Simulation and Analysis of Kikuchi Patterns
Including Double Diffraction Effect

(SAKI3d)

User’s manual

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1. Introduction

Selected area electron diffraction (SAED) analysis has been extensively used in materials science for phase identification, interpretation of twins and coexisted multiple phases and so forth. Simulation of electron diffraction pattern plays an important role to interpret experimental results.

Electron diffraction patterns produced by the selected-area mode in transmission in the electron microscope can be of three different types in Table 1.

<table>
<thead>
<tr>
<th>Diffraction pattern</th>
<th>TEM Specimen</th>
<th>Landyne software</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a) Ring pattern</td>
<td>Polycrystalline specimen</td>
<td>PCED3 and QPCED3</td>
</tr>
<tr>
<td>(b) Spot pattern</td>
<td>Single-crystal region of the specimen</td>
<td>SAED3 and QSAED3</td>
</tr>
<tr>
<td>(c) Kikuchi line pattern</td>
<td></td>
<td>SAKI3 and SPICA3</td>
</tr>
</tbody>
</table>

(b) and (c) often occur on the same diffraction pattern. Kikuchi patterns consist of pairs of parallel bright and dark lines. Kikuchi patterns are taken from a single-crystal region of the specimen which is reasonably thick (~1/2 maximum usable penetration) and has a low defect density.

Kikuchi patterns are very important for the following reasons [Edington, 1975],

(i) They enable the tasks be performed more accurately than with spot patterns.

(ii) They constitute a “Kikuchi map” which describes the distribution of Kikuchi lines within a unit triangle of the stereogram, which can be simulated with Landyne SPICA. The specimen may be tilted in a self-consistent manner while working in the microscope.

(iii) They determine the sign and magnitude of the deviation $s$ from the exact Bragg position. The relationship between the bright Kikuchi line and its associated diffraction spot is used.
(iv) They define the sense of tilt because the Kikuchi pattern behaves as if it is fixed to the bottom of the crystal and consequently it moves in the same sense as the crystal that is the lines move in a direction perpendicular to the tilt axis and towards the edge of the specimen tilted up.

(v) Small angular tilts, $\delta$, can estimated, where $\delta = \frac{m}{L}$ and $m$ is the linear displacement of the Kikuchi line pattern, normal to the tilt axis; $L$ is the camera length.

(vi) Large angular tilts can be measured by identifying specific $B = g_1 \wedge g_2$ accurately.

(vii) They determine crystal symmetry because, unlike spot patterns, Kikuchi patterns exhibit real crystal symmetry.

SAKI3 is designed for simulation and analysis of Kikuchi patterns with double diffraction effect. The current version is 3d. It is one of the key computer programs in Professional Java Electron Crystallography Toolkits (ProJECT), which is developed by Dr. X.Z. Li for electron diffraction simulation and crystallographic analysis. Each program in ProJECT covers one subject in the application of electron diffraction simulation and analysis. ProJECT can be used mainly as a tool for researchers in TEM works, as well as a teaching aid on fundamental crystallography.

SAKI3 is mainly coded in Java and partially coded for PC with Microsoft windows only. The features of SAKI3 are listed here,

- Template for creating new crystal structures.
- Simulation of SAED pattern of spot and Kikuchi lines with double diffraction effect.
- Flexible labels of the indices for SAED patterns of spots and Kikuchi lines.
- Convert between Miller and Miller-Bravais index.
- Drag and drop action for loading experimental diffraction patterns (.jpg and .tif).
- Resize/rotate/invert experimental SAED/Kikuchi pattern.
- Compare with experimental SAED pattern with complex Kikuchi lines to find the precise orientation of the crystal phase.
- Compare with experimental SAED pattern to reveal the occurrence of forbidden diffraction due to the double diffraction effect.
- Allow to save SAED pattern with Kikuchi lines to .jpg and .tif.
2. Theory background

When the TEM sample is reasonable thick, but is still largely transparent to electrons, incoherent scattering contributes a diffuse background to the diffraction pattern. More interesting, intersecting sets of straight lines appear on top of this diffuse background. These are Kikuchi lines, which maybe either bright or dark, but are impressively straight and regularly arranged. They provide important crystallographic information about the sample.

The main features of the geometry of Kikuchi patterns can be understood by making use of the simplified treatment first proposed by Kikuchi in 1928 in which the lines are considered to arise from diffraction of electrons that have been previously inelastically scattered by the specimen, more details are in the books listed in reference. These inelastically scattered electron are present in most spot electron diffraction patterns, both as diffuse halo around the transmitted and strong diffracted spot and as an overall faint background intensity. Since many more electron suffer small energy changes than large ones the diffuse halo loses intensity with increasing distance from the transmitted beam, and the intensity distribution may be described in terms of the polar diagram in Figure 1.

2.1 Simulation of Kikuchi pattern

Kikuchi lines are useful for precise determination of the specimen orientation in a TEM. The location of the Kikuchi line is related the sign and magnitude of the deviation parameter, s of the diffraction spot with a vector of g,

\[ x = \frac{sR}{g^2 \lambda} \]

where x is the distance between the diffraction spot at R and its corresponding bright Kikuchi line, and \( \lambda \) is the wavelength of the incident beam.
2.2 Analysis of Kikuchi pattern for precise orientation

The most accurate analysis requires a minimum of three nonparallel Kikuchi line pairs to obtain B unambiguously from one pattern. The incident beam (O) can be specified by the angles to the zone axes of the Kikuchi poles (A, B and C),

\[ \phi = R/L \]

Where R is the distance OA, OB, OC and L is the camera length.

Due to inward curvature of Ewald sphere, indices and zone axes satisfy,

\[ (hkl) \ast [uvw] > 0 \]
Here the (hkl) of the outside line of each Kikuchi pair and other Kikuchi pole of zone axis [uvw]. SAKI3 provide a step-by-step approach to analyze the precise orientation from a complex pattern of Kikuchi lines from three different zones.

The precise orientation can be derived as a zone axis from the three angles between the incident beam and Kikuchi poles. SAKI3 analysis can be applied to all seven crystal systems.

2.3 Kikuchi map

Kikuchi maps consist of a montage of Kikuchi patterns taken with different incident beam positioned in the corresponding place in the unit triangle of the stereogram such that all the Kikuchi lines join up to produce a ‘road’ map of the unit triangle. The simulation of Kikuchi is given in SPICA software.

2.4 Double diffraction effect

When a TEM specimen is thin enough, only single scatterings need be considered. Double diffraction, where an electron diffracted twice before leaving the specimen, requires that that beam from the first diffraction serve as the incident beam for a second diffraction. Most TEM samples are sufficiently thick that electrons can undergo multiple diffractions, especially when $s \approx 0$. A dynamical theory is often required to analyze these problems in detail. However, some aspects of multiple diffractions can be understood by straightforward geometrical considerations and kinematical theory, 1) occurrence of forbidden diffraction and 2) interactions between crystallites. SAKI3 deals with the first case. The central idea is that a diffracted electron acts an incident beam for a second diffraction.

3. Graphic user interface of SAKI3

3.1 Main interface
The main interface of SAKI3 is a frame with a menu and a toolbar and a calculation dialog is show in Figure 2. The frame of SAKI3 with an experimental SAED pattern and an analysis dialog are shown in Figure 3. Menu gives more text description and organized in groups while toolbar shows in graphics and easily to access. Although most functions of menu and toolbar are same, however, there are some functions are only provided either in menu or toolbar.

Figure 2. Snap-shot of the main panels in SAKI3 with simulated Kikuchi pattern of Al [001].
3.2 Tools and options

Beside the main interface shown above, a variety of tools and options are available to assist for the simulation and analysis process. New crystal data file can be prepared with a template, as shown in figure 4. Experimental diffraction pattern can be processed for resizing, rotation and alignment. The precise orientation can be expressed in angles and rational indices of zone axis. The indices of spots and Kikuchi pattern can be displayed using intensity as threshold. Miller index and Miller-Bravais index can be converted into each other. The output area can be set up and saved as in .jpg or .tif format. A periodic table of elements and a table of space group are provided.
4. Usage of SAKI3

To run the software, a recent version of Java Runtime Environment (e.g., JRE 1.8.0_151), must be installed on a PC for Microsoft window (XP or up). The SAKI3 is one key component in the Landyne software, which can be download from Landyne website:

http://landyne.scienceontheweb.net
Decompress the installation file landyne.z7 in a selected directory and execute landyne.exe by double click. The usage of SAKI3 is straightforward, firstly to load structure data and to set up input parameters, then simply click **Simulate** button in **Simulation** control panel.

The basic steps for using SAKI3 are listed here,

i) The toolbar displays the most frequently used functions, which can be hidden for a small monitor.

ii) New crystal data file can be created using the template, which is available in all Landyne software.

iii) Crystal data file can be loaded in menu file system.

iv) Experimental diffraction pattern in .jpg or .tif format can be loaded in menu file system or directly by the drag and drop using mouse.

v) The precise orientation can be derived using three Kikuchi poles in the experimental diffraction pattern.

vi) Click the simulate button in the simulation dialog to create a diffraction spot and Kikuchi pattern, to make resizing and rotation adjustment, chose appearance, and other options for display.

vii) Spot pattern and Kikuchi lines can be indexed separately.

viii) Miller and Miller-Bravais index can be converted with Hexagonal system dialog.

ix) Final result can be saved as an image in format of .jpg or .tif.

**4.1 Prepare new data file**

Structure data file can be prepared using the **Crystal new data** dialog window in Figure 4 or using a text editor to modify other structure data file. The dialog window provides assistant in preparation of the data file and also makes sure to meet the requirement of the format of the file.

- **Description** field can be filled in a name of the phase.
- **Bravais lattice** can be selected a list. Lattice types are provided for each crystal system.
- **Lattice parameters** are preset according to Bravais lattice.
- **Space group number** is limited to possible group according to Bravais lattice.
- Fill the info of each atom in and then add to the atom list. For modifying or removing the list, user should select the atom row in the list and click the Remove button.
- Notes field can be used for the purpose for simulation or reference.

To save the data structure click the Save button or to make new one click the New button.

### 4.2 Simulation

Kinematical diffraction theory is used in the simulation. Basic parameters for simulation, e.g., high voltage, pattern zoom and intensity scale can be adjusted in Simulation menu.

After structure data for simulation is loaded, thickness, zone axis, tilt angles are set up, new diffraction pattern is generated by simply click Simulate button. The pattern can be adjusted by changing other parameters.

**Orientation and mirror operation** are used to orient the simulated pattern to match the experimental pattern and to generate various twins.

**Diffraction Pattern** can be viewed in open or solid circles and in various color. Kinematical or double diffraction pattern can be displayed. Index can be labeled for basic reciprocal vectors and for diffraction spots selected by the intensity level. Basic vectors and Laue center can be displayed and hidden.

### 4.3 Determination of index of Kikuchi pattern and precise orientation

SAKI3 can be used to determine the index and zone axis of an experimental diffraction pattern, and precise orientation using three Kikuchi poles if it belongs a known crystal structures. Experimental diffraction pattern can be loaded and centered by drag and drop operation. The size and orientation can be adjusted if needed. Figure 3 shows the analysis dialog window.

**Step 1.** Set up R-spacing marker using the scale bar on experimental pattern. Adjust calibration circle using the pattern zoom and then locked it down.

**Step 2.** In analysis dialog window, chose incident beam (default) or Kikuchi pair1, pair2, pair3 and check show box. Adjust the width, the orientation and the position with mouse.

**Step 3.** Define the tolerance value for width (default as 3 pixel) and angle (2°), save possible indices to a file and then search the suitable index.
Step 4. The zone axes are generated with the input indices of the Kikuchi pairs.

Step 5. The precise orientation can be expressed in the angles to the Kikuchi poles; or the zone axis in rational number. The Miller-Bravais index for hexagonal system can be converted with the optional tool.

Step 6. Double diffraction effects on SAED diffraction pattern can be added on by simply selecting the option.

![Simulation and Analysis of Kikuchi lines](image)

Figure 5. Experimental SAED pattern of Cr₃Ge phase which was loaded in the display panel of the SAKI3.

5. Application examples
Kikuchi pattern has various applications as listed in the introduction part. Two examples are given in the following.

5.1 Simulation of spot and Kikuchi diffraction patterns

Cr$_3$Ge phase was observed in CoFeCrGe Heusler alloy [Jin, Li, Sellmyer 2018]. Figure 5. Shows the experimental diffraction pattern of Cr$_3$Ge phase. The zone axis was determined to be [210].

Figure 6. (a) Simulated spot pattern in kinematical diffraction, (b) with double diffraction effect. (c) Simulated Kikuchi pattern supposed on experimental pattern (d) the simulated pattern only for clarity.
Figure 6 shows simulated patterns in kinematical theory. In comparison with experimental pattern, some spots doesn’t show due to extinction in (a) and appear when double diffraction effect is considered in the simulation. Simulated Kikuchi lines (c) with and (d) without experimental pattern.

5.2 Precise orientation determination

Figure. 7. (a) Experimental complex pattern of Mg in Edington’s book. (b-d) Three simulated Kikuchi patterns along the zone axes of [0 -1 2], [-1 -2 2] and [1 3 -3].
The example of the precise orientation determination is taken from Edington’s book (1975), which is a complex Kikuchi pattern of Mg sample. The re-analysis parameters and results are shown in Figure 3. The simulated patterns were carried out separately along all three Kikuchi poles, in which it shows how the zone axes and Kikuchi line pairs match with the experimental pattern.

![Image](image.png)

*Figure 8. The tool for converting Miller indices to Miller-Bravais indices.*

Figure 8 shows the tool for converting Miller indices to Miller-Bravais indices for hexagonal system. The results are listed in the table 2 for comparison.

<table>
<thead>
<tr>
<th>Analysis</th>
<th>Φ₁</th>
<th>Φ₂</th>
<th>Φ₃</th>
<th>[u v t w]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Edington’s book</td>
<td>3.1°</td>
<td>11.1°</td>
<td>6.5°</td>
<td>[1, -6.024, 5.04, 13.98]</td>
</tr>
<tr>
<td>SAKI3</td>
<td>3.21°</td>
<td>10.45°</td>
<td>6.63°</td>
<td>[1, -4.095, 3.095, 8.194]</td>
</tr>
</tbody>
</table>

In the calculation, the high tension of the microscope is assumed to be 200 kV and the crystal structure of Mg phase, hcp, P63/mmc, a =3.1094 Å and c=5.2108 Å is used. Due to the accuracy in measurement of the Kikuchi pairs in Edington’s book and from SAKI3, the results are slightly different.

6. Related programs
All programs in Landyne suite are using the input structure data in the same format. A few of them are listed here, which can be used in combination with SAKI3. Users may check the program specification or user’s manual for each program in detail.

6.1 **JECP/SVAT2**

The need of a structural viewer/an analytical tool, using the same input data format as the JECP and ProJECT software, becomes obvious with the growth of the JECP and proJECT programs and users.

6.2 **SAED3**

SAED3 can simulate spot patterns from multiple crystal files for comparison or for simulation of various twins and coexisted structures with fixed orientation. SAKI3 is used for analysis of precise orientation and SAED3 is used for determination of the zone axis. SAKI3 and SAED3 can be viewed as an extension of each other.

6.3 **SPICA3**

SPICA3 is designed for stereographic projection with an application for specimen orientation adjustment using TEM holders. SPICA3 can be used to generate Kikuchi map.

Reference


