MultiLaue: A Technique to Extract d-spacings from Laue XRD

Zack Gainsforth[1], Matthew A. Marcus[2], Nobumichi Tamura[2] and Andrew J. Westphal[1]

[1.] University of California Berkeley, Space Sciences Laboratory, Berkeley, CA, 91020, USA
[2.] Lawrence Berkeley Laboratory, Advanced Light Source, Berkeley, CA, 91020, USA

Broad spectrum X-ray Diffraction (XRD) is named Laue after Max von Laue, and is the original XRD technique [1]. Today, monochromatic XRD is more common because Bragg's equation allows determination of d-spacings where Laue does not. Laue still remains in use for single crystal systems because it can be used to make very accurate unit cell determinations as well as for strain and orientation mapping. A Laue technique which could provide unambiguous determination of lattice spacings, a la Bragg's equation would be a huge leap forward, especially for multiphase samples such as meteorites, interplanetary dust particles and some geological specimens.

We introduce a new technique we call multiLaue which allows such determination. First an unfiltered Laue pattern is acquired. Then a filter is inserted between the radiation source and the sample – in this case a 100 um thick fused silica wafer – and a new Laue pattern is acquired. A shield protects the detector from radiation scattered off the filter. Low energy X-rays are more strongly absorbed by the filter than high energy X-rays so reflections excited by low energies are more attenuated relative to those excited by high energies. We developed a computer model simulating the process, and use it to quantitatively determine the X-ray energy exciting a reflection based on the reduction of intensity across a set of three filters (100, 200 and 300 micron fused silica).

We tested multiLaue on a Si chip on beamline 12.3.2 at the Advanced Light Source synchrotron. The unfiltered pattern is shown in Figure 1. We analyzed 32 reflections (Table 1) and computed best fit energies. These are compared with energies based on indexation via the XMAS software [2]. The best fit energies matched well between 11 and 23 keV, with a mean relative error of 1.9% and a standard deviation of the error of 1.4%. This means we can compute d-spacings with an accuracy of < 5% (2σ) within this region. Figure 2 shows the flux curve of the beamline overlaid with the relative errors for calculations of reflections at different energies. The flux curve is poorly characterized above 21 keV, and reflections with energies > 21 keV have large errors. Reflections < 11 keV have a harmonic contribution from the spectrum above 21 keV and also show large errors. Therefore, accurate characterization of the radiation source, and optics is crucially important to achieving high accuracy quantification.

References:

[1] Laue, von, M. Physikalische Zeitschrift, 14, (1913), p. 1075–1079.
[2] Tamura, N. In Strain and Dislocaton Gradients from Diffraction Spatially-Resolved Local Structure and Defects , ed. R. Barabash & G. Ice, (Imperial College Press, London) p. 125.
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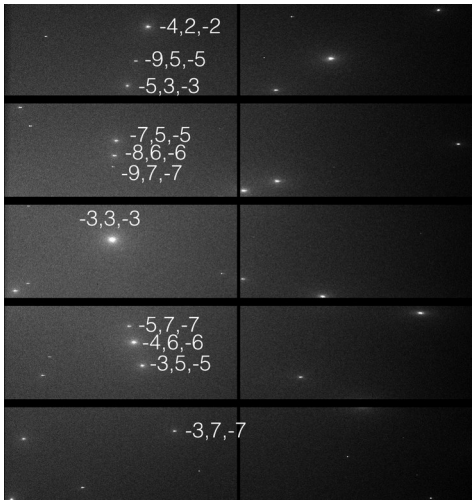

Figure 1: Unfiltered Laue XRD pattern of Si with one row indexed.

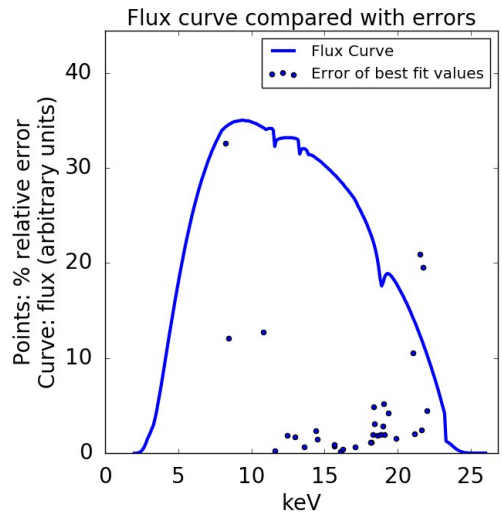

Figure 2: Flux curve of 12.3.2 plotted alongside relative errors computed at different energies.

hkl	Energy (eV)	Fit energy (eV)	% rel error		hkl	Energy (eV)	Fit energy (eV)	% rel error
-2,4,-2	12473	12240	1.87		-8,6,-6	18976	18430	2.88
-3,3,-1	11619	11650	0.27		-3,5,-7	13591	13500	0.67
-4,2,-2	8259	10950	32.59		-6,6,-8	17111	17220	0.64
-3,3,-3	8449	9470	12.09		-9,5,-3	21629	22160	2.46
-5,3,-1	14519	14300	1.51		-7,5,-5	16223	16290	0.41
-6,4,-2	16108	16080	0.18		-3,7,-7	18124	17910	1.18
-3,5,-3	14380	14040	2.37		-5,7,-7	18414	17850	3.06
-6,6,-4	18211	18000	1.16		-10,6,-8	21054	23280	10.57
-5,5,-3	15664	15540	0.79		-6,8,-10	21501	26000	20.93
-5,3,-3	10826	9450	12.71		-9,5,-5	19049	18050	5.24
-4,6,-6	15673	15530	0.91		-3,7,-9	18629	18980	1.88
-3,5,-5	12968	12740	1.76		-9,5,-7	18378	19280	4.91
-3,7,-5	19108	18730	1.98		-5,7,-9	18819	19190	1.97
-7,5,-3	18276	17920	1.95		-9,7,-7	21745	26000	19.57
-6,8,-6	21981	21000	4.51		-7,7,-9	19861	20170	1.56
-5,7,-5	19332	20150	4.23		-11,5,-7	21174	21600	2.01

Table 1. List of reflections which were calculated using our model. "hkl" identifies the reflection. "Energy" is the actual energy of the first harmonic of the reflection, "Fit energy" is the energy determined from the Laue images. "% rel error" is the relative error between the two energy values.