Automated Indexing of Wide Bandpass Laue Images

Jin-Seok Chung
Metals and Ceramics Division
Oak Ridge National Laboratory

ABSTRACT

An important goal of x-ray microdiffraction is to characterize texture and strain in two or three dimensions with micron resolution. With this scanning x-ray microdiffraction, it is essential to accelerate data collection and to automate the diffraction data analysis. One solution is to collect wide bandpass Laue images which have information equivalent to that of many monochromatic diffraction scans. However, to get a scanned image, tens of thousands of Laue images must be analyzed in reasonable time; automated indexing of Laue points is essential. By comparing measured angles between scattering vectors of Laue points to angles between possible indexes derived from the bandpass, a unique set of indexes can be found. Indexing multi-grain images can also be done by repeating the indexing process for points which do not match an allowed reflection.

Scanning X-ray Microdiffraction

The availability of third generation synchrotrons and newly developed x-ray microfocusing optics[1, 2] have enabled the use of micron-size x-ray beams with significant intensities. This new measuring tool makes new kinds of measurements possible. One of them is micro-diffraction for the measurement of phase, texture and micro-strain with micron resolution. By scanning a microprobe x-ray beam over the sample, the strain distribution can be measured non-destructively with the spatial resolution of the beam size. Until recently, the size of x-ray beams has been on the order of mm^2; this probe size is large compared to the spatial inhomogeneity of most technologically interesting samples. With intense micron-size beam, new information about strain texture and phase inhomogeneities can be obtained with micron spatial resolution.

The basic principle of measuring strain in microdiffraction follows from Bragg's law; the same principle is used in traditional measurements;

$$2\,d_{hkl}\,\sin\theta = \lambda = \frac{hc}{E}. \quad (1)$$

Here d_{hkl} is the distance between scattering planes. The strain can be measured from $\Delta d\,/\,d$. But to get a scanned image, the strain has to

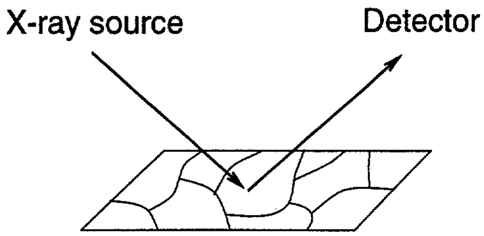

Figure 1: Scanning x-ray microdiffraction

be measured for tens of thousands of different points in the sample and conventional measurements take too much time.

To make scanning x-ray microdiffraction useful in practice, it is necessary to achieve a strain resolution of $\sim 10^{-4}$ and a spatial resolution of $\sim 1\mu$m. At these performance levels, this new analysis tool will benefit many material studies like stress induced cracking, electromigration, etc. Until recently, measurements with such small beams have been impractical because of weak sources and limited x-ray optics. With various techniques like hard x-ray zone plates[3], Kirkpatrick-Baez mirrors[4, 5], and glass capillary optics[6], x-ray beam sizes smaller than 1μm have been recently reported. With these new developments in hardware, scanning x-ray microdiffraction is now practical.

The biggest obstacle in scanning x-ray microdiffraction is processing measured data fast enough to get a scanned image in a reasonable time. With conventional techniques, the measurement of strain at a single point depends strongly on the nature of the sample, and can range from a few seconds for a single crystal sample to a few hours for a polycrystalline sample with unknown crystal orientations. To repeat measurements at tens of thousands of different points in polycrystalline samples, a new method of measuring strain should be developed, since the conventional method would take days, weeks or years.

The white-beam Laue method greatly accelerates data collection compared to measuring rocking curves with monochromatic x-rays. Each reflection in a Laue image has information equivalent to a rocking curve. Thus a single Laue image avoids the time of orienting and rocking a crystal at many reflections. Just one image gives almost all the information about the crystal.

White beam Laue patterns can be digitized by an x-ray sensitive CCD area detector, an image plate or a 2D wire proportional counter. CCD's are favored because they have fast read out and can collect intense beams without damage. Modern CCD's like one in our setup, typically have more than a million pixels with a pixel size of $< 25\mu$m and well capacities of more than 5×10^5 electrons. These CCD's provide enough dynamic range and angular resolution for obtaining precision diffraction information from Laue points.

Figure 2 shows a typical experimental setup for taking Laue images with a CCD detector. Since x-rays from a synchrotron have strong polarization in the plane of the orbit, the CCD is installed in the vertical scattering plane.

WIDE BANDPASS LAUE MEASUREMENTS

Typical bandpass of undulators is only about 1%[7]. There are some benefits of having narrow bandpass. As shown in Fig. 3, the range of angles for each reflection decreases with smaller bandpass. Thus when the diffraction angle, θ is measured from a CCD image, there are only a few possible indexes for the reflection. Indexing is simpler in this case. The disadvantage

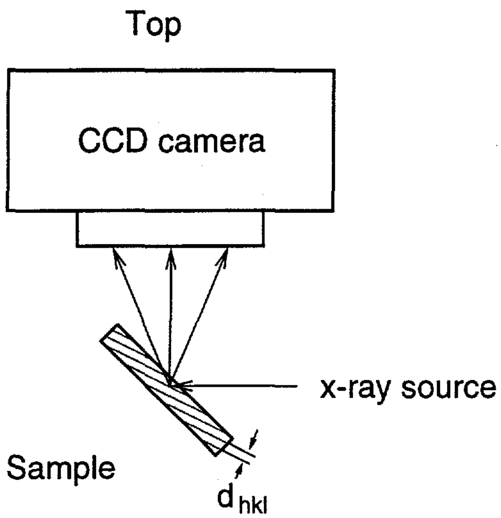

Figure 2: A side view of a experimental setup for microdiffraction

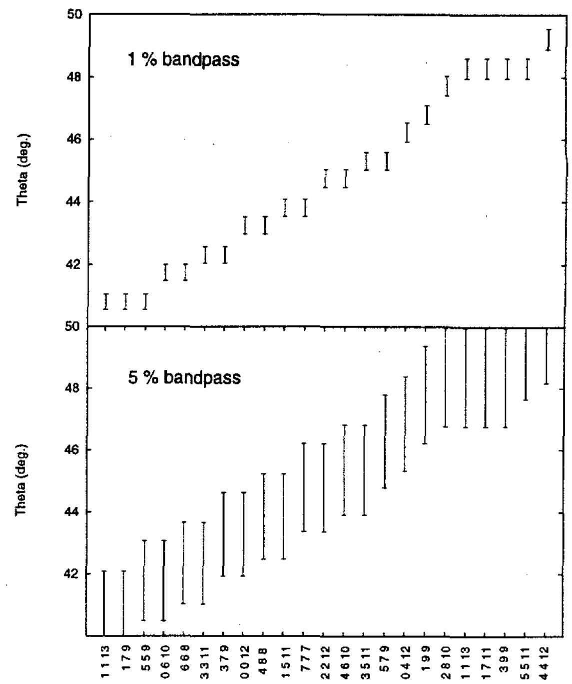

Figure 3: Changes in the ranges of angles for different bandpasses. Each line represents the range for the reflection written at the bottom.

of a small bandpass is that it generally doesn't generate enough Laue points. To measure the strain tensor, at least three non-coplanar reflections have to be measured; with an undulator source, energy may have to be scanned to collect enough reflections. The scanning of energy requires extra measuring time, and would reduce the advantage of using a CCD.

Wider bandpasses can be achieved by using off axis synchrotron radiation or a tapered undulator[7]. Even though indexing is slightly more complicated with wider bandpass, more than three linearly independent reflections can be measured in a single image. With a 10% bandpass around 20 keV, a single Laue image from an fcc lattice has enough points to determine the unit cell, yet indexing time was negligible. Wide bandpass Laue images seem to be more efficient for microdiffraction experiments.

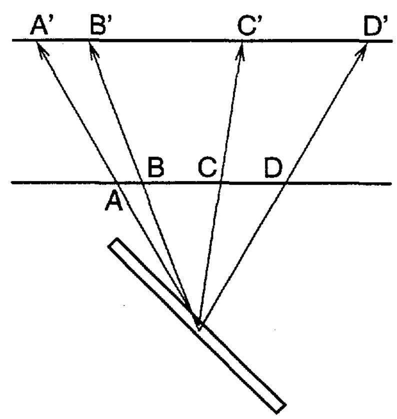

Figure 4: Ray tracing method in finding the center of diffracted beams

Scanning x-ray microdiffraction using wide bandpass Laue images can be done by automating the following procedures. First, since the accurate distance between the sample and the CCD screen is essential, at least two images have *to* be taken at two different distances. The distance and the center of diffracted beams can be derived by using ray tracing methods as shown in Fig. 4. Then from the image, an automated algorithm has to find and fit the precise positions of the Laue spots. Typical difficulties are eliminating backgrounds from the image and finding the centers of distorted spots. Next the Laue spots must be indexed. This critical step is covered in detail in the next section. From the indexes of the Laue spots, the orientation and strain can be derived. By repeating these procedures at different locations in the sample, a scanned map of strain and orientation can be achieved.

AUTOMATED INDEXING

We have developed an algorithm that generates possible sets of indexes from the Laue spot locations, the known crystal structure, and the lattice constants of a unstrained crystal. For a Si (001) wafer, it was able to find indexes uniquely.

Our algorithm is based on the fact that the direction of the scattering vector $\vec{q}$ can be measured from $\hat{k}_{out}$, the direction vector of the diffracted beam. Since we also know $\hat{k}_{in}$, the direction vector of the incident beam, the direction of $\vec{q}$ can be derived from

$$\vec{q} \parallel \vec{k}_{out} - \vec{k}_{in} \tag{2}$$
$$|\vec{k}_{out}| = |\vec{k}_{in}|.$$

Thus, the angles between $\vec{q}$'s can be derived from the locations of Laue spots. Later, these *measured angles* can be compared with the angles between possible indexes of reflections.

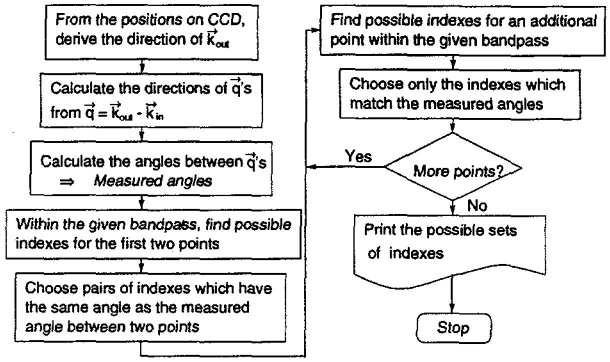

Figure 5: The flow chart of automated indexing

As the indexes get larger, the reflections becomes weaker because the atomic scattering factor diminishes for large scattering vectors. In addition, the incident beam wavelength sets an upper bound on $|h^2 + k^2 + l^2|$. Thus only small integers like integers between -20 and 20 need to be considered as possible indexes. By imposing a limited bandpass on the incident beam, only manageable number of indexes need to be considered for each reflection. In our calculation, with a 10% bandpass, each reflection has $200 \sim 500$ possible indexes. The angles between possible indexes are calculated and compared with *the measured angles* one by one. Usually when angles of more than three points are checked, there are only a few choices left. The algorithm is summarized in Fig. 5.

In a test of our algorithm for Si (001) crystal, a conventional x-ray tube with W target was used. To simulate the 10% bandpass of a tapered undulator, a pair of Mo and Zr foils were calibrated as balanced filters[8]. The image with the Mo filter was subtracted from the image with the Zr filter. The result is shown in Fig. 6. White dots represent diffracted beam with energy between 18 to 20 keV. Some black dots also showed up

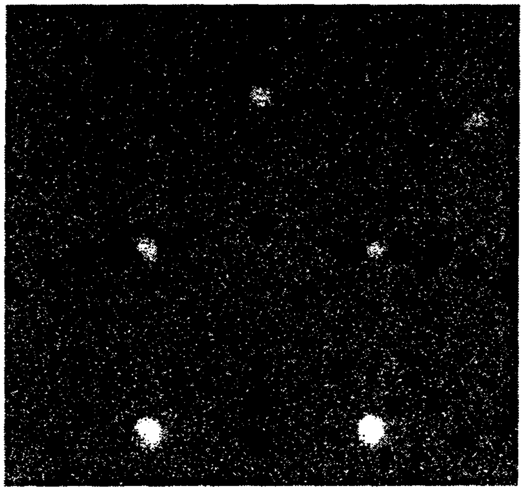

Figure 6: Subtracted Laue image of Si (001) crystal with balanced Mo & Zr filters

in the image because of imperfect balancing for $h\nu > 20$keV.

As a reference, a program called *laueX* written by Alain Soyer[9] was used to get a simulated Laue image. The result of the simulation for Si crystal in the (001) orientation is presented in Fig. 7 with all the indexes.

The result of automated indexing is summarized in Table 1 and Table 2. The key parameter in this calculation appears to be the bandpass of x-ray energy. The bandpass should allow enough Laue points to specify the unit cell, but generate only manageable number of possible indexes. In this example, the 10% bandpass worked well. Table 1 shows how our algorithm narrows down the possibilities as we increase the number of points of which angles were compared. After three or four points, it generated almost a unique set of indexes. This is expected from the fact that three non-coplanar reflections define the unit cell parameters and the orientation.

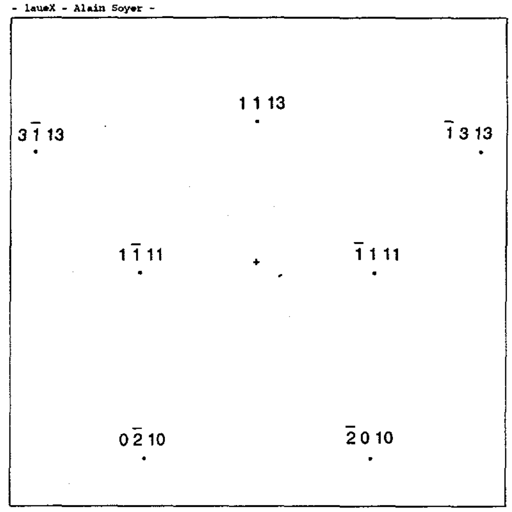

Figure 7: Simulated Laue pattern from *laueX* written by Alain Soyer [9]

Table 1: Progress of automated indexing with increased number of points for Si (001) crystal. N_{band}, the number of possible indexes only with bandpass requirement. N_{angle} is the number after comparing angles.

Points	N_{band}	N_{angle}
1st	452	452
2nd	503	324
3rd	367	67
4th	264	2
5th	246	2
6th	367	2

Table 2: Final results of automated indexing

Point	Simulation	Auto-indexing
1	1 1 13	1 1 13
2	1 $\bar{1}$ 11	1 $\bar{1}$ 11 or $\bar{1}$ 1 11
3	$\bar{1}$ 3 13	3 $\bar{1}$ 13 or $\bar{1}$ 3 13
4	$\bar{1}$ 1 11	1 $\bar{1}$ 11 or $\bar{1}$ 1 11
5	0 $\bar{2}$ 10	0 $\bar{2}$ 10 or $\bar{2}$ 0 10
6	$\bar{2}$ 0 10	0 $\bar{2}$ 10 or $\bar{2}$ 0 10

Since our algorithm uses arc-cosines to decide angles, two indexes with mirror symmetries cannot be distinguished. Because of that, the number of final results are two in most cases. As shown in Table 2, the final results are identical to that of the simulation except additional points with mirror symmetries. The whole process took only less than 2 seconds of CPU time with a 200MHz Pentium Pro processor.

For multi-grain Laue images, this algorithm can be generalized. Some reflections may not find any index which satisfies the angle requirements because they are from different grains. These points are left out and the program continues to index as many points as possible. Later the same algorithm can be re-applied to get separate sets of indexes from the points left out. Different sets represent different grains in the crystal.

SUMMARY

We have shown that our algorithm can index wide bandpass Laue images quickly. This is a key step towards automated scanning x-ray microdiffraction. The result of indexing for Si (001) crystal was identical to that of a simulated Laue image. The bandpass of the incident x-ray beam is critical to measurement efficiency; it has to be wide enough to get more than three Laue spots in a single image, but narrow enough not to leave too many possible indexes.

Acknowledgments

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[9] Alain Soyer, *laueX*, Laboratoire de Minéralogie - Cristallographie associé au CNRS, Université P. et M. Curie et D. Diderot, Place Jussieu, Paris Cedex 05, France. This Unix-based program can be downloaded from `ftp.lmcp.jussieu.fr` in `/pub/sincris/software/general/laueX/`.