Conf-9110146--8

Submitted to: 7th National Conference on
Synchrtron Radiation Instrumentation
Baton Rouge, LA October 28-31, 1991

BNL--47048

DE92 007167

Measurement of rocking curve wings at high x-ray energies

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Abstract

Measurements done recently at the NSLS have indicated that the level of intensity found in the wings of diffraction peaks from silicon at higher x-ray energies (>20keV) far exceeds the value which would be predicted based on the dynamical theory. We have measured Si(220) double crystal rocking curves at the 40keV fundamental and harmonics with various crystal scattering geometries: Bragg-Bragg, Laue-Bragg, Laue-Laue. The comparison of the Bragg and Laue case diffraction geometries was done to determine scattering volume effects. Comparisons with dynamical theory calculations will be discussed.

These measurements have been carried out in order to assess the level of harmonic contamination which will be present from a double crystal monochromator being designed for the X17 Superconducting Wiggler Beamline.

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Introduction

Recently we have done measurements to assess the performance of various monochromator double crystal geometries at higher x-ray energies (>20keV).

Originally measurements were done with a silicon (220) Bragg type double crystal arrangement [1] to determine the level of harmonic contamination which would be present as a function of detuning of the second monochromator crystal [2]. In an effort to model the expected throughput of a double crystal monochromator as a function of crystal detuning we found that the measured intensities in the wings of the rocking (or detuning) curve far exceeded predictions based on dynamical theory. At 40% detuning of the 40keV fundamental from a silicon (220) the measured first harmonic at 80keV from the Si(440) was a factor of 10 higher than the predicted value and the second harmonic at 120keV was a factor of 100 higher than the prediction.

In an effort to better understand the origin of the excess scattering in the rocking curve wings at higher x-ray energies, we undertook a study of various crystal scattering types. The crystal arrangements investigated were Laue-Laue (LL), Laue-Bragg (LB) and Bragg-Bragg (BB). The first crystal type was the fixed monochromator crystal and the second crystal type was the analyzer crystal which was scanned to give the rocking curves.

The intent was to show that the excess scattering was due to thermal diffuse scattering (TDS) of the x-rays. At high x-ray energies the absorption length, l_a, of the incident x-ray beam (6mm at 40keV) allows the off-Bragg peak beam to penetrate deep into the crystal and enhance the probability of TDS scattering to occur for a Bragg type crystal. However for a Laue type crystal the scattering volume is limited by the crystal thickness. If the crystal is thin compared to the absorption length but thick compared to the extinction length then good intensity can be obtained while minimizing the total scattering volume.

For the Bragg case the scattering volume is proportional to $Al_a/2$, where A is the cross-sectional area of the beam and l_a is the absorption length [3]. For the thin crystal Laue case the scattering volume is At; proportional to the crystal thickness, t, and beam area, A.

For the cases reported here at 40keV, the ratio of scattering volume for the Bragg case crystals compared to the Laue case is about 10. Therefore, one might expect the intensity of the wings for the BB, LB and LL at a given rocking angle away from the central peak to follow the approximate relation: BB = 10xLB = 100xLL.

In all cases the TDS should fall off with rocking angle. The TDS has a $1/q^2$ dependence on relative scattering vector, q, away from the Bragg reciprocal lattice vector. For small angle deviations away from the Bragg peak, then we expect that the rocking angle will be proportional to q.

Experimental

The measurements were done at the NSLS Superconducting Wiggler beamline in the materials science hutch, X17B1. The angular resolution required to obtain the sub-arc-second rocking curves was achieved by using a high-energy spectrometer [4]. The axes of this spectrometer were driven by long tangent arms pushed by stepper motor controlled micrometers giving a minimum step size of 0.02 arc-second. The crystals were float zone silicon which were strain relief mounted to the axes.

Long steel collimators on the incident beam path, between crystals and after the second crystal helped reduce the background level reaching the detector after the second crystal. The detector was an intrinsic germanium detector 17mm thick which gave good stopping power and energy resolution at 40keV and the harmonics. The 40keV fundamental from the Si(220) and the 80keV first harmonic were simultaneously recorded as a function of rocking angle of the second (analyzer) crystal. The use of a solid state detector also helped control the background in the measurement to a few counts per minute. Single channel analyzers were used to energy analyze the amplified detector pulses.

The count rates were limited by filtering the incident beam on the first crystal such that the count rate at the fundamental very nearly equaled the count rate at the first harmonic. Any further filtering would only enhance the intensity of the first harmonic relative to the fundamental. If only filtering is used to control the total count rate to a pileup free value (approx. 10kcps) then there is very little intensity left at the fundamental wavelength. This makes the measurement very difficult to perform since the statistics are poor for the fundamental and the spectrum is altered if filters are removed to increase the intensity for the far wings of the rocking curves.

The total count rate in this measurement was limited by a variable horizontal width tungsten slit upstream of the first crystal. Using a horizontal slit will not alter the spectral content of the filtered synchrotron beam striking the first crystal nor will the horizontal divergence of the beam affect the diffraction properties of the double crystal system since the scattering occurs in the vertical plane. The V-wedge shaped slit was mounted such that moving the wedge vertically would prepare a variable horizontal width beam. The rocking curve of the Bragg peak was taken at near minimum horizontal beam width (approx. 0.1mm). In the far wings where the count rate was low the horizontal beam width was increased (approx. 1.0mm) as well as the counting time per point and the step size per point. This allowed us to obtain better statistics in a shorter data acquisition time. At no time was the total count rate allowed to exceed 10kcps.

With this system the total +/- 100 arc-second scan range was made up of seven overlapping scans; one central peak scan with three symmetric scans on the high and low angle sides of the central peak.

This procedure allowed us to measure in a nearly pileup free way the wings and Bragg peak over a wide dynamic range without a significant background contribution.

Once the basic setup and scan ranges were established, then all three crystal geometries reported here were measured in a similar fashion. It should be noted that the factors which scaled the wing scans to each other are the same for all three geometries (LL, LB and BB).

Results

Data were taken at the 40keV Si(220) fundamental and also at the 80keV Si(440) harmonic. Figure 1 shows the data as taken for the LL (figure 1a), LB (fig. 1b) and BB (fig. 1c). The solid lines are the data for the fundamental and the dotted line are for the first harmonic. A logarithmic vertical axis was chosed to emphasize the wings of each. However, the logarithmic axis does not show the structure of the rocking curve near the peak region. Table 1 shows the measured and calculated rocking curve widths for the three crystal geometries for the 40keV fundamental. It is interesting to note that the measured BB width is somewhat narrower than the calculation. Three measurements of the BB rocking curve at later times gave similar widths to the measured 0.88 arc-second value quoted. However, for the LL and LB cases the measured and calculated values give good agreement.

Figure 2 shows a comparison of the LL, LB and BB cases for the 40keV fundamental alone. This figure shows the surprising result that the BB case has the lowest intensity wings in conflict with the hypothesis that the excess scattering in the wings was related to scattering volume effects. In the far wings the order of intensity is: LB > LL > BB. In the wing region below 20 arc-seconds the intensity order is: LL > LB > BB. In either case the BB case has the lowest wing contribution.

Figure 3 shows the calculation for the same scattering cases for comparison with figure 2. The convoluted throughput [5] shown here has wings which drop much more rapidly than any of the measured throughputs as expected based on the previous results [2].

The next figure (fig. 4) shows excess scattering which is the difference of the calculated intensities from the measured values. Here the plotted data is the average of the wings on the high and low angle side of the central peak. The LL case has the lowest excess scattering which indicates that part of the measured wing contribution is a result of the thin crystal Laue case diffraction properties. However, the LB case has the highest excess scattering and the BB case has an intermediate value in conflict with the TDS scattering hypothesis.

Finally, the last figure (fig. 5) shows the reciprocal of the square root of the excess scattering shown in figure 4. The purpose of this plot is to graphically indicate if the excess scattering has a $1/q^2$ dependence indicative of TDS which would appear as a straight line on this plot. None of the cases display a true $1/q^2$ behavior, though at relatively small rocking angle (<20 arc-seconds) the dependence is nearly so.

Conclusions

In conclusion, we have measured the rocking curves of various case double crystal scattering geometries (Laue-Laue, Laue-Bragg and Bragg-Bragg) in an effort to understand the origin of excess scattering in the wings of the profiles. We believe that the excess scattering is due to a sample volume effect which is more important at higher x-ray energies due to beam penetration. When the excess scattering is plotted the order of intensities does not completely follow the order expected for a sample volume effect alone (measured order of intensities: LB > BB > LL as compared to the expected result of BB > LB > LL).

We are planning future measurements to confirm the results reported here and hope to understand better the origin of the excess scattering in the rocking curve wings.

Acknowledgments

This work was performed under US Department of Energy contract number DE-AC02-76CH 00016 to the National Synchrotron Light Source, Brookhaven National Laboratory.

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FIGURES

Figure 1

Rocking curves for Si(220) at 40keV (solid lines) and Si(440) at 80keV (dotted lines) for the Laue - Laue (figure a), Laue - Bragg (figure b) and Bragg - Bragg (figure c) cases.

Figure 2

Comparison of the measured Si(220) rocking curves at 40keV for the Laue-Laue (solid line), Laue-Bragg (dotted line) and Bragg-Bragg (dashed line) cases.

Figure 3

Comparison of the calculated Si(220) rocking curves at 40keV for the Laue-Laue (solid line), Laue-Bragg (dotted line) and Bragg-Bragg (dashed line) cases.

Figure 4

Excess scattering (difference of the measured and calculated intensities) for the Si(220) at 40keV as a function of rocking angle away from the central peak for the Laue-Laue (solid line), Laue-Bragg (dotted line) and Bragg-Bragg (dashed line) cases.

Figure 5

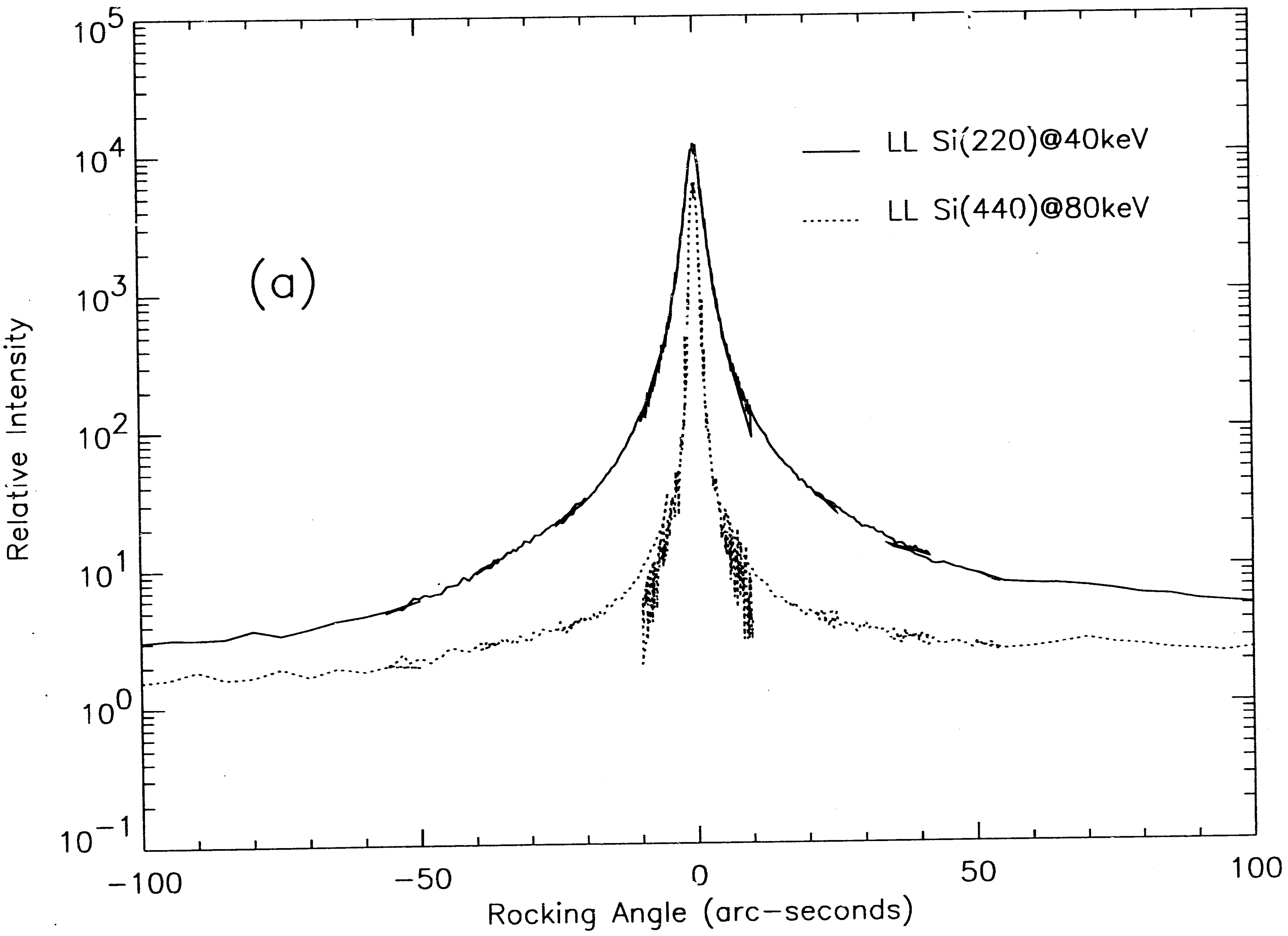
Plot of the reciprocal of the square root of the excess scattering from figure 4 for the Laue-Laue (solid line), Laue-Bragg (dotted line) and Bragg-Bragg (dashed line) cases.

TABLES

	FWHM Widths (arc-seconds)	
Crystal Arrangement	**Measured**	**Calculated**
Laue - Laue (LL)	2.40	2.10
Laue - Bragg (LB)	2.10	1.87
Bragg - Bragg (BB)	0.88	1.33

Table 1

Comparison of measured rocking curve widths with calculated widths for the LL, LB and BB cases for the Si(220) at 40keV.



(a)
LL Si(220)@40keV
LL Si(440)@80keV
Relative Intensity
10^{5}
10^{4}
10^{3}
10^{2}
10^{1}
10^{0}
10^{-1}
−100
−50
0
50
100
Rocking Angle (arc−seconds)

FIGURE 1a.

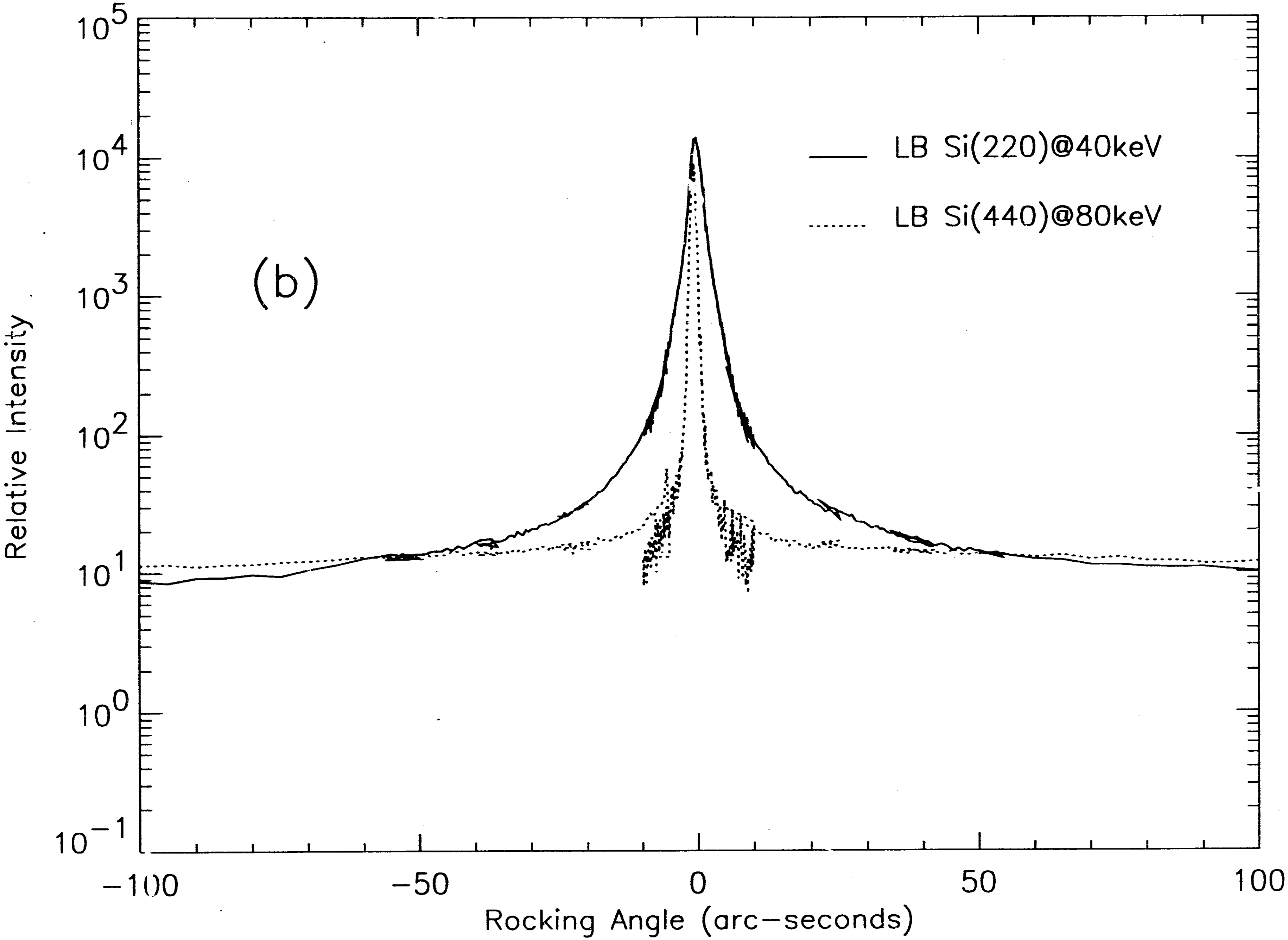

(b)
LB Si(220)@40keV
LB Si(440)@80keV
Relative Intensity
10^{-1}
10^{0}
10^{1}
10^{2}
10^{3}
10^{4}
10^{5}
−100
−50
0
50
100
Rocking Angle (arc−seconds)

FIGURE 1b.

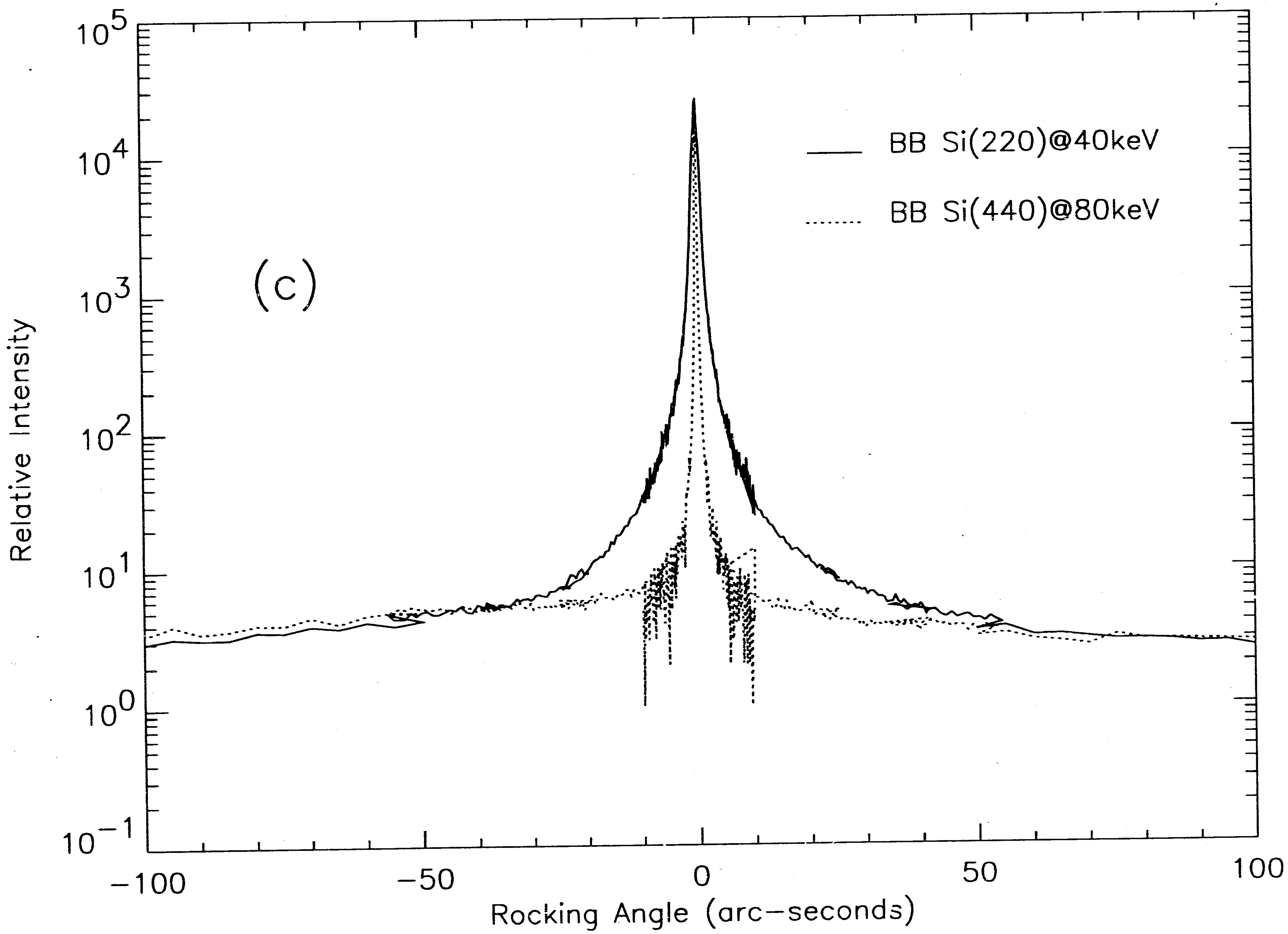

(c)
BB Si(220)@40keV
BB Si(440)@80keV
Relative Intensity
10^5
10^4
10^3
10^2
10^1
10^0
10^{-1}
−100
−50
0
50
100
Rocking Angle (arc−seconds)

FIGURE 1c.

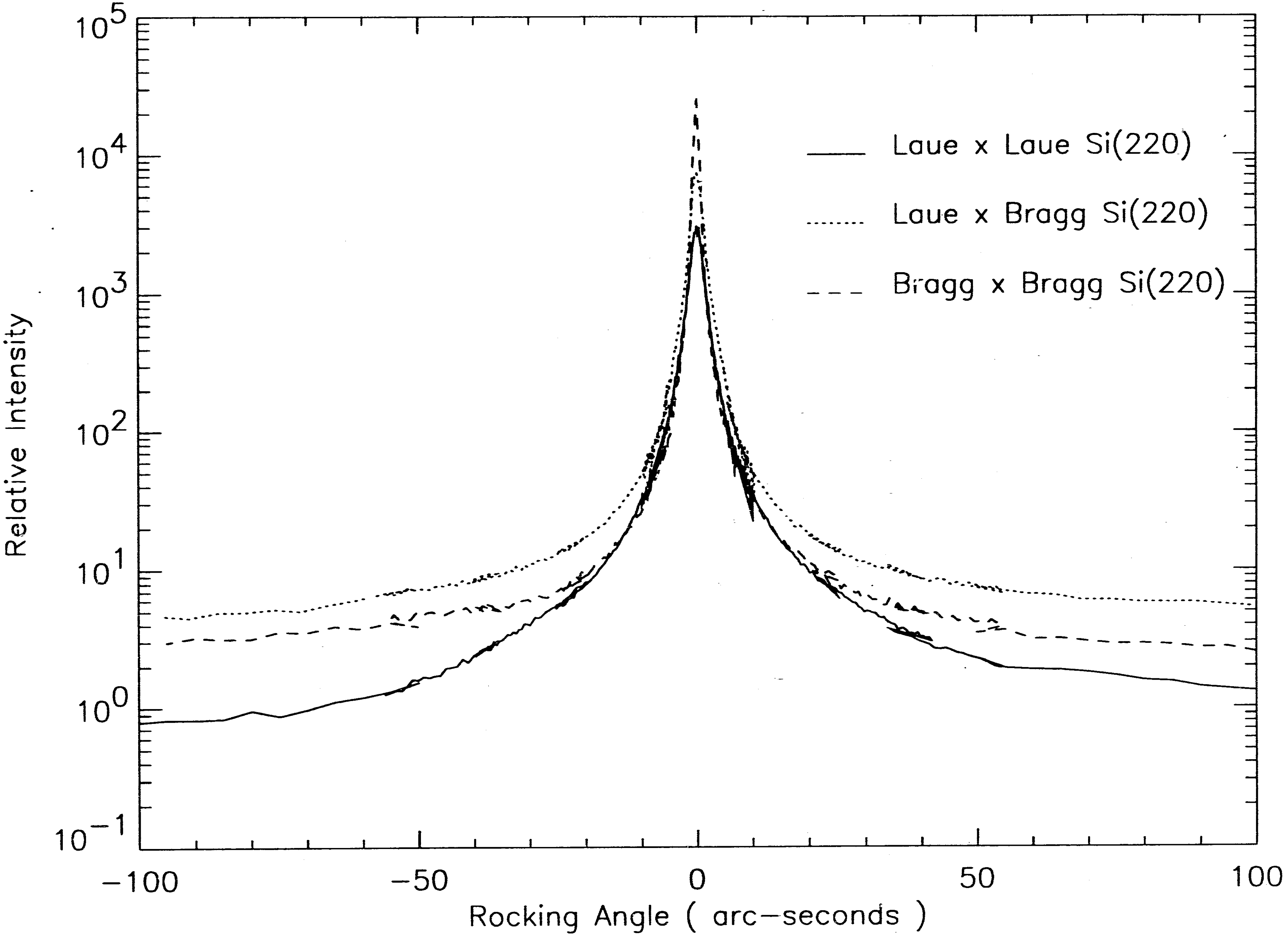
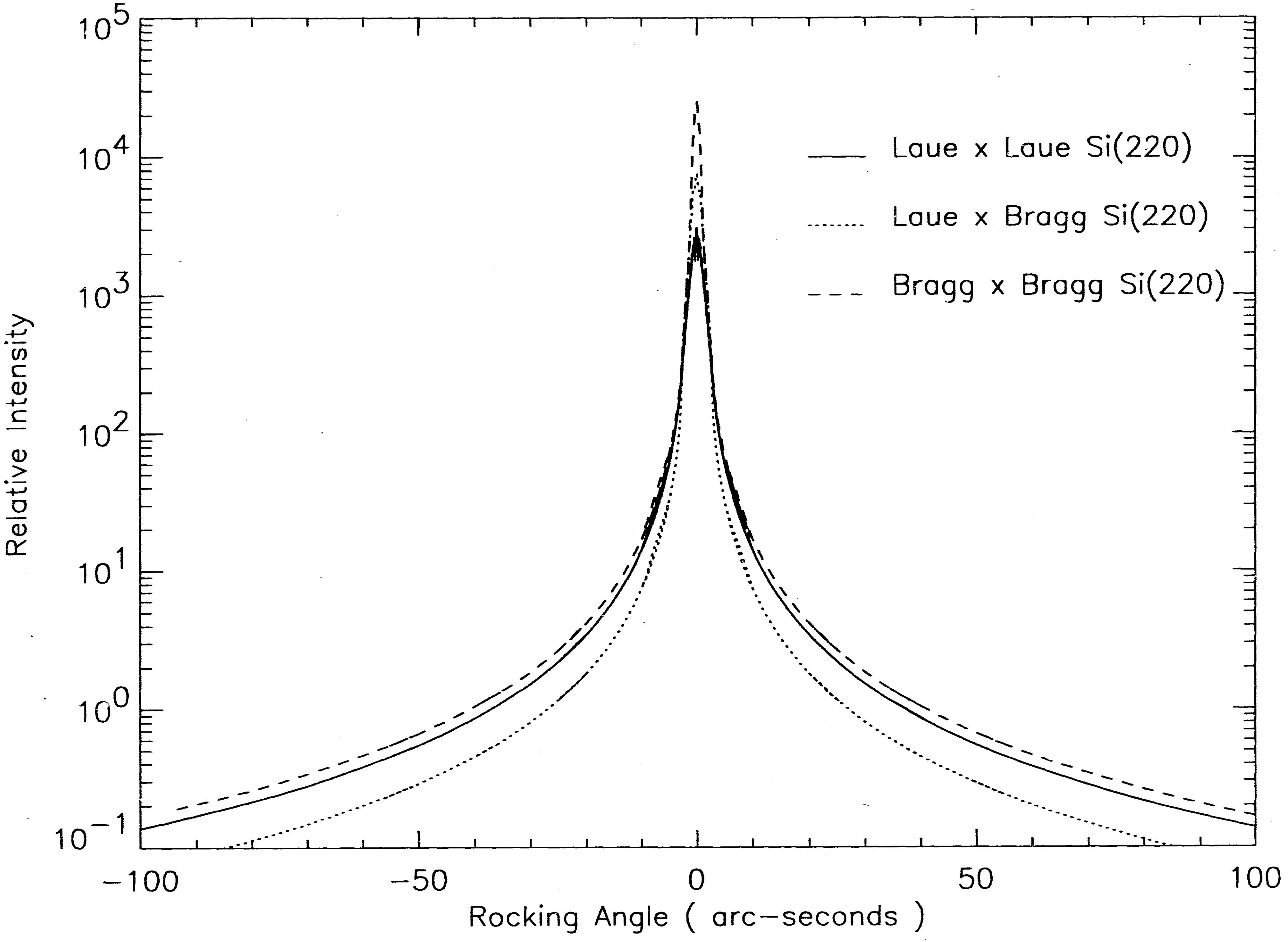

Laue x Laue Si(220)
Laue x Bragg Si(220)
Bragg x Bragg Si(220)
10^5
10^4
10^3
10^2
10^1
10^0
10^{-1}
Relative Intensity
−100
−50
0
50
100
Rocking Angle (arc−seconds)

FIGURE 2.



10^5
10^4
10^3
10^2
10^1
10^0
10^{-1}
Relative Intensity
Laue x Laue Si(220)
Laue x Bragg Si(220)
Bragg x Bragg Si(220)
−100
−50
0
50
100
Rocking Angle (arc−seconds)

FIGURE 3.

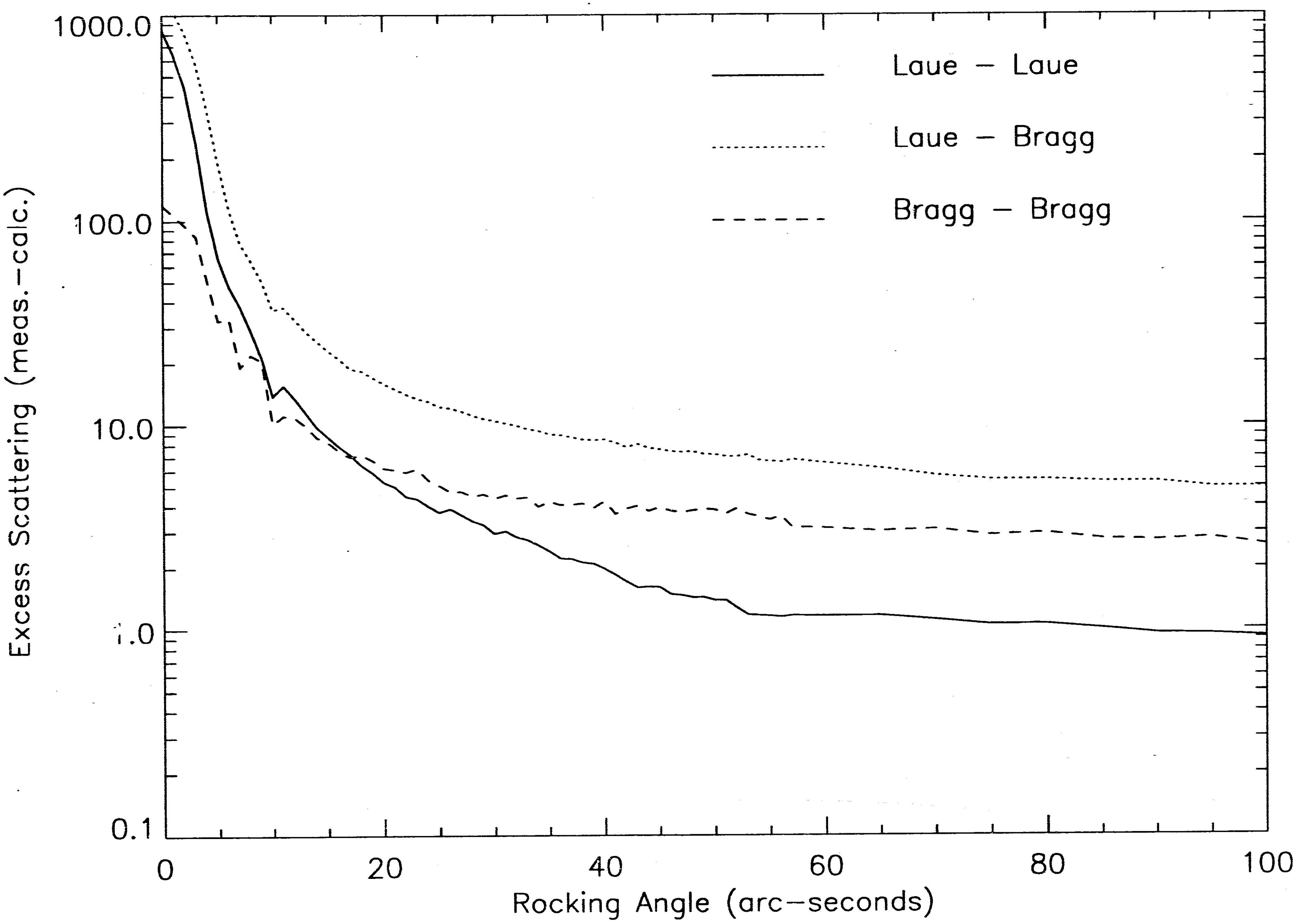

Excess Scattering (meas.–calc.)
1000.0
100.0
10.0
1.0
0.1
0
20
40
60
80
100
Rocking Angle (arc–seconds)
Laue – Laue
Laue – Bragg
Bragg – Bragg

FIGURE 4.

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