fig. 1. Working flowchart of the Zones program.

Tilting a crystal from one zone axis to another (**Function A**) has two stages.

Stage1 is to determine the zone axis of current orientation. To do this, the program needs correct inputs, including:

(1) The SAED pattern of the current orientation of the crystal.
(2) The crystal structure parameters files, which give information about the *d* values.
(3) The pixel size of the pattern, which depends on the camera length.
(4) Several manual clicks on the pattern to locate the positions of direct beam and diffraction spots.

Stage 2 is when the current orientation of the crystal is correctly determined, and the program calculates the tilting angles of the double-tilt holder to the desired zone axis. It needs:

(1) The current position of the double-tilt holder.
(2) The wanted zone axis.

Stage 2 requires a successful Stage 1. That is the reason why the program is called "linear". So, operators just need to follow the instructions of each stage and give correct inputs to the program. Of course, if things go wrong, users may go back to the beginning of each stage.

When tilting a crystal to the exact zone axis with the help of Laue ring (**Function B**), the determination of the current zone is not required. It only needs:

(1) The current position of the double-tilt holder.
(2) Several manual clicks on the pattern to locate the Laue ring.

Users may choose whether to operate **Function A** (tilt from zone to zone) or **Function B** (tilt to exact zone axis with Laue ring) at the beginning of the program. So, if the crystal is tilted to a close but not exact zone axis based on the calculate result of function A, users may go back to the beginning and run the function B. The combination of these two functions can produce very accurate result.

3. Detailed information about every step

3.1 Confirm the pixel size. Here is the very first window. The pixel size is a constant and is dependent on the camera length and the number of pixels on a camera. Pixel size may vary in operations, so make sure the pixel size of the pattern we choose later is correct. In the program, the default value is 0.00015360 ($Å^{-1}$). One of the other common values is 0.00045148($Å^{-1}$). You can calculate this value using your own electron diffraction pattern.

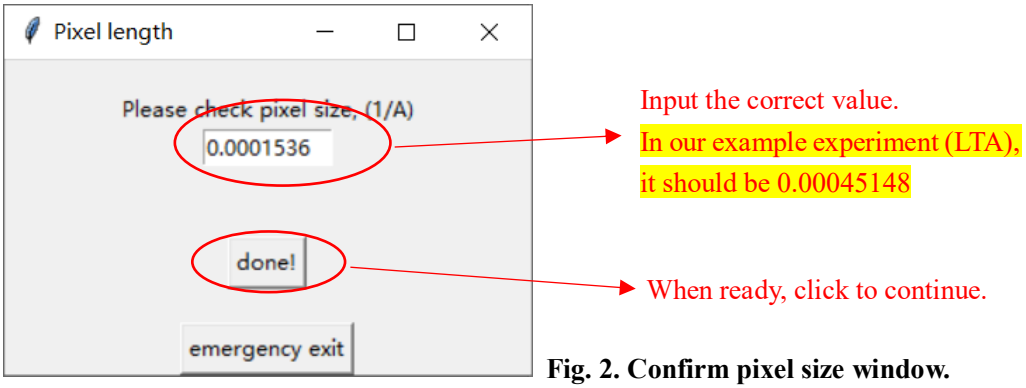


Fig. 2. Confirm pixel size window.

3.2 Choose the files. The next window requires the user to input files. Firstly, we select the image of the SAED pattern by clicking the upper button. A file selector will pop up. After that, if we need to calculate a zone-to-zone tilting angle (function A), then click on the second button to continue, otherwise click on the third button to run Laue circle calculation (function B).

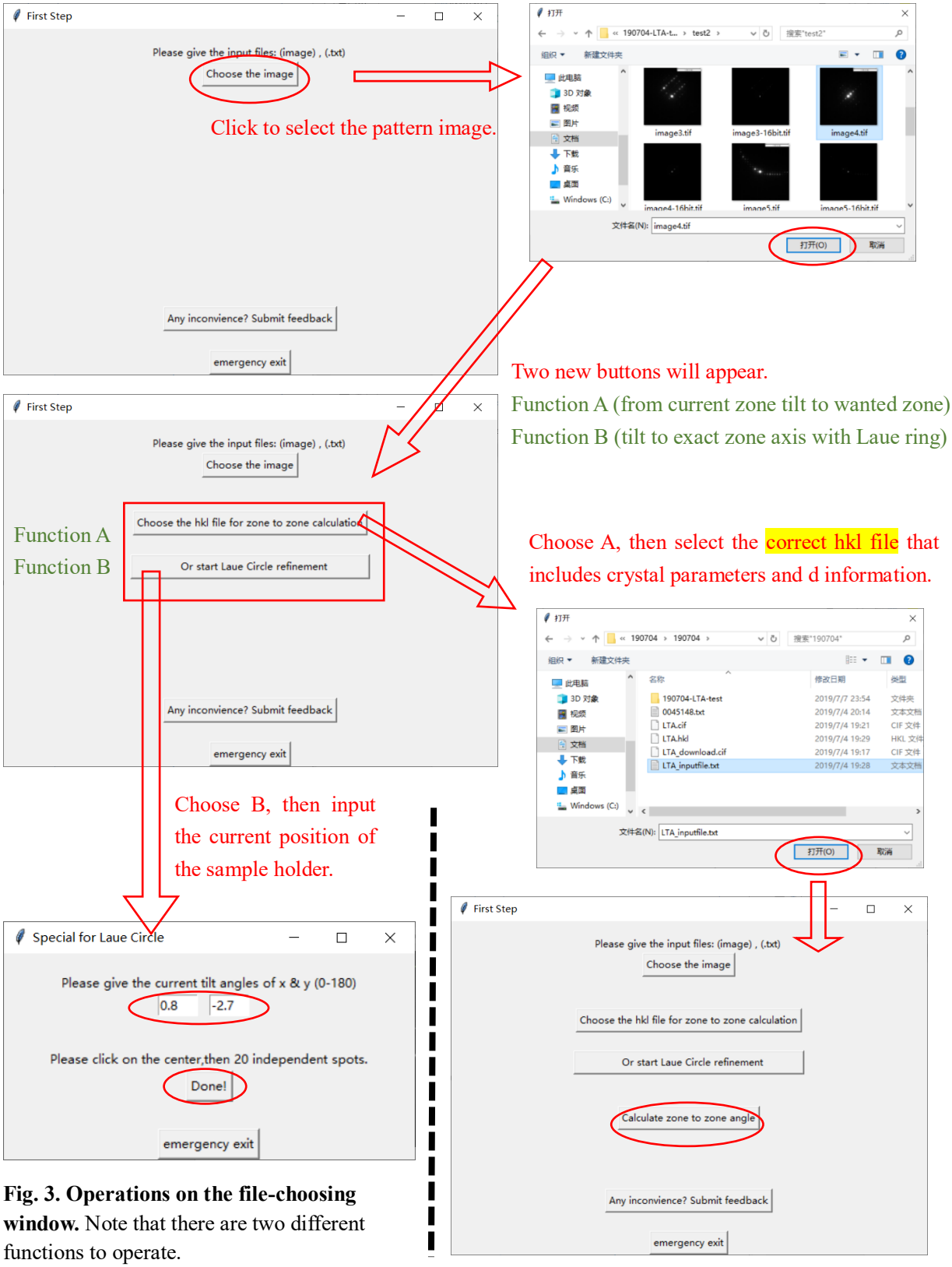
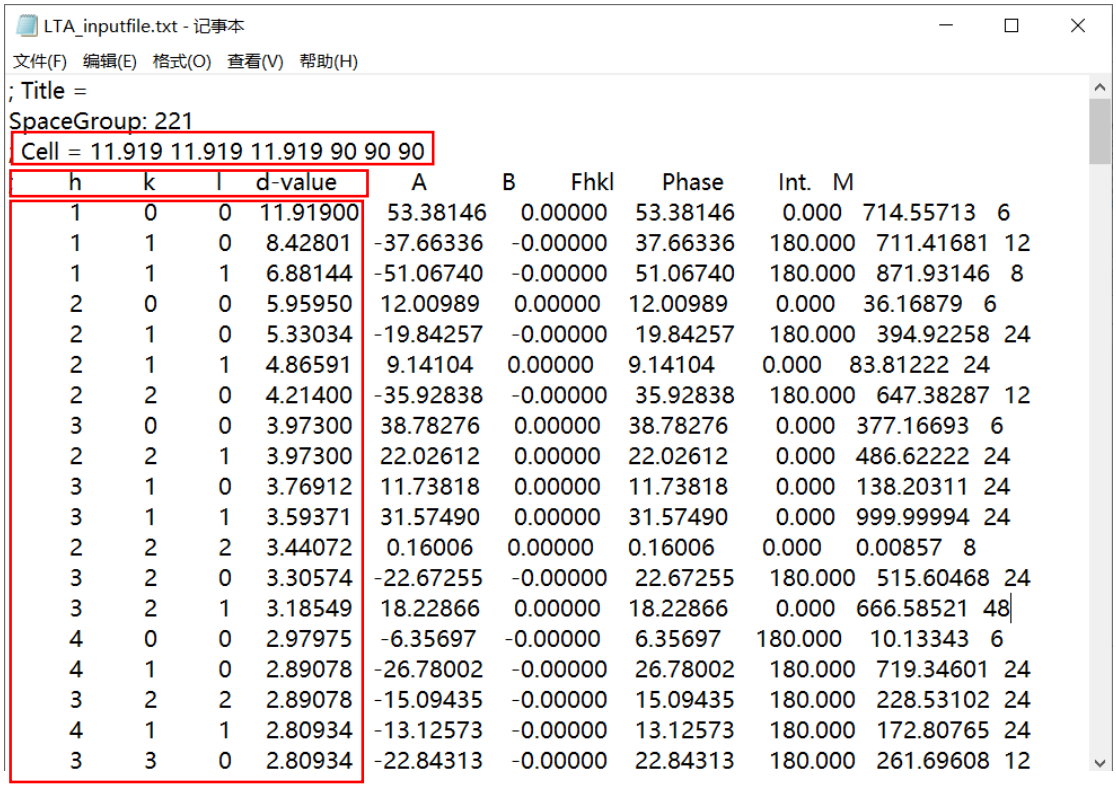


Fig. 3. Operations on the file-choosing window. Note that there are two different functions to operate.

In function A, the program needs to index the current orientation. That means the crystal parameters and d values are required. They should be stored in the hkl file, and be in a standard format shown in Fig. 4. Users should convert a crystal information file (.cif) to this standard format (.txt) by certain software. Our case is *emap*.


LTA_inputfile.txt - 记事本

文件(F) 编辑(E) 格式(O) 查看(V) 帮助(H)

; Title =

SpaceGroup: 221

Cell = 11.919 11.919 11.919 90 90 90

h	k	l	d-value	A	B	Fhkl	Phase	Int.	M
1	0	0	11.91900	53.38146	0.00000	53.38146	0.000	714.55713	6
1	1	0	8.42801	-37.66336	-0.00000	37.66336	180.000	711.41681	12
1	1	1	6.88144	-51.06740	-0.00000	51.06740	180.000	871.93146	8
2	0	0	5.95950	12.00989	0.00000	12.00989	0.000	36.16879	6
2	1	0	5.33034	-19.84257	-0.00000	19.84257	180.000	394.92258	24
2	1	1	4.86591	9.14104	0.00000	9.14104	0.000	83.81222	24
2	2	0	4.21400	-35.92838	-0.00000	35.92838	180.000	647.38287	12
3	0	0	3.97300	38.78276	0.00000	38.78276	0.000	377.16693	6
2	2	1	3.97300	22.02612	0.00000	22.02612	0.000	486.62222	24
3	1	0	3.76912	11.73818	0.00000	11.73818	0.000	138.20311	24
3	1	1	3.59371	31.57490	0.00000	31.57490	0.000	999.99994	24
2	2	2	3.44072	0.16006	0.00000	0.16006	0.000	0.00857	8
3	2	0	3.30574	-22.67255	-0.00000	22.67255	180.000	515.60468	24
3	2	1	3.18549	18.22866	0.00000	18.22866	0.000	666.58521	48
4	0	0	2.97975	-6.35697	-0.00000	6.35697	180.000	10.13343	6
4	1	0	2.89078	-26.78002	-0.00000	26.78002	180.000	719.34601	24
3	2	2	2.89078	-15.09435	-0.00000	15.09435	180.000	228.53102	24
4	1	1	2.80934	-13.12573	-0.00000	13.12573	180.000	172.80765	24
3	3	0	2.80934	-22.84313	-0.00000	22.84313	180.000	261.69608	12

Fig. 4. Standard format of the crystal parameters and d-values file. Also known as hkl file. Note that an hkl file in wrong format may lead to failure.

3.3 (Function A) Click on the pattern to locate the positions of direct beam and diffraction spots.

If a proper hkl file is selected, the program will continue to the next section. In this section, users will need to manually locate the direct beam first.

The first click on the pattern will zoom in the image around the click, so that other clicks will be easier. Then users need to click on the position of the direct beam twice in order to increase accuracy. The program will take the average position of these two clicks as the location of the direct beam. Note that it is fine to take a diffraction spot as the direct beam – in our indexing method, the only thing matter is the vector (from a selected beam center to any diffraction point).

Fig. 5. Locate the direct beam by manual clicks in function A.

After the position of the direct beam is recorded by the clicks, the next step is to input five

pairs of independent vectors in reciprocal space (the pattern). As the vectors starting point (the direct beam, marked as star on the pattern) is recorded, the users need to click on five pairs of independent points. That is the 1st and 2nd click will be considered as one pair of spots, then 3rd and 4th, etc.

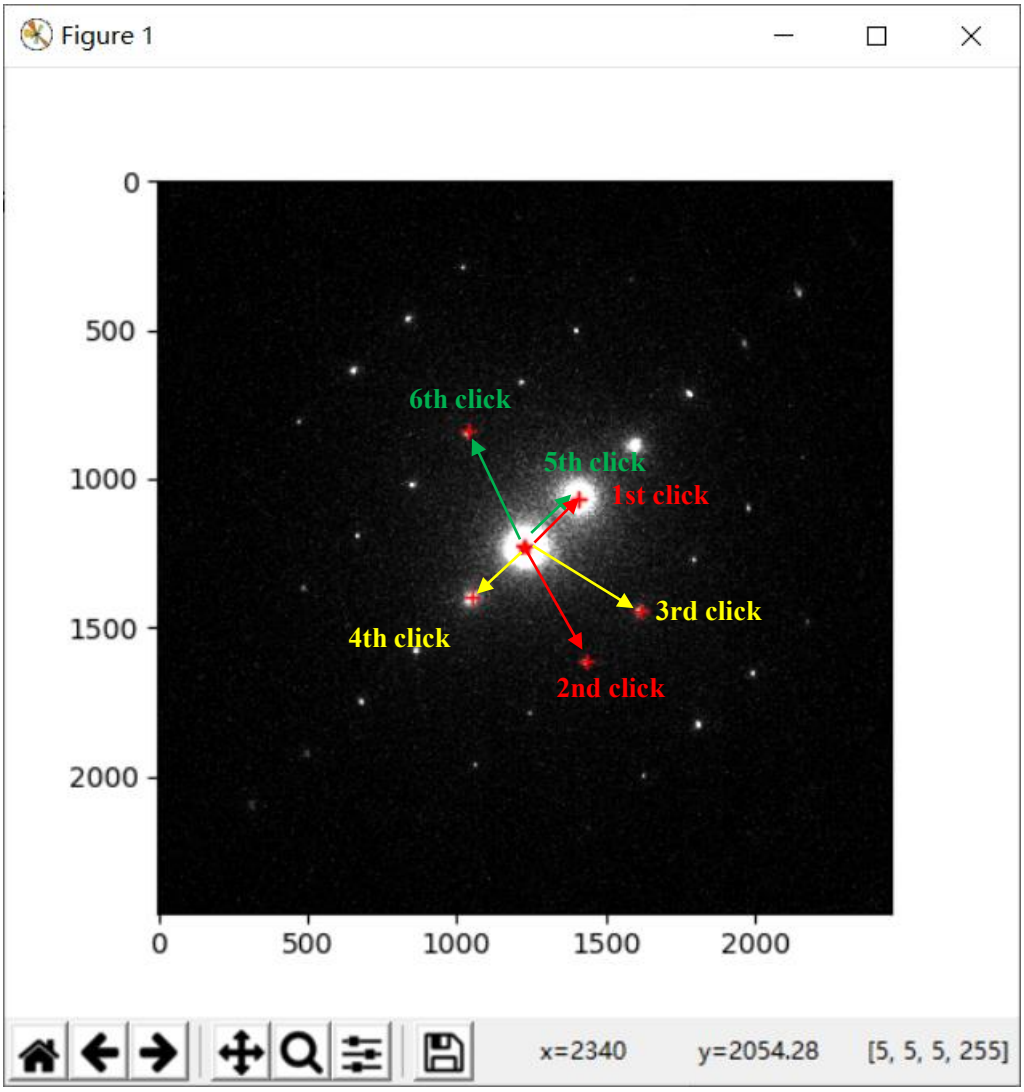


Fig. 6. Locate five pairs of independent spots. That means ten clicks in total are required. Note that it is NOT necessary to choose different spots for different pairs. For example, 1st and 5th click may be on the SAME spot. But 1st and 2nd click must be on different spots nearby the selected center (vectors need to be independent).

3.4 (Function A) Determine the zone axis of the current orientation. Based on the input locations of the direct beam and diffraction spots, the program will run for a while to index the current zone. Note that in our index method, the index results are equally highly likely, and more than one zone axes might be given in some cases if diffraction patterns of different zone axes are similar to each other. It is up to the operators to determine which one is the most probable result.

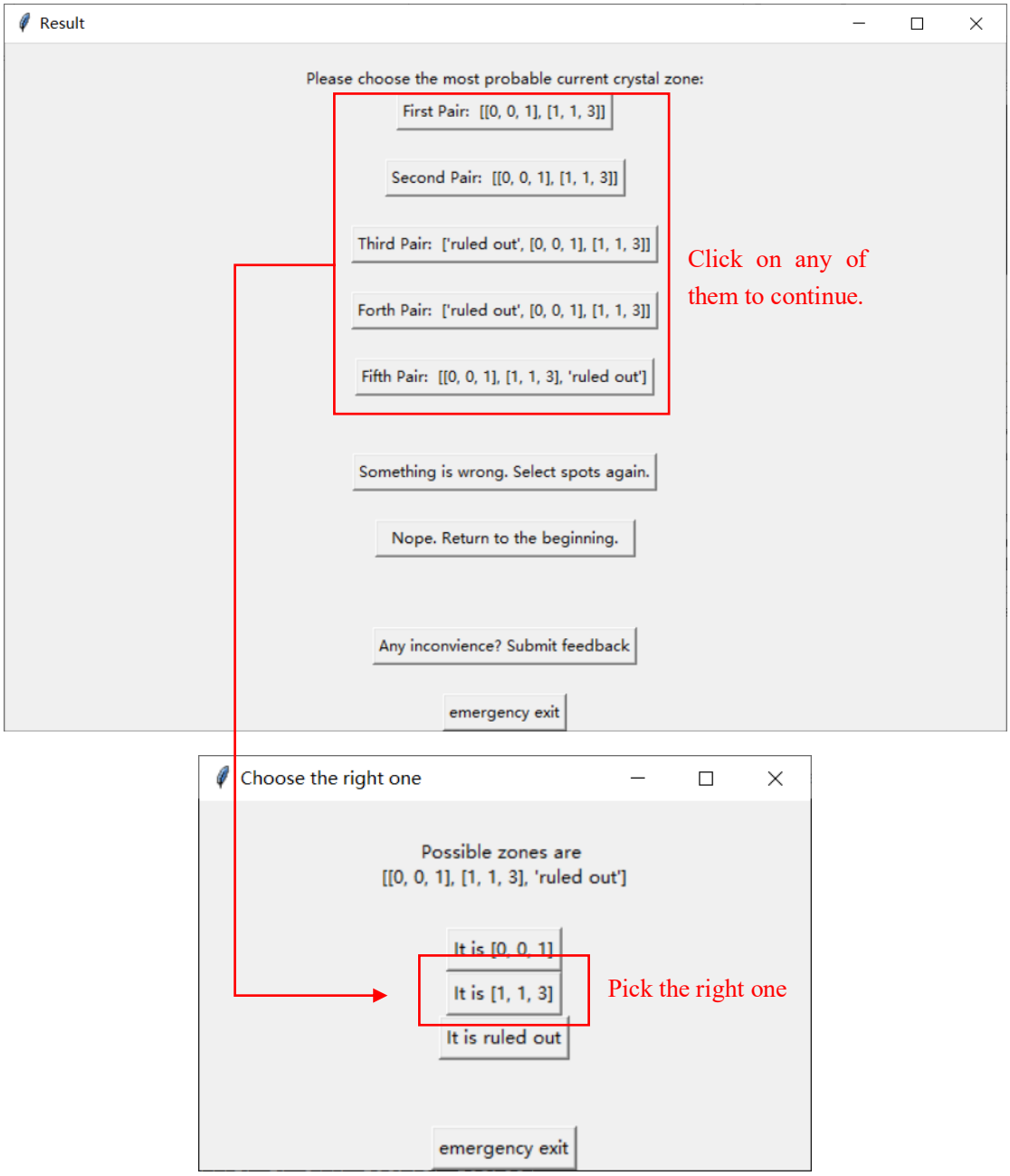


Fig. 7. Index results of the current orientation. In our example case (LTA), the diffraction pattern of the [001] zone is different from the experiment pattern. So [001] should be ruled out by the operator and [113] zone should be chosen manually.

3.5 (Function A) Input the current holder position. In the example experiment, the pattern was taken as the double-tilt holder in a position of (x = -24.8°, y = -0.5°).

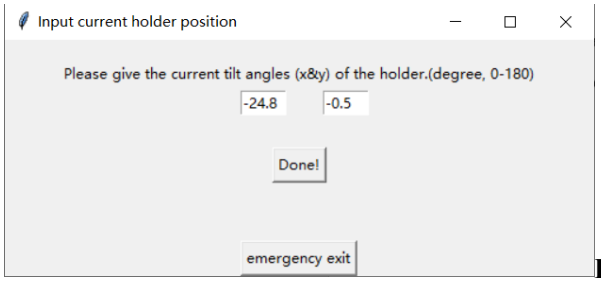


Fig. 8. Input holder angles window. Herein,

x and y means α and β tilting angles of the holder.

3.6 (Function A) Tilt to the desired zone based on the calculation result. The program will process all the input information and then show you how to tilt to default zones. Ideally, If we follow the result and tilt the crystal, the crystal will move to the desired orientation accurately - within an error less than ±3°. That's all for the Function A part.

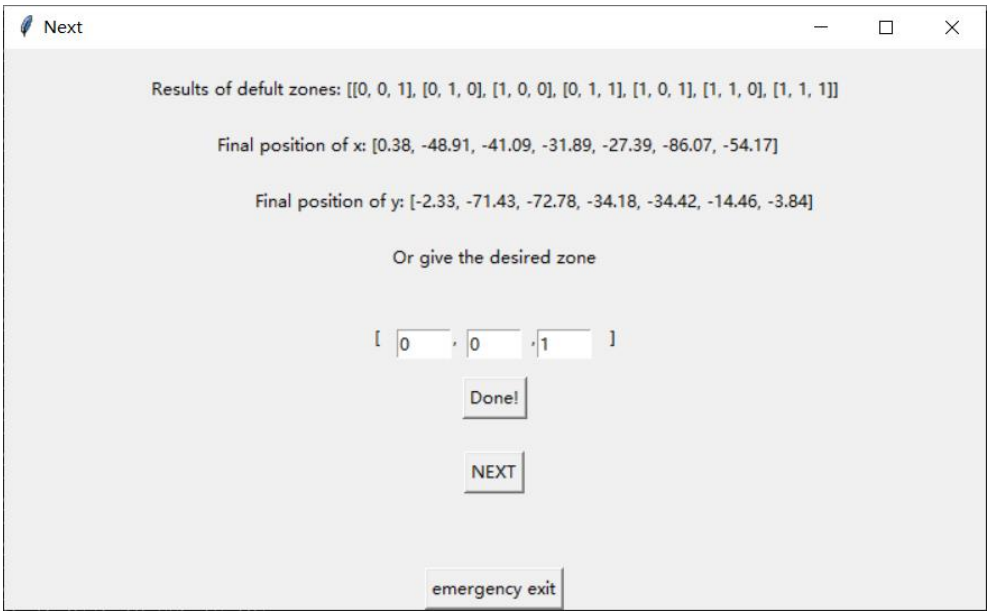
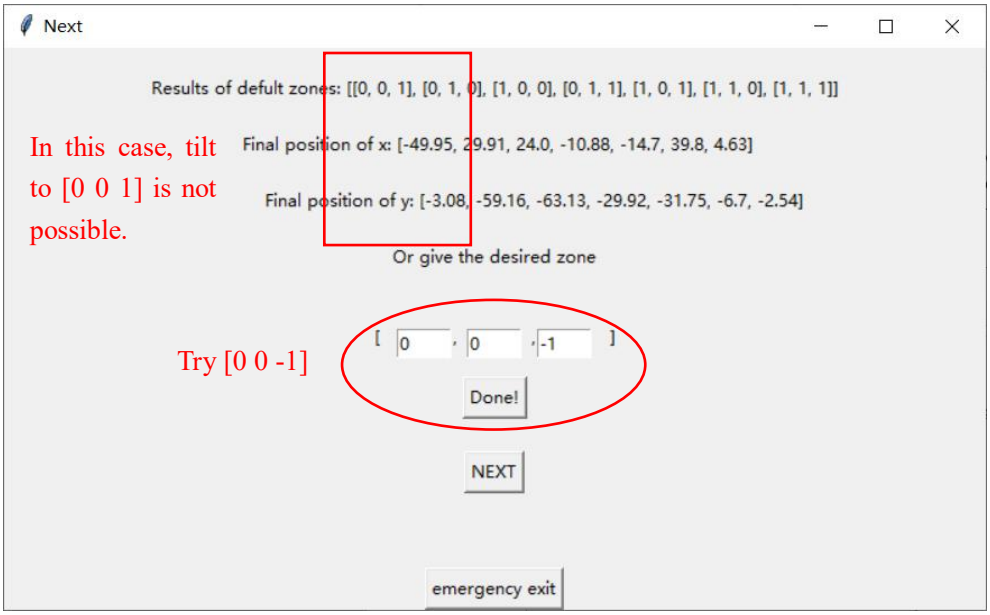


Fig. 9. Final result of the holder positions. For example, if we want to turn to [001], then the double-tilt holder should tilt to the position of (x = 0.38°, y = -2.33°).

In some cases, the calculation result may lead to an opposite direction. This is because of these two directions are indistinguishable to the program. If you believe the index of current zone is correct but the result is wrong, try the opposite direction, e.g. try [0 0 -1] zone instead of [0 0 1].



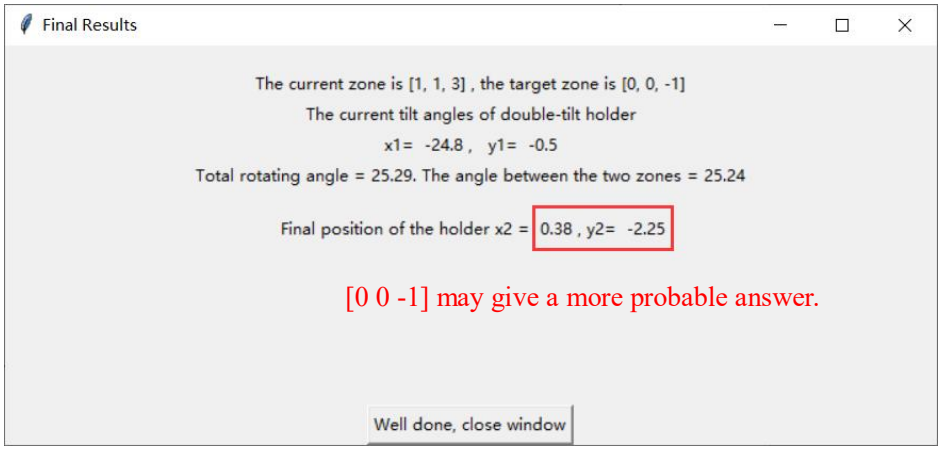


Fig. 10. Try the opposite direction may produce correct answer.

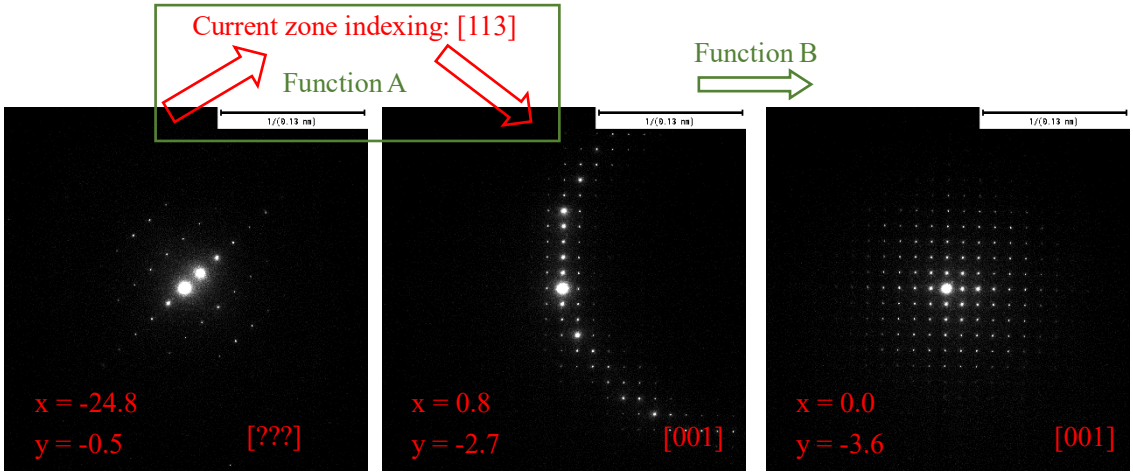
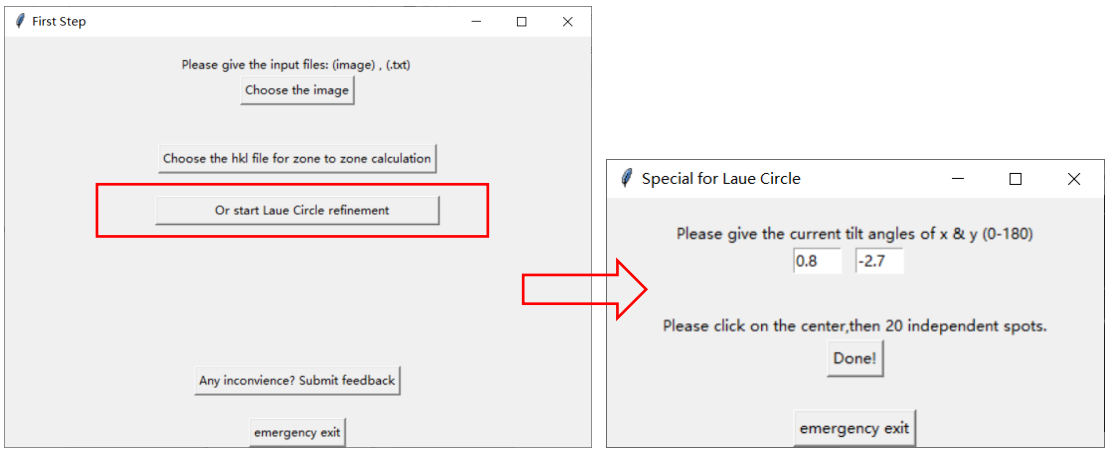


Fig. 11. Review the whole process with experiment data.

3.7 (Function B) Locate the Laue circle in the pattern, then get result. After the operations of function A, the crystal may turn to a position slightly different from the desired zone. (Fig. 11.) A Laue circle may occur, and function B of the program is helpful in this situation.

Again, as the SAED image was selected, if we wish to run function B (that is to use the Laue circle to slightly tilt the crystal to a proper zone), then we should click on the third button on the window. The current holder position firstly needs to be input. Then follow the instructions.



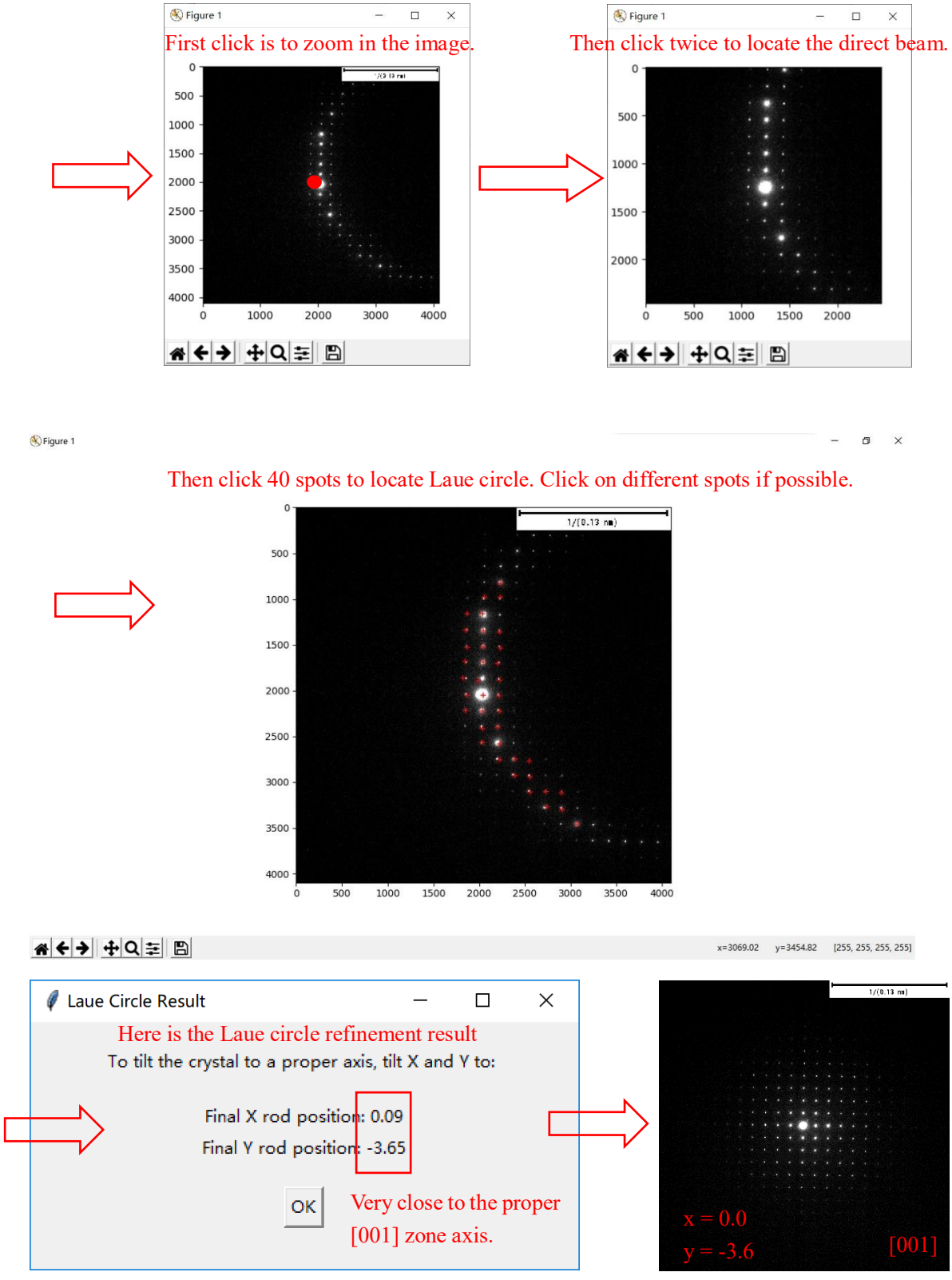


Fig. 12. The whole process of function B. Laue circle refinement doesn't require a current zone axis determination, thus a simpler operation than function A.