Goals and results of the SNL crystal calibration work

Goals

1) Measure accurate rocking curve of bent crystals to help the understanding of measurements taken on Z (opacity, photoionized plasmas, non-thermal emission, liner spectra…)
   e.g The iron opacity measurements on Z require integrated reflectivity, crystal resolution, and 2^{nd} to 1^{st} order reflectivity ratio (2^{nd} order correction below 950eV)

2) Evaluating absolute source intensities, instrumental broadening, plasma line-widths.

3) Develop intuition and a path to model crystal performance that could be used in a larger parameter space (various crystal curvatures, geometries, orders, photon energy…)

Results

1) We accurately measured integrated reflectivities for a set of KAP crystal curvatures (flat, 2,4, 6 and 9in) and diffraction orders (1^{st}, 2^{nd} and 3^{rd})
2) Integrated reflectivities show good agreement with XOP multilamellar model with a set of Debye-Waller factors (temperature factors) depending on crystal curvature and order of diffraction. It is possible that they could also depend on photon energy but the present data is too limited to be conclusive.
4) Width for 2^{nd} and 3^{rd} order agree relatively well with XOP multilamellar model
5) Widths in 1^{st} order are systematically measured higher than any calculation due to extra instrumental broadenings, this might be solved through deconvolution.
6) Width and spectral shape in 1^{st} order are measured with a conventional x-ray source
7) Crystal efficiencies were measured to high accuracy using NIST calibrated KERMA source

Crystal spectrometric properties are required to unfold instrumental effects in HEDP experiments

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Crystal spectrometric properties are:

1) Crystal integrated reflectivity (R_{int})
2) Crystal resolution and spectral shape

These are readily obtained through the measurement of crystal ***rocking curve***, nevertheless either one quantity is more readily measurable accurately

Outline of this presentation

1) Synchrotron beamline measurements → R_{int} and some resolution
2) Uncalibrated conventional x-ray source → resolution and spectral shape
3) Calibrated conventional x-ray source → R_{int}

1) Using the ALS LBNL beamline to measure crystal rocking curve and probe the effect of bending in KAP

Rocking curve calibrations principle and requirements

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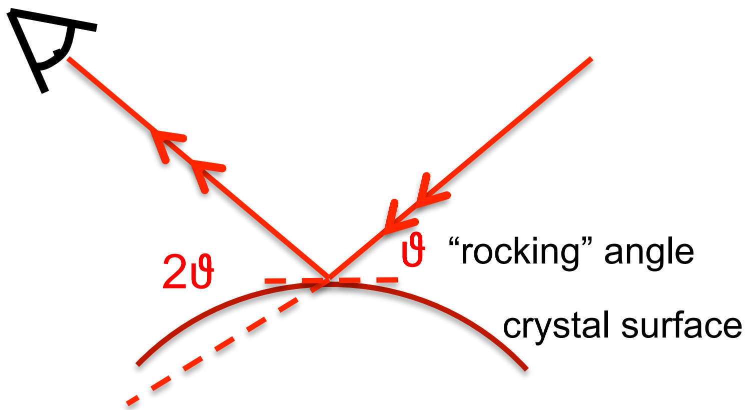
Principle of measurement



1) Select photon energy (double-monochromator)
2) measure I_0 with the crystal out of the way
3) rock the crystal (ϑ) and detector (2ϑ) and measure reflected counts

Requirements

Integrated reflectivity

1) No significant higher order contamination in the counts
2) Rock the crystal about a fixed point (alignments crystal, beam, stages)
3) High x-ray flux for reasonable S/N and fast data collection

Shape and width → limit systematic broadenings introduced by other effects

1) beam intrinsic spectral width small (high spectral purity)
2) beam size small
3) bending effect small or understood
4) keep misalignments small

Rocking curve calibrations principle and requirements

Principle of measurement

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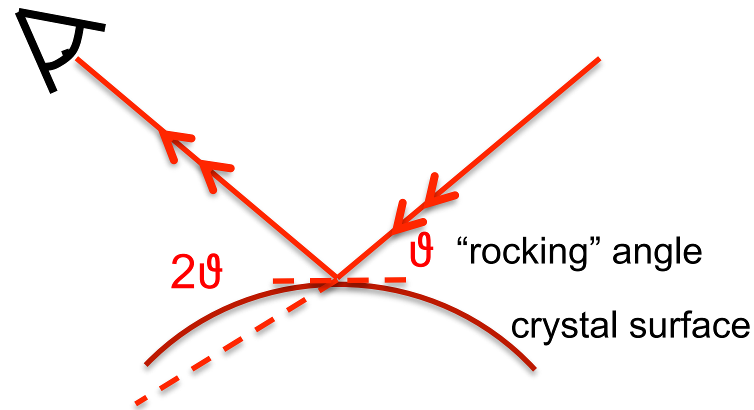

Requirements

Integrated reflectivity

1) No significant higher order contamination in the counts ✔✔ (ALS+ detector)
2) Rock the crystal about a fixed point (alignments crystal, beam, stages) ✔ (precision stage)
3) High x-ray flux for reasonable S/N and fast data collection ✔✔✔ (ALS)

Shape and width → limit systematic broadenings introduced by other effects

1) beam intrinsic spectral width small (high spectral purity) ✔✔✔ (ALS beamline)
2) beam size small ✔ (pinhole and collimated beamline)
3) bending effect small or understood
4) keep misalignments small

need to be evaluated

Predicted effect of bending on integrated reflectivities

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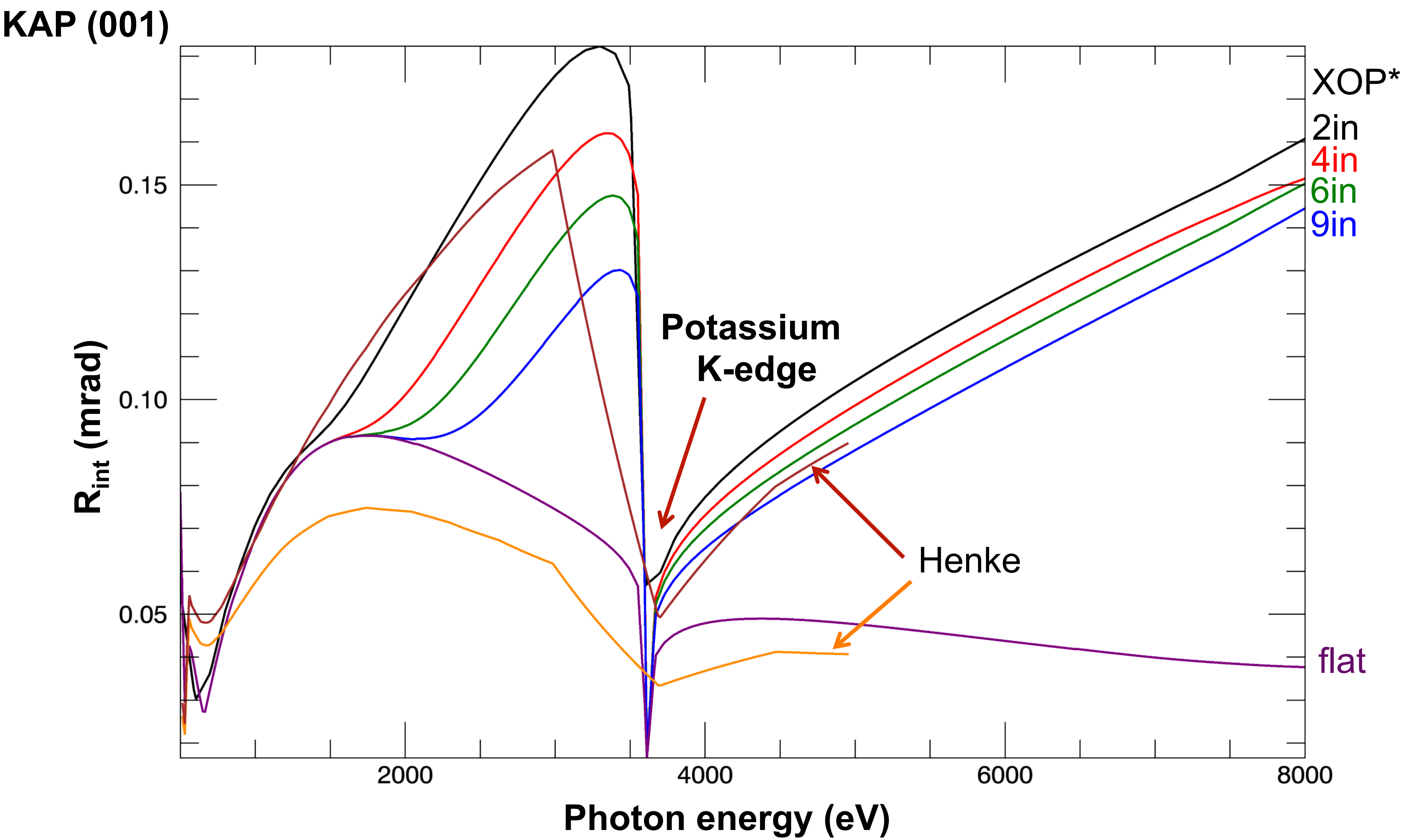
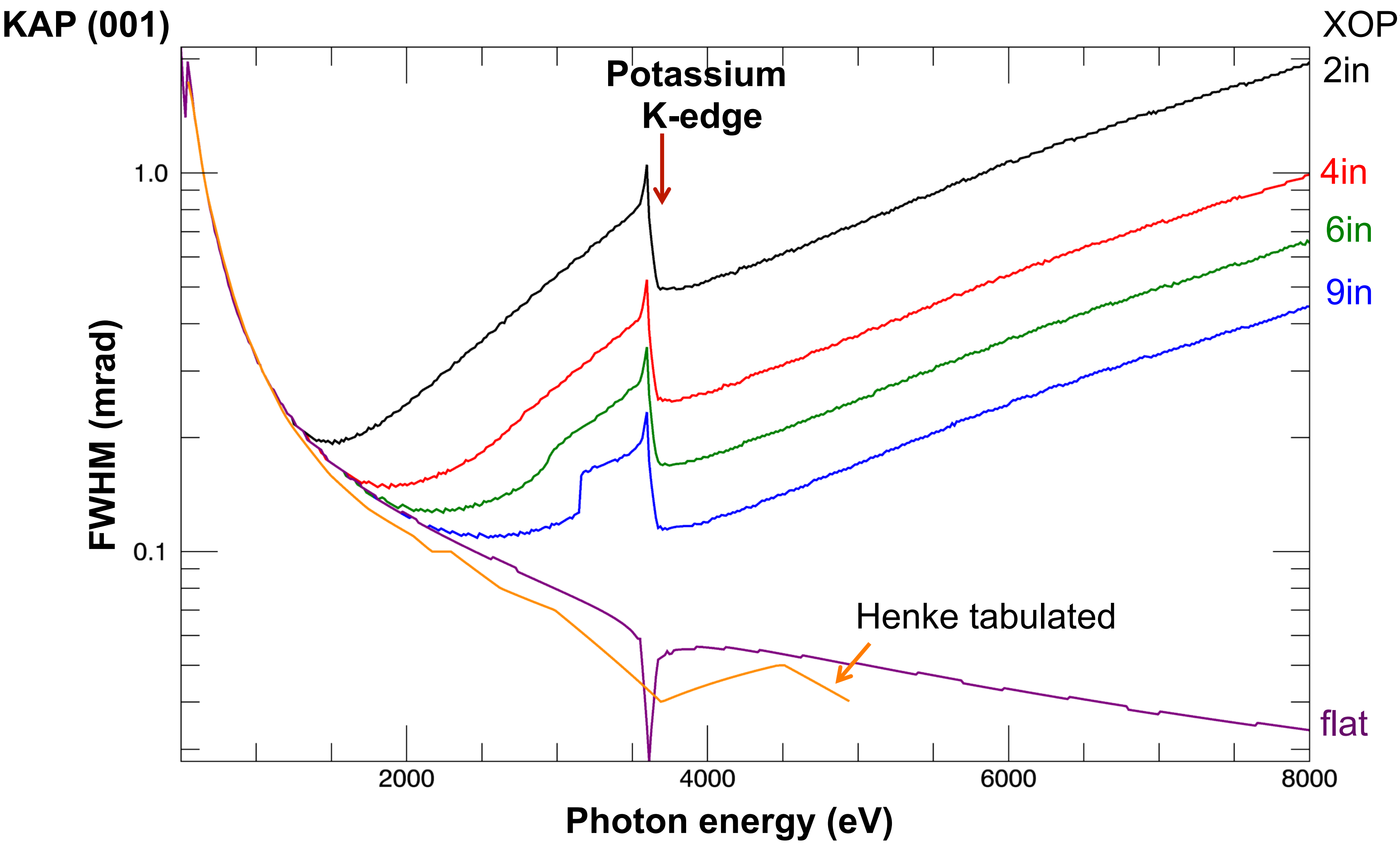


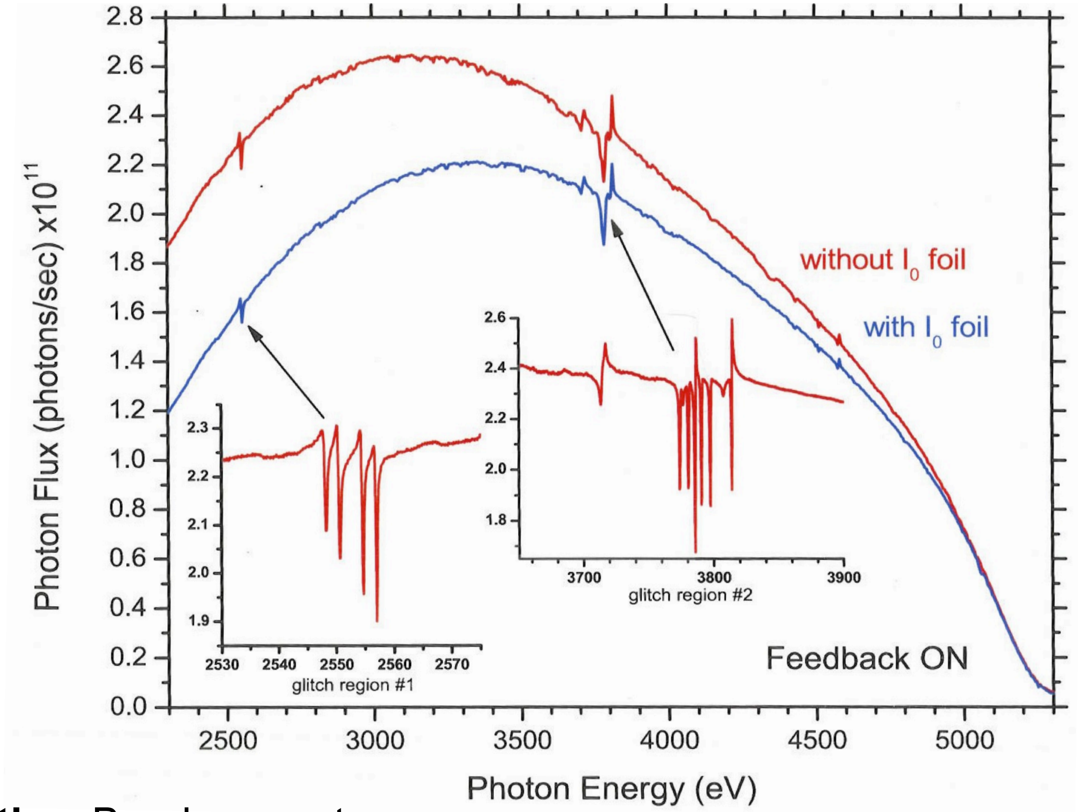
*XOP : Sánchez del Río, Dejus, SPIE proceedings (2011) 8141. Sánchez del Río I. U. Cr (2015)

Predicted effect of bending on FWHM



*XOP : Sánchez del Río, Dejus, SPIE proceedings (2011) 8141. Sánchez del Río I. U. Cr (2015)

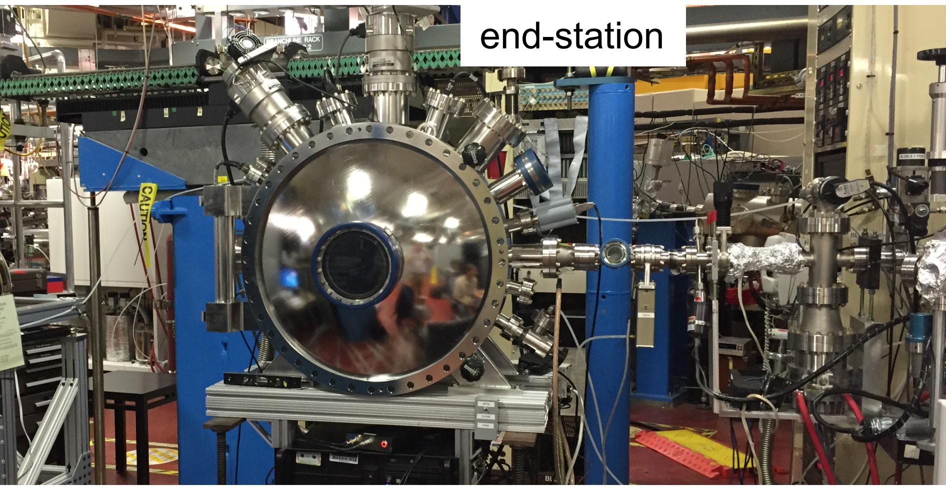
The ALS beamline 9.3.1 has high photon flux in 2.3-5.keV

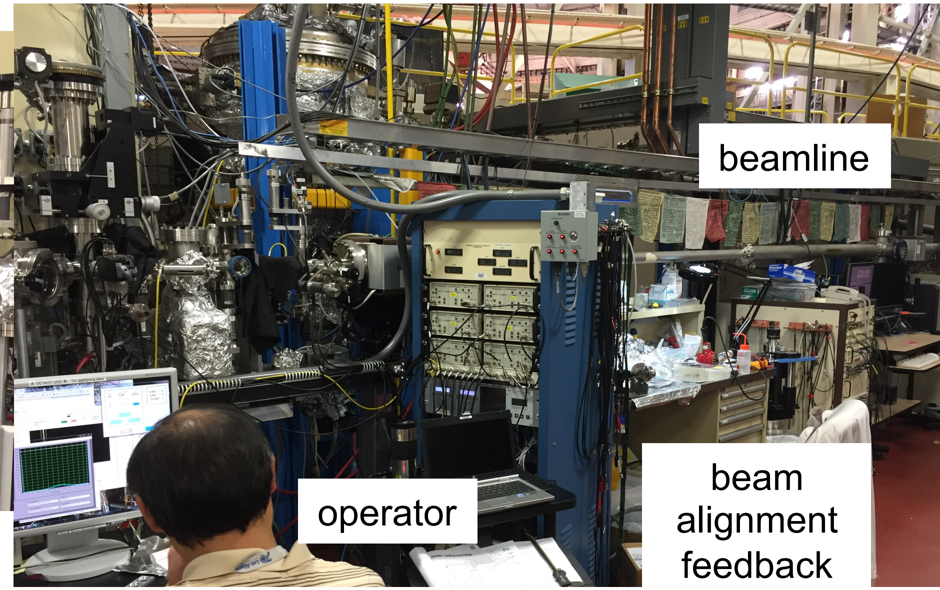


Source characteristics Bend magnet
Energy range 2.3-5.2 keV
Monochromator Double Si(111) crystal
Measured flux (1.9 GeV, 300 mA) 10^{11} photons/s in 30 bunches mode.
Resolving power (E/ΔE) 3000-7200
Beam size Adjustable with 2nd mirror
Focused: 1.0 mm x 0.7 mm (~0.5 mm square at 2800 eV)
Unfocused: 10 mm x 10 mm or larger

ALS beamline 9.3.1 experimental setup

Ring electron current 35.2mA ± 0.3mA, high stability, ~24/7





Monochromator

2x Si(111) crystals , energy range 2300eV-5200eV , resolving power 3500-7000

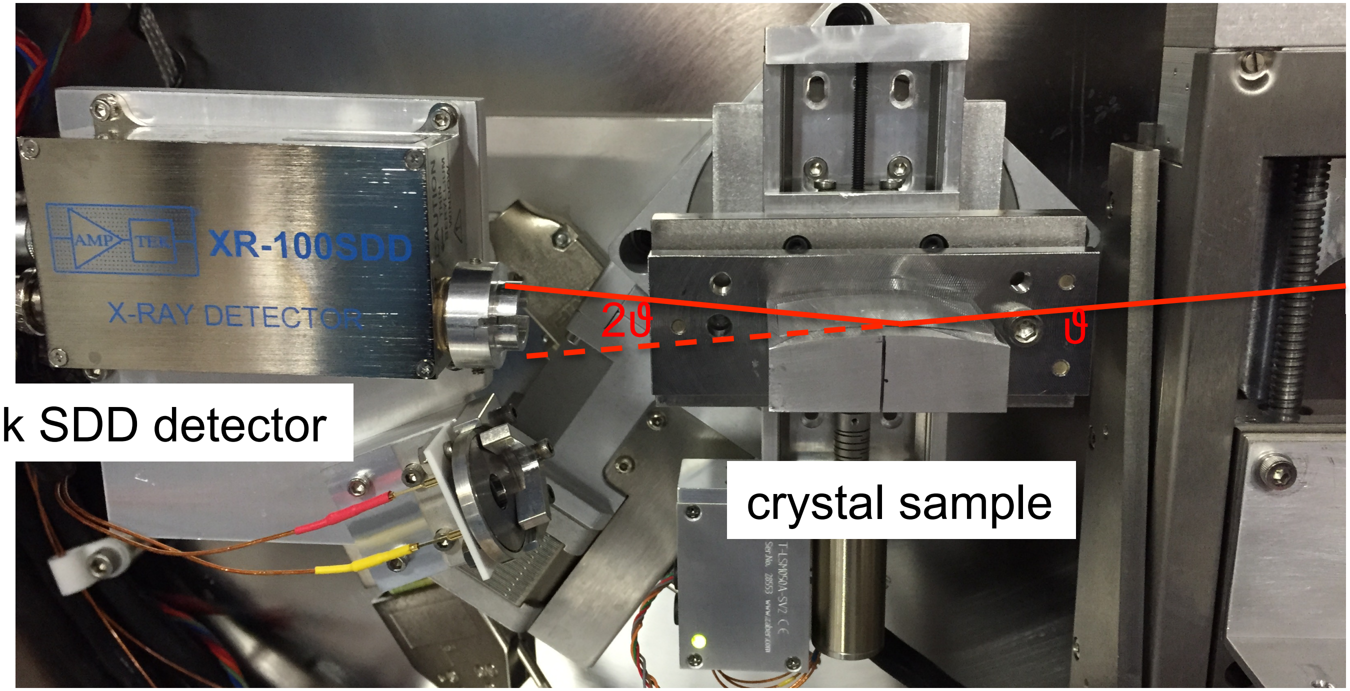
Imager

x-ray beam imaged (4mrad width, ½ mrad height) at the crystal location

imager = 2 Ni toroidal mirrors (1m long each), Mag=1

grazing 11mrad incidence (rejection at > ~5.4keV)

Calibration end-station



pinhole mount

Amptek SDD detector

crystal sample

Crystal stages: **ϑ** rotation (Bragg angle), resolution=0.001°, z translation
Detector stage: **2ϑ** rotation, res=0.005°
Pinhole mount : vertical translation
Pinhole sizes : 5.0, 3.0 or 1.2μm

Crystals: Saint Gobain crystal **flat, 2, 4, 6** and **9**in radius of curvature, all new

Detection and statistics

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The Amptek SDD (Silicon Drifted Detector) is an energy **dispersive** detector.
Can count up to 1e6 cts/s, linearly in 1-50keV.

Simultaneous events appear as a 'second order' signal, such that 2 counts add up together and appear as a 2x energy single count.

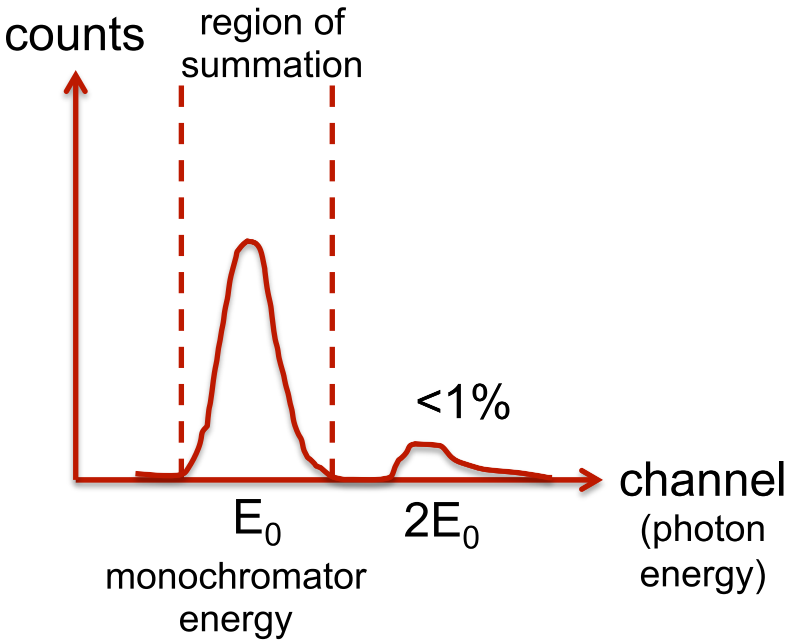
Rocking curves RC are obtained by dividing the reflected counts to the direct beam counts number at a given Bragg angle.

Rocking curve 1-σ uncertainty is inferred through Poisson statistics of counts C:

$$RC = \frac{C}{C_0} \qquad \sigma = \frac{C}{C_0}\sqrt{\frac{1}{C} + \frac{1}{C_0}}$$

with C_0 the non-reflected (direct beam) counts, assuming detector dominated statistics.

Example of detected signal at a given Bragg angle



integration time 3-30s / angle

Data can be collected 24/7

We collected ~200 rocking curves, each energy/crystal set measurement is doubled - 184 processed here

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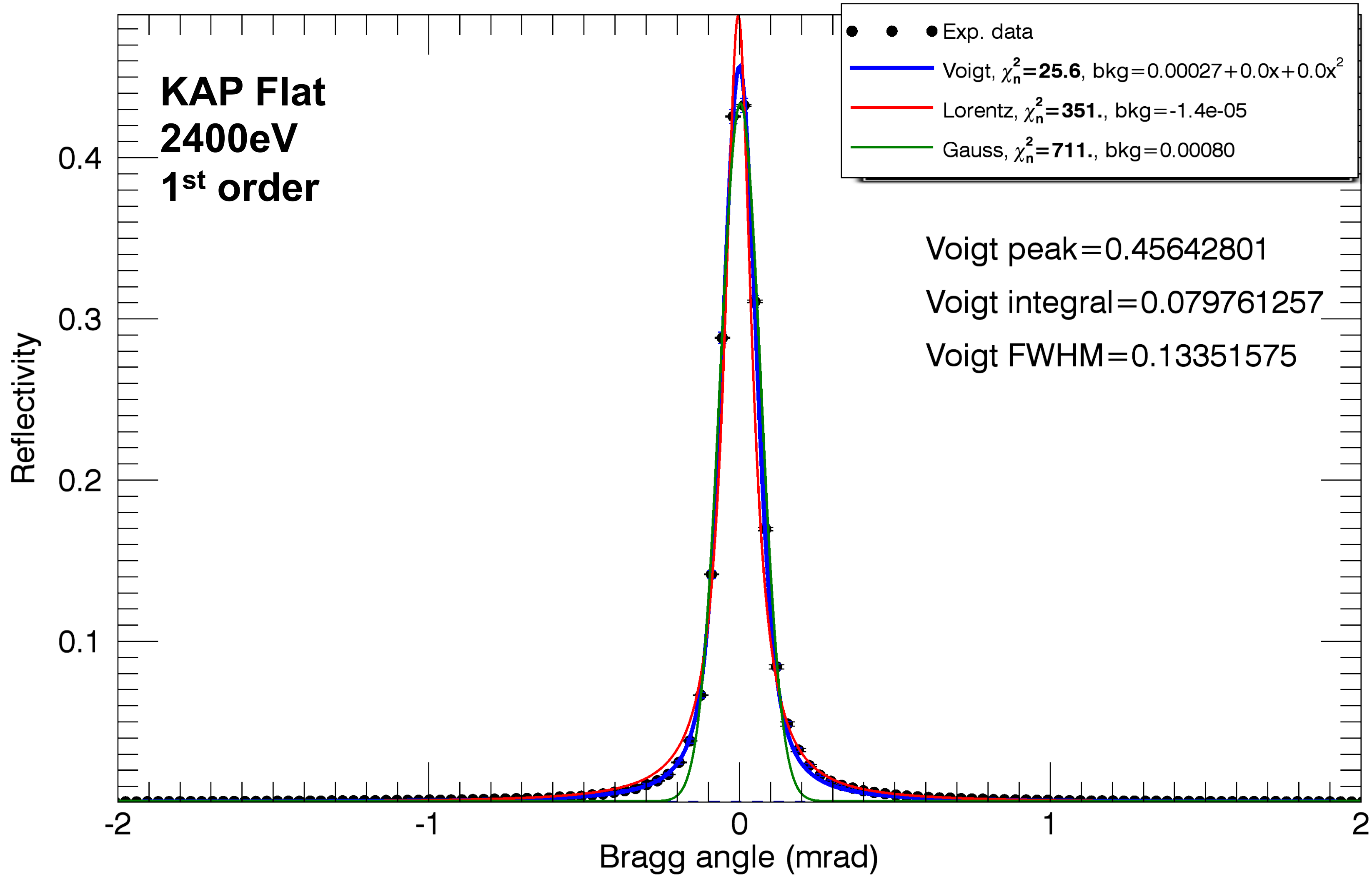
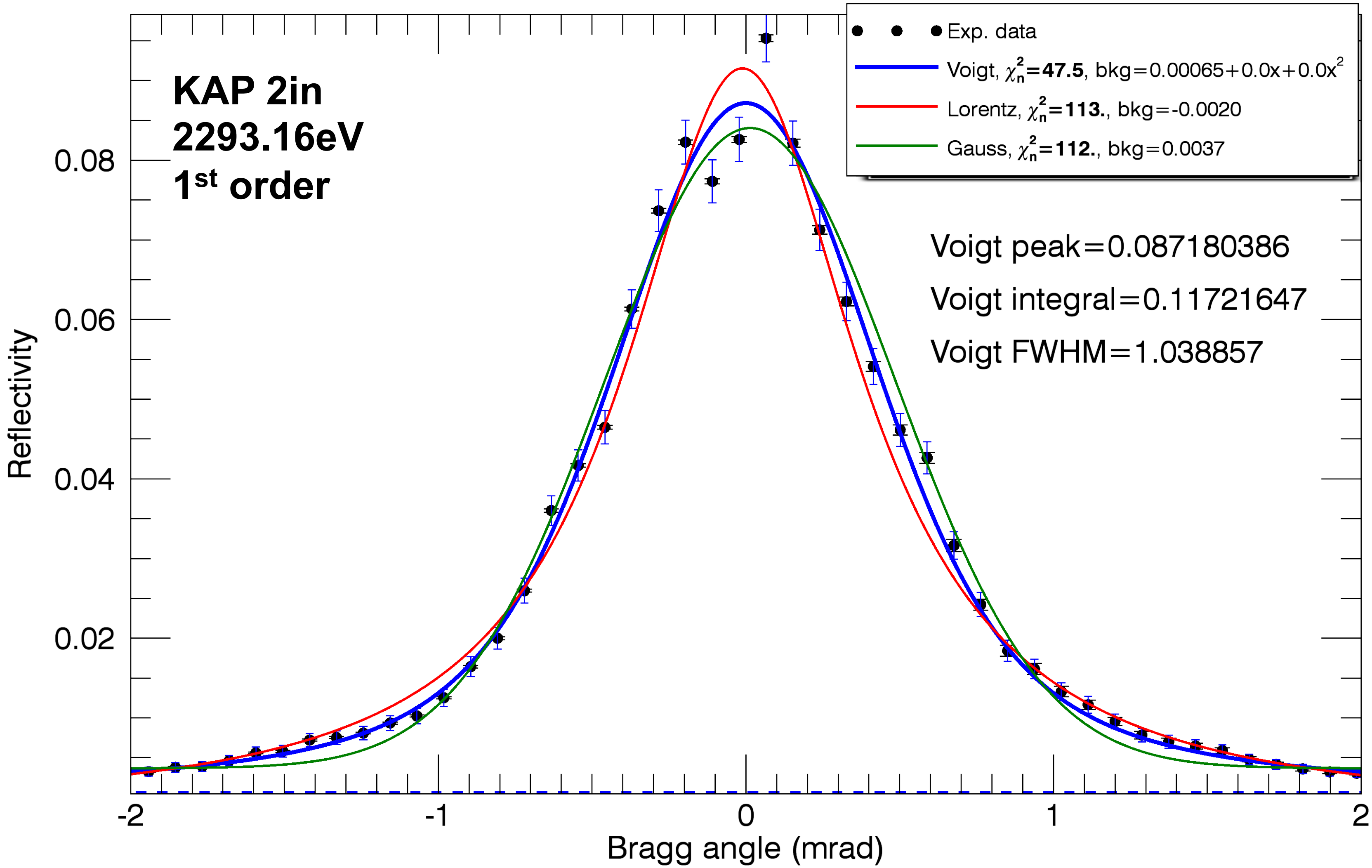
Date	Crystal radius (in)	Photon energy (eV)	Bragg angle (deg)	pinhole size (µm)	Order of diffraction	time stamp1	time stamp2	time stamp3	Rint1 (µrad)	Rint2 (µrad)	Rint3 (µrad)	FWHM1 (µrad)	FWHM2 (µrad)	FWHM3 (µrad)	Rp1 (%)	Rp2 (%)	Rp3 (%)
Tuesday April 7th	4	4510.84	5.92	5	1				64.6	62.2		601	597				
	4	4000.00	6.68	5	1	12:29	12:48		55.4	55.7		570	569			6.5	
	4	3658.40	7.31	5	1	14:07	14:24		42.9	42.7		630	634				
	4	3633.40	7.36	5	1	19:25	19:39		40.3	39.9		601.5	599				
	4	3608.40	7.41	5	1	14:47	14:59		14.1	14.4		696	667				
	4	3583.40	7.47	5	1	20:10	20:29		110	113		786	806				
	4	3558.40	7.52	5	1	15:29	15:45 --16:08 (nonuniforn grid)		111	117		809	801				
	4	2984.31	8.98	5	1	17:00	17:23		129	136		674	679				
	4	2293.16	11.71	5	1	18:23	18:39		113.8	113		496.9	491				
Wednesday April 8th	4	2293.16	11.71	5	1	14:04	X		100	X		545	X				
	4	2293.16	11.71	3	1	15:32	15:07		66	26		846	957				
	4	2293.16	11.71	1	1	16:10	X		94	X		1193	X		5.70		
	4	3558.40	7.52	3	1	21:31	X		102	X		918	X		7.2		
	4	3558.40	7.52	1	1	18:52	19:08		94	90		1215	1239		5.44		
	4	4510.84	5.92	3	1	21:07	X		49	X		1297	X		2.99		
	4	4510.84	5.92	1	1	20:00	20:22		51	50		1477	1447		2.93	2.6	
	4	2293.16	23.96	5	2	22:46	22:59		8.8	8.6		293	292		1.9	1.9	
	4	2984.31	18.18	5	2	23:31	23:45		11.6	12		393	398.2		1.98	1.97	
Thursday April 9th	6	4510.84	5.92	5	1	21:43	22:08		71	77		962	936		6.2	6	
	6	4000.00	6.68	5	1	21:14	21:27		62.5	61.2		826	856		5.6	5.1	
	6	3658.40	7.31	5	1	20:44	20:55		43.8	43.6		446	466		6.44	6.2	
	6	3633.40	7.36	5	1	20:06	20:18	2028	37.7	36.7	39.11	405	416	401	6.25	5.8	6.3
	6	3608.40	7.41	5	1	19:40	19:53		14.87	14.9		410	410		2.3	2.4	
	6	3583.40	7.47	5	1	19:05	19:23		110	109		511	510		14	14.25	
	6	3558.40	7.52	5	1	18:20	18:35	1845	126	124	121	517	503	502	16.4	16.65	16.4
	6	2984.31	8.98	5	1	11:39	11:58		125	114		525	522		17	18.2	
	6	2293.16	11.71	5	1	10:06	10:49	2223 , 2241	95	96	89 , 94	382	382	353 , 375	19.70	19.8	16 , 16.6
	6	2293.16	23.96	5	2	23:19	23:37		8.38	8.55		268	245		2.7	2.4	
	6	2293.16	37.52	5	3												
Friday April 10th	2	4510.84	5.92	5	1	18:23	18:38		61	78		x	2659		2.2	2.1	
	2	4000.00	6.68	5	1	17:48	18:00		61	64		2219	2271		2.2	2.2	
	2	3658.40	7.31	5	1	16:18	16:33		47	43		1137	1197		3	2.6	
	2	3633.40	7.36	5	1	15:52	16:03		38	39		1105	1107		2.4	2.5	
	2	3608.40	7.41	5	1	15:22	15:35		14.2	14.1		1193	1153		0.8	0.9	
	2	3583.40	7.47	5	1	14:09	14:28	1438	133	131	111	1725	1454	1538	6.5	5.7	5.7
	2	3558.40	7.52	5	1	13:22	13:41		133	127		1453	1454		6.7	6.5	
	2	2984.31	8.98	5	1	10:17	10:54		162	160		1089	1082		10	10.5	
	2	2293.16	11.71	5	1	8:13	9:07	9:30, 19:03, 19:20	113	109	110, 117, 136	965	959	956, 935, 935	9	8.5	9.2, 9.5, 10.2
	2	2293.16	23.96	5	2	19:53	20:15		8.1	7.52		567	552		1.1	1.0	
	2	2293.16	37.52	5	3	21:28			0.07298			442.3			0.0165		
	2	2293.16	11.71	3	1	0:51	1:03		122	125		1730	1702		4.9	5	
	2	3558.40	7.52	3	1	1:58	2:11		120	125		2447.8	2331		3.8	4	
Saturday April 11th	2	2293.16	11.71	5 - closer	1	9:03	9:26	20:43	145	139	121	556	549	549	16.8	16.8	16.8
	2	2984.31	8.98	5 - closer	1	14:07	14:16		152	152		761	761		15	15	
	2	3583.40	7.47	5 - closer	1	14:57	15:11	15:24	115	113	112	1015	1023	1000	8.5	8.5	8.5
	2	4510.84	5.92	5 - closer	1	16:06	16:35	16:52	80	74	68	962	919	969	6.1	5.9	5.6
	2	2293.16	11.71	3 - closer	1	18:38	18:53		107	100		902	920		9.6	8.5	
	2	2984.31	8.98	3 - closer	1	19:24	19:33		81	126		805	969		8.55	11	
	2	3583.40	7.47	3 - closer	1	19:49	19:57		99.8	101		1233	1195		7	7.1	
	2	4510.84	5.92	3 - closer	1	20:10	20:24		55	56		1476	1533		3.7	3.6	
	2	2293.16	23.96	5 - closer	2	23:00	23:12		8	8.1		365	357		1.5	1.6	
	2	2293.16	37.52	5 - closer	3	20:53	21:08		0.11	0.094		316	300.3		0.0223	0.023	
	2	3608.4	14.95	5 - closer	2	23:37	23:50		30	34.2		616	560		3.7	4.5	
Sunday April 12th	9	2293.16	11.71	5 - closer	1	11:31	11:48	12:03	100.3/96	99.2/94.1	95.4/88.3	189/229.5	192/229.8	194.6/229.8	33.1	33.1	33
	9	2984.31	8.98	5 - closer	1	13:36:00 PM	13:51	14:01	92.83	89	86.6	193.7	229.8	229.8	31.1	31.8	31
	9	3558.40	7.52	5 - closer	1	14:26	14:37	14:48	87.3/71.9	83.6/84.8	84.96	229.7/233.2	268.2/229.4	223.1	25.2	25.7	24.9
	9	3583.40	7.47	5 - closer	1	18:23	18:35	18:46	84.6	78.9	79.9	246.3	254.7	247.9/229.9	22.7	20.6	22.6
	9	3608.40	7.41	5 - closer	1	19:38	19:54	20:02	13.3	12.8	12.6	229.8	262.8	261.3	3.9	3.65	3.74
	9	3633.40	7.36	5 - closer	1	20:20	20:50	21:00	31.2	31.9	33.1	268.8	336.6	330.8	8.23	6.78	7.31
	9	3658.40	7.31	5 - closer	1	21:26	21:35		35.6	34.9		319.3	311.6		8.39	8.32	
	9	4000.00	6.68	5 - closer	1	22:03	22:16		42.1	41.4		322.6	320.5		9.85	9.6	
	9	4510.84	5.92	5 - closer	1	22:49	23:15		51.1	46.7		367.8	371.3		10.2	9.3	
	9	2293.16	11.71	5-closer	1	23:51			103.2			204.5			32.8		
	9	2293.16	23.96	5 - closer	2	0:26	0:43		8.39	8.28		156.6	161.6		3.43	3.37	
	9	3608.4	14.95	5 - closer	2	1:47	1:57	2:14	33.05	35.3	36.6	225.4	256.2	253.1	9.86	9.8	10.1
	9	[illegible]	[illegible]	[illegible]	[illegible]												

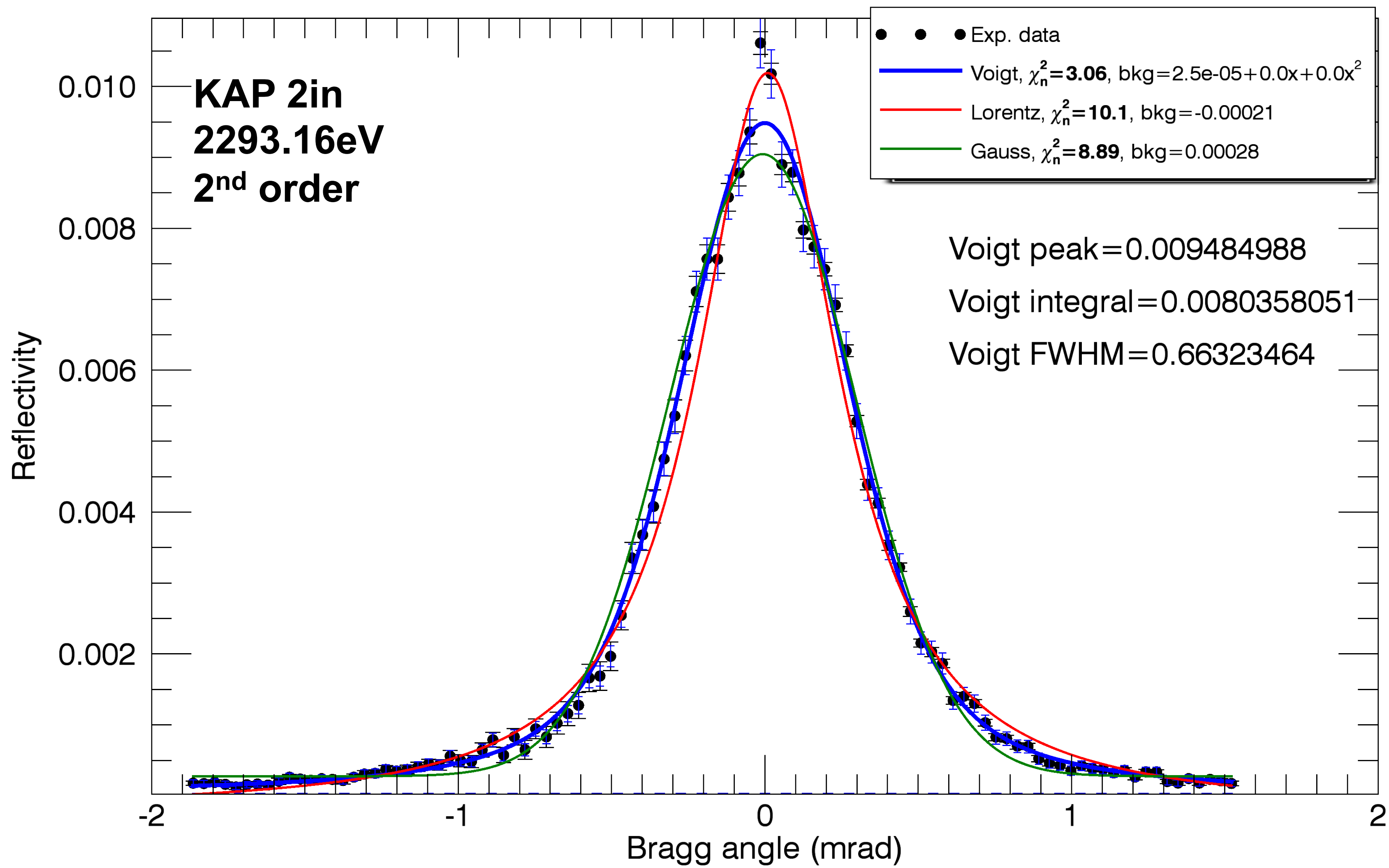
Example of rocking curve (4)



Example of rocking curve (2)



Example of rocking curve (2)



Example of rocking curve (3)

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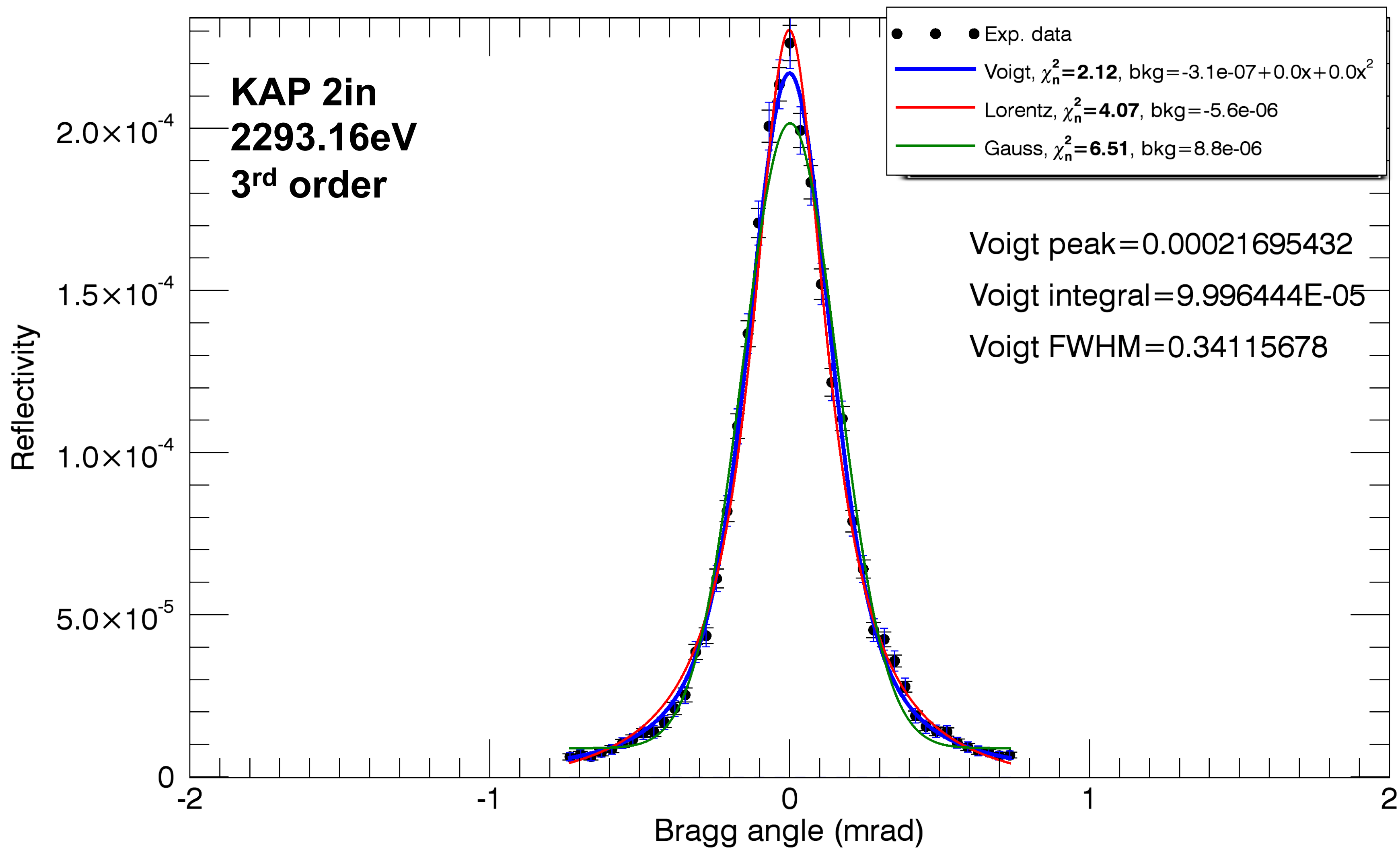
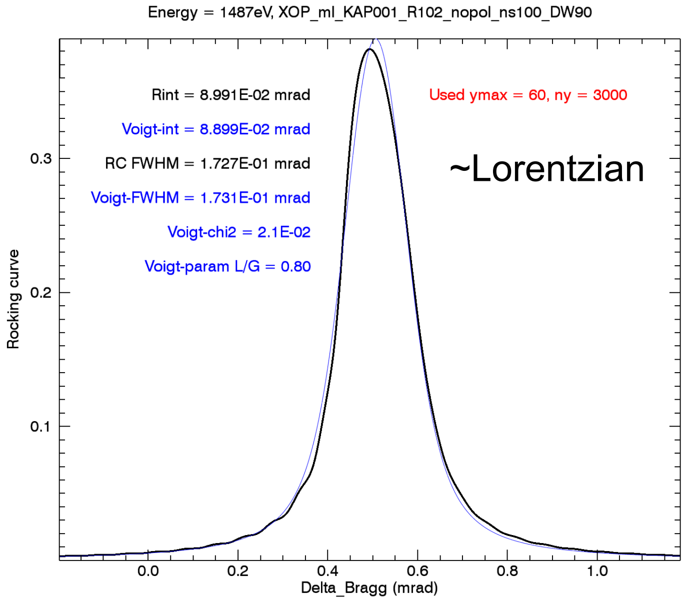


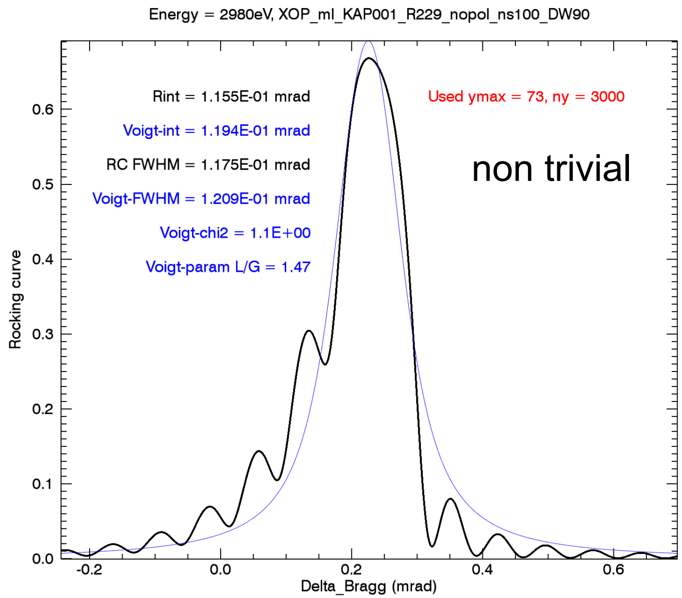
The Voigt fit parameters are compared with XOP model for R_{int} and FWHM

XOP 2.4 Multi-Lamellar model, dynamical theory of diffraction, Zachariasen equations , imperfect mosaic crystal.

- 5 curvatures: flat, 9in, 6in, 4in, 2in crystals
- orders: 1, 2, 3
- polarization: σ, π & none
- temperature factors: 0.8, 0.9, 1.0
- energy grid 500pts [2270 – 4533] eV

The KAP *c* lattice parameter was updated in XOP to correct lattice spacing and 013 plane tilt



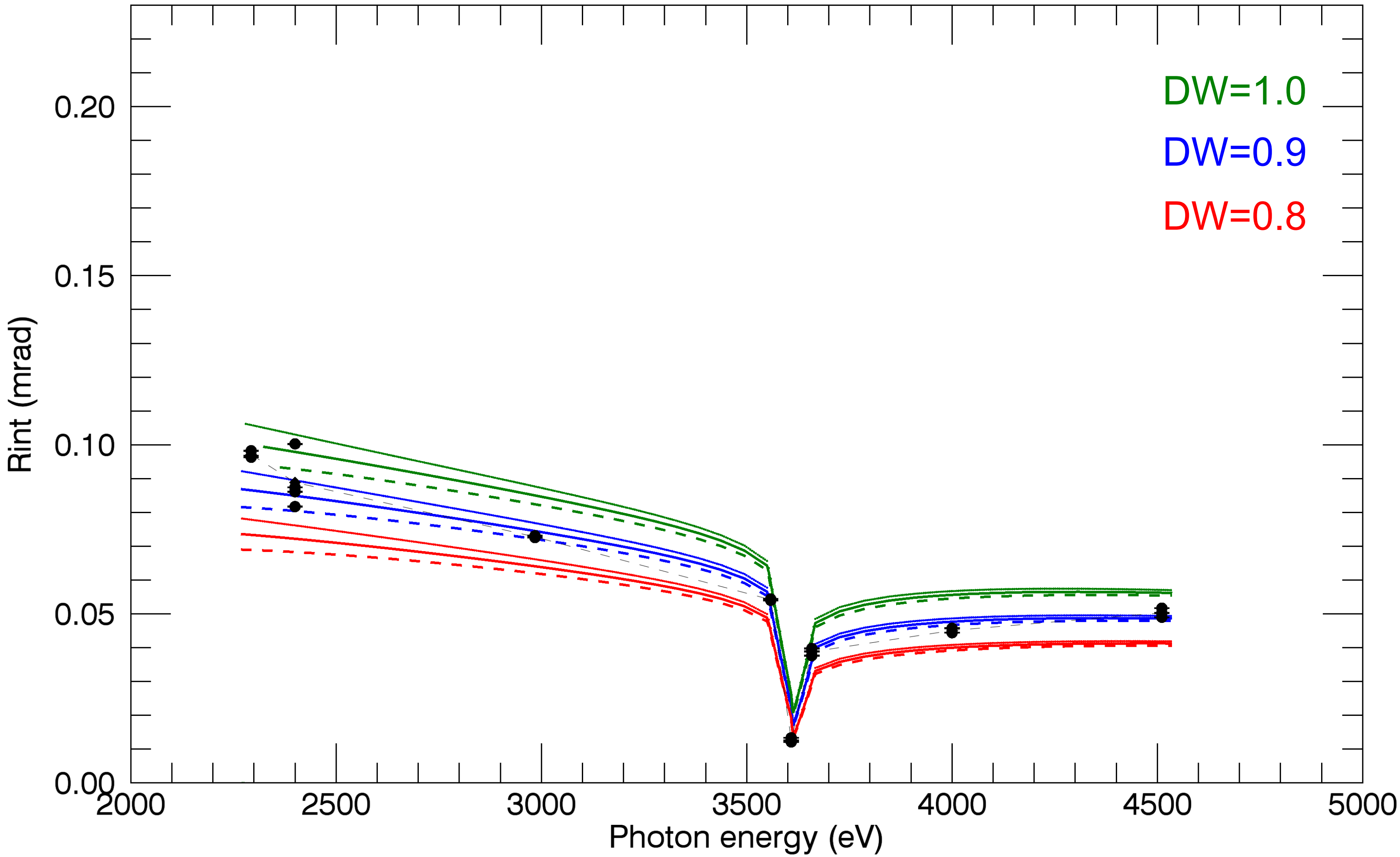


*XOP : Sánchez del Río, Dejus, SPIE proceedings (2011) 8141. Sánchez del Río I. U. Cr (2015)

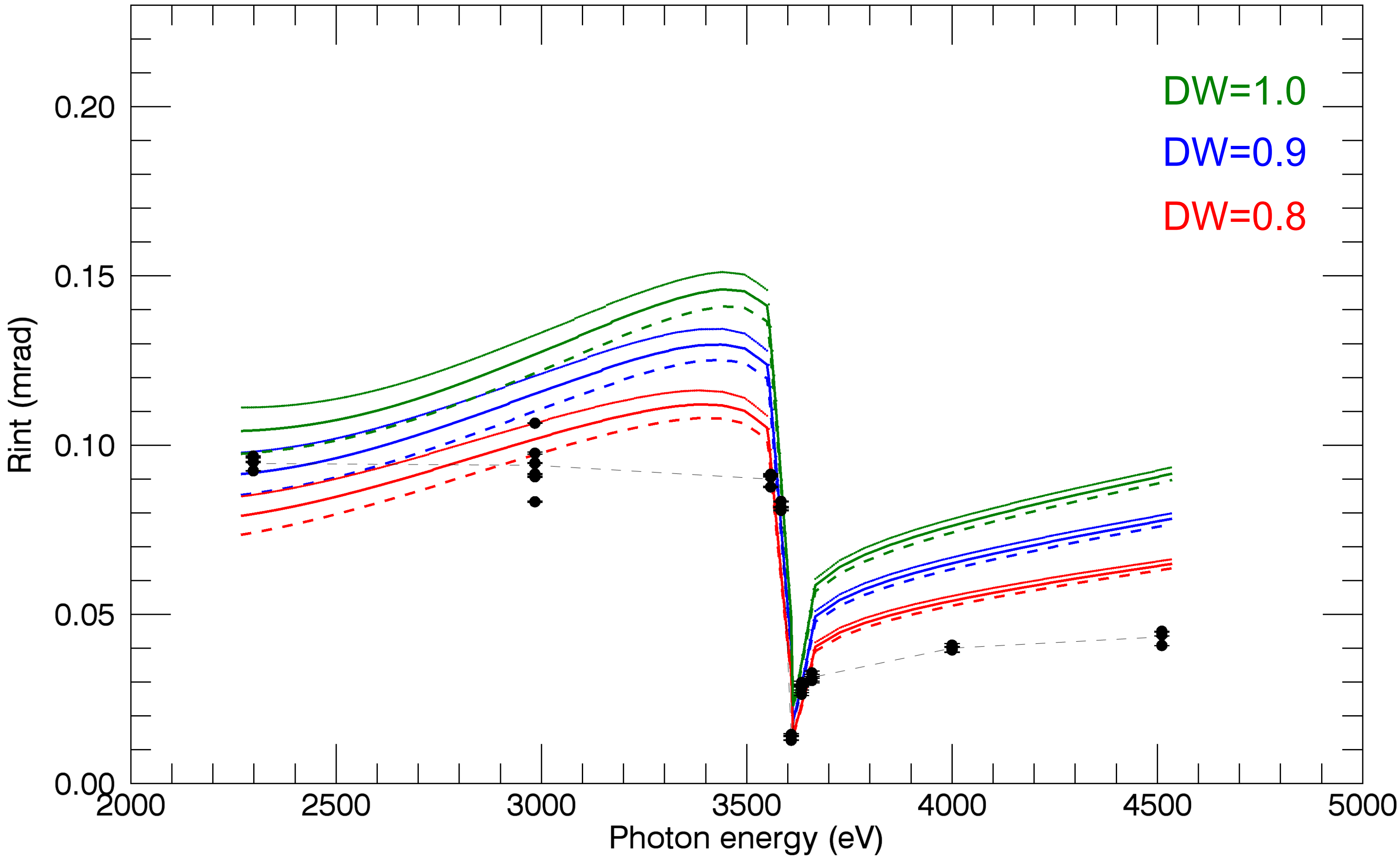
Integrated Reflectivity

1^{st} order

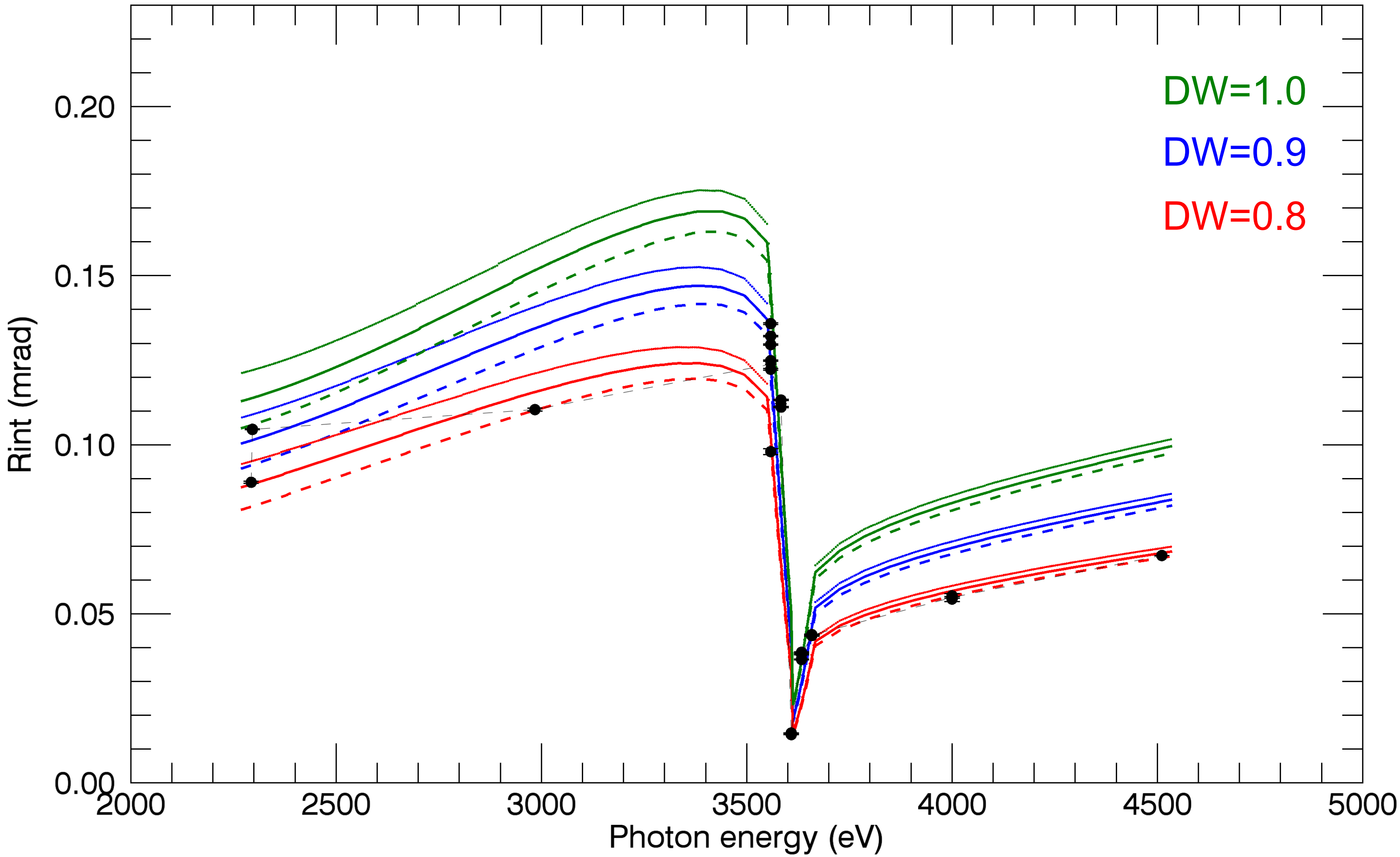
FLAT – 1st order



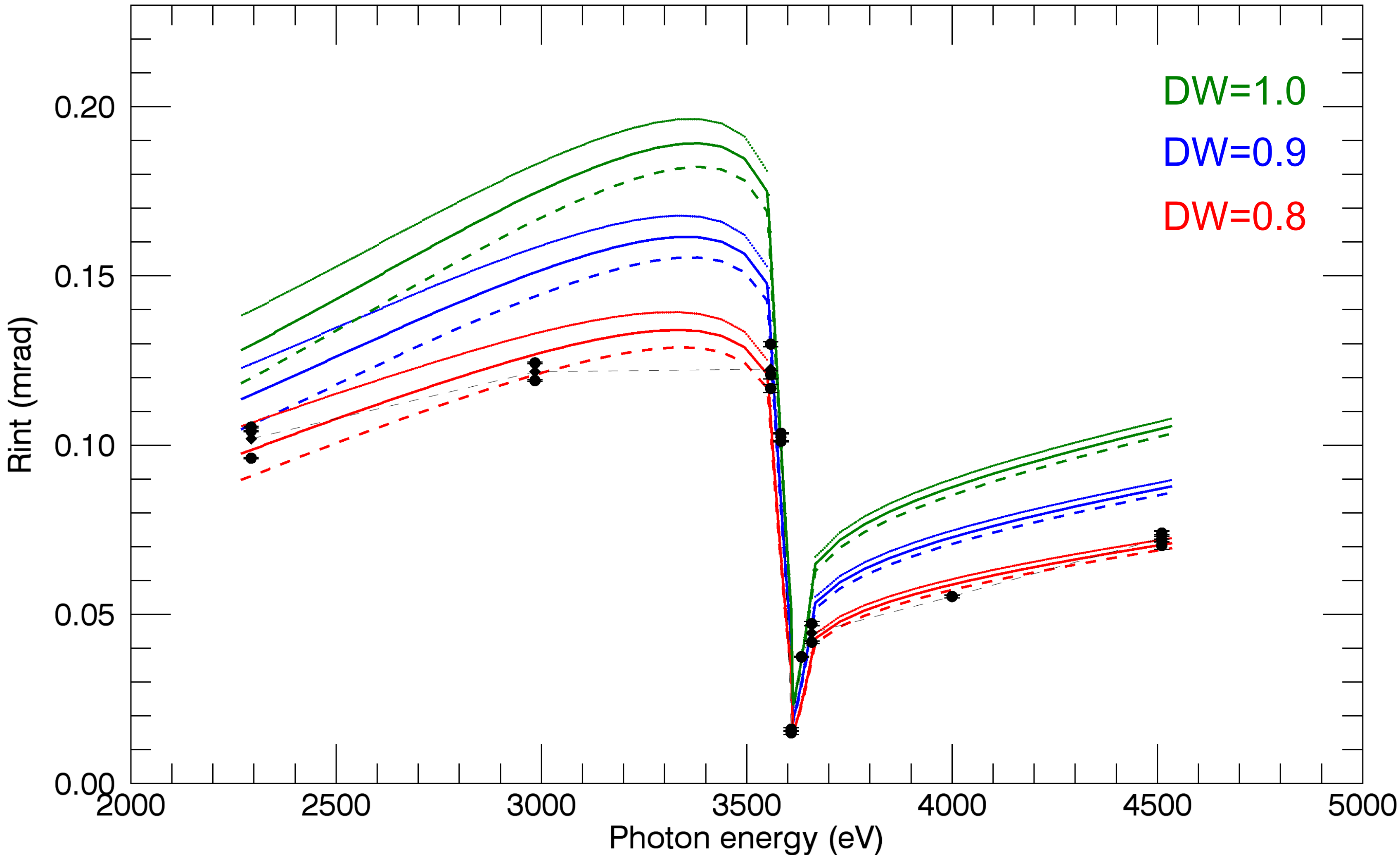
9in – 1st order



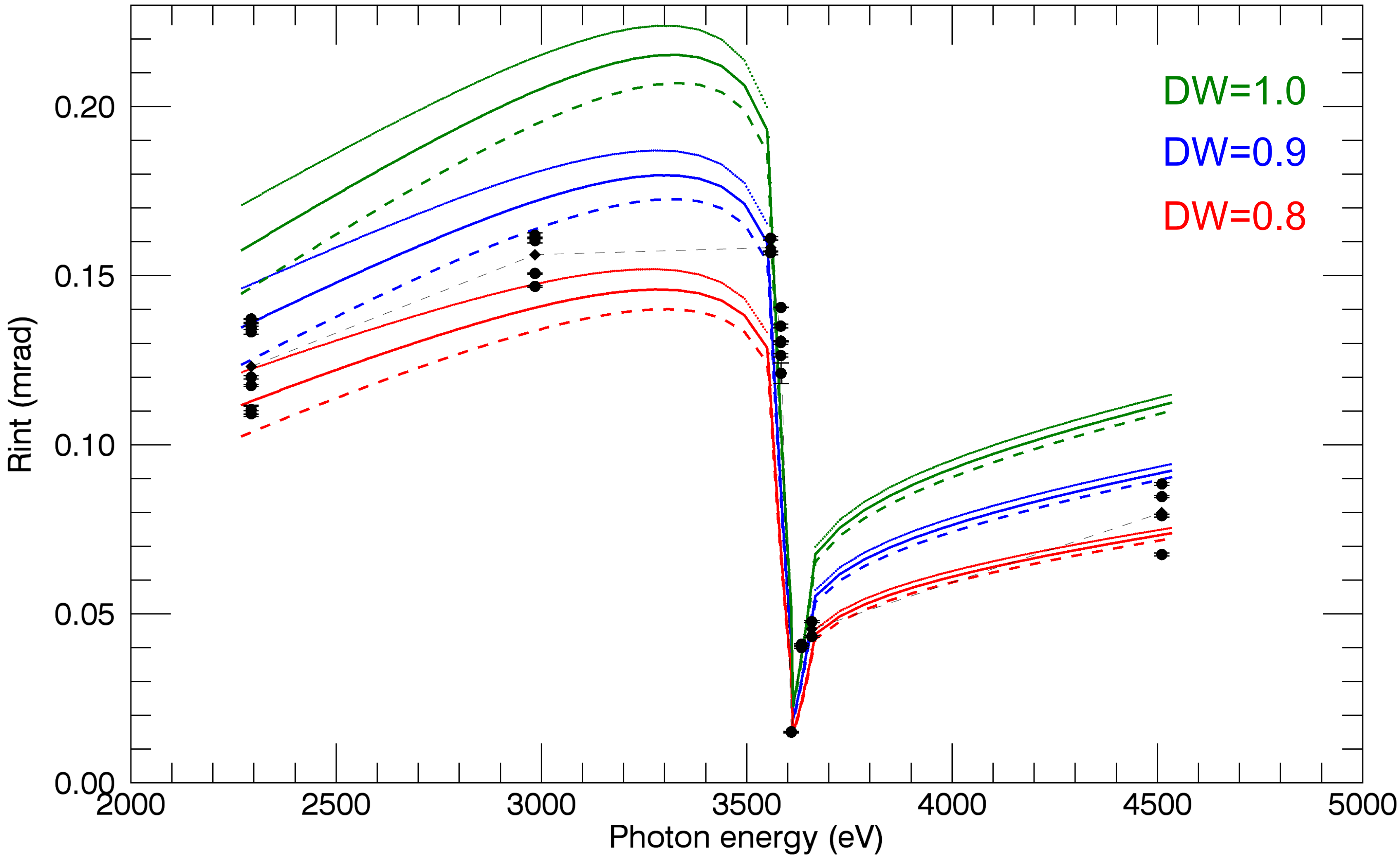
6in – 1st order



4in – 1st order



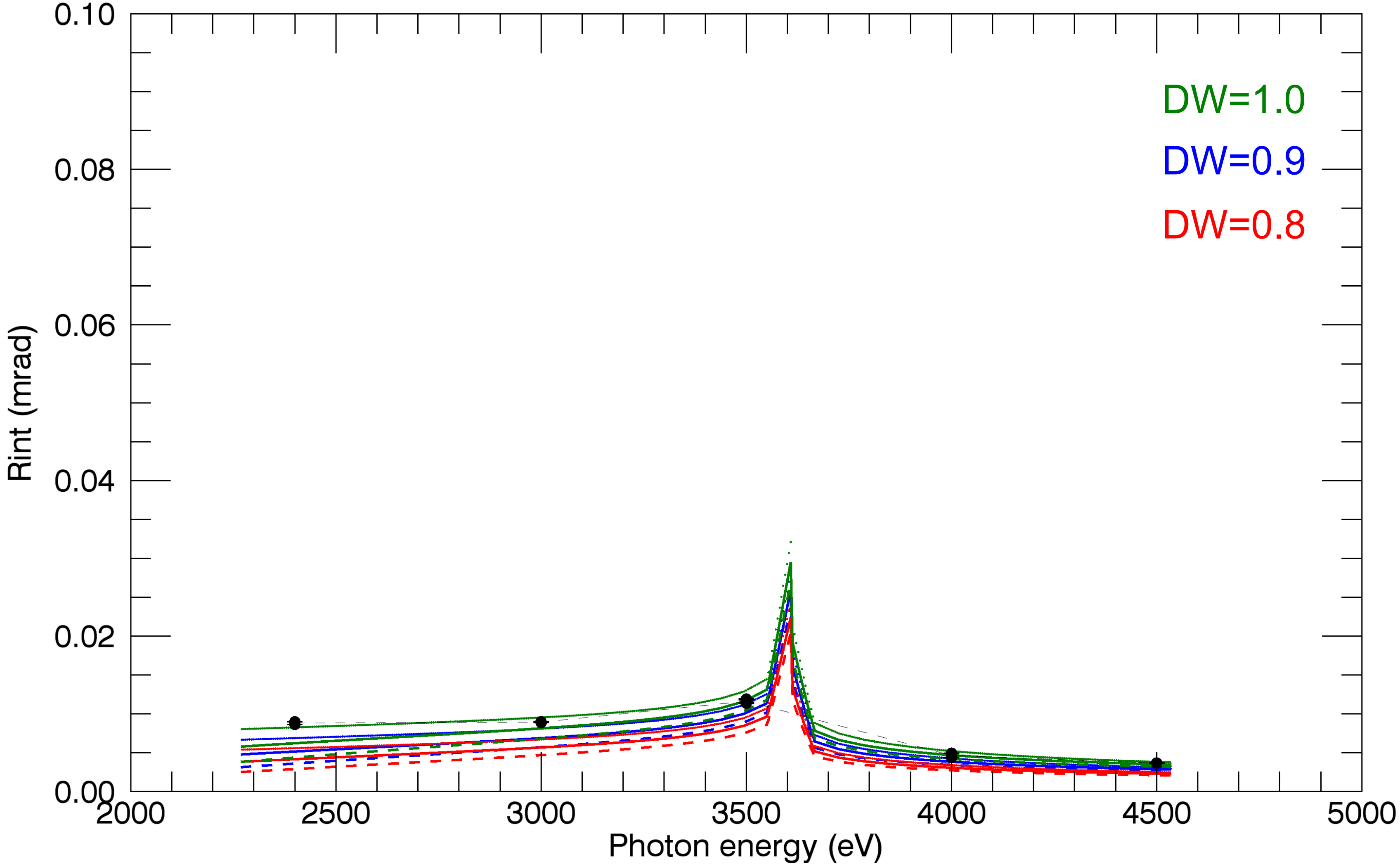
2in – 1st order



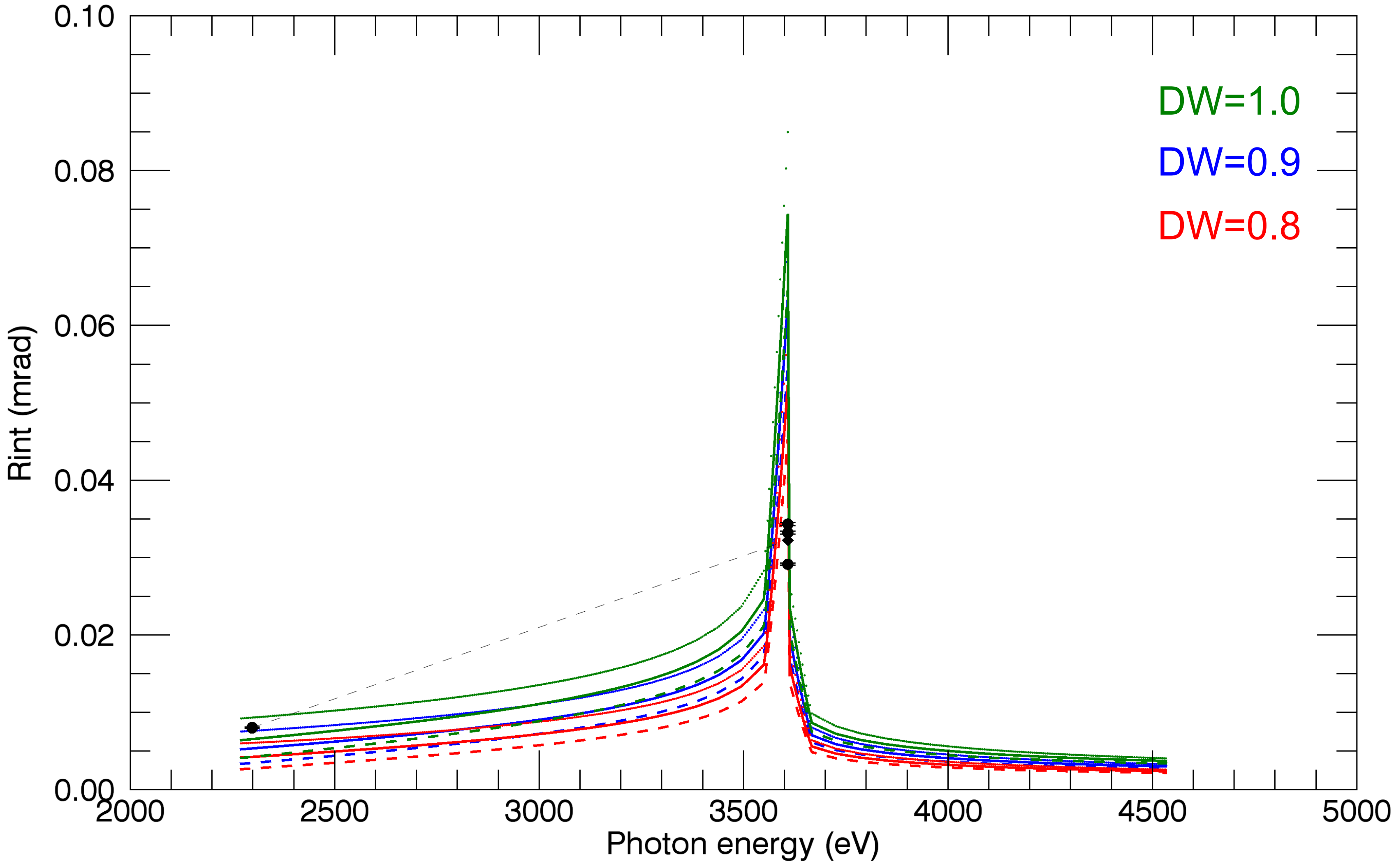
Integrated Reflectivity

2^{nd} order

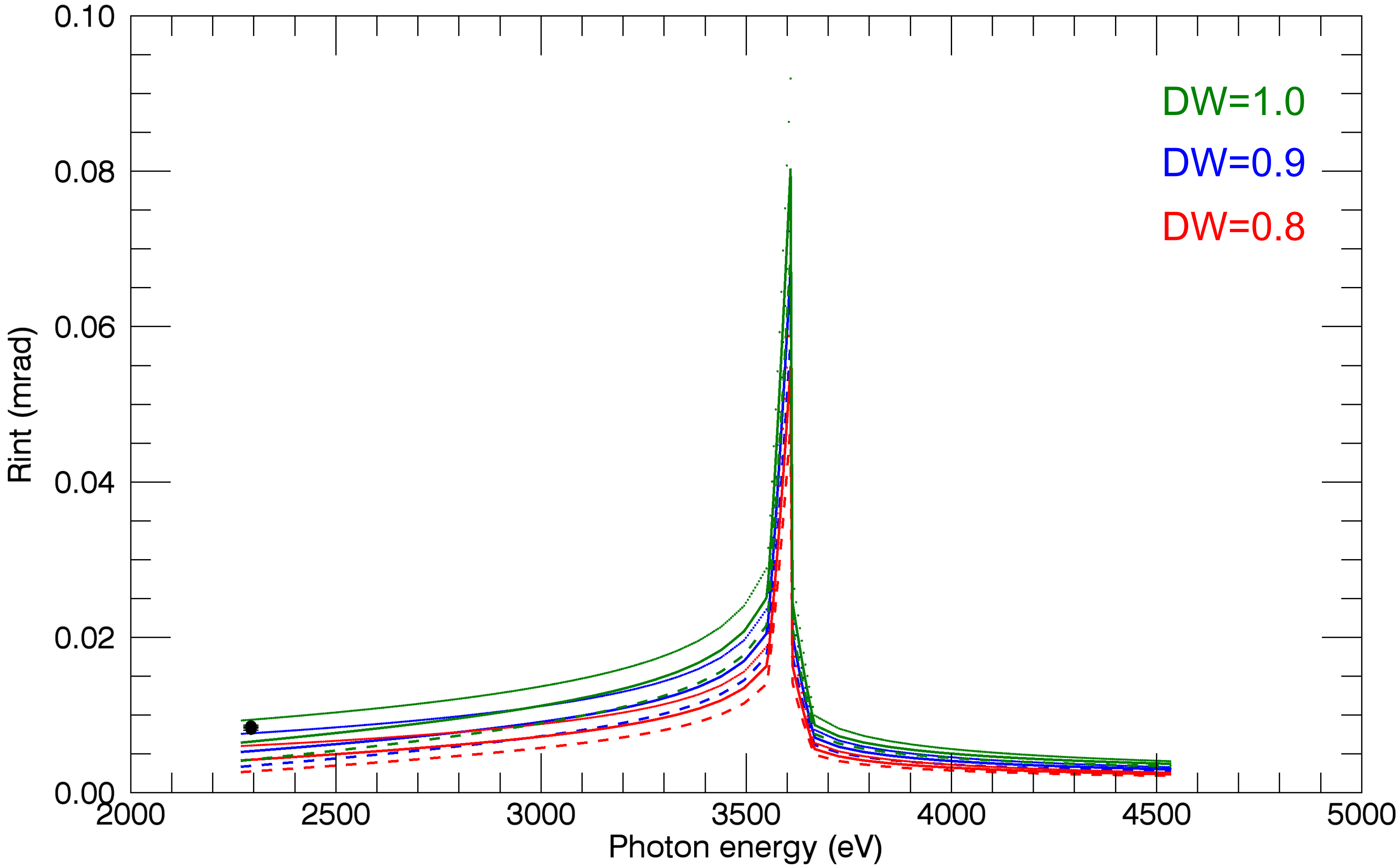
FLAT – 2nd order



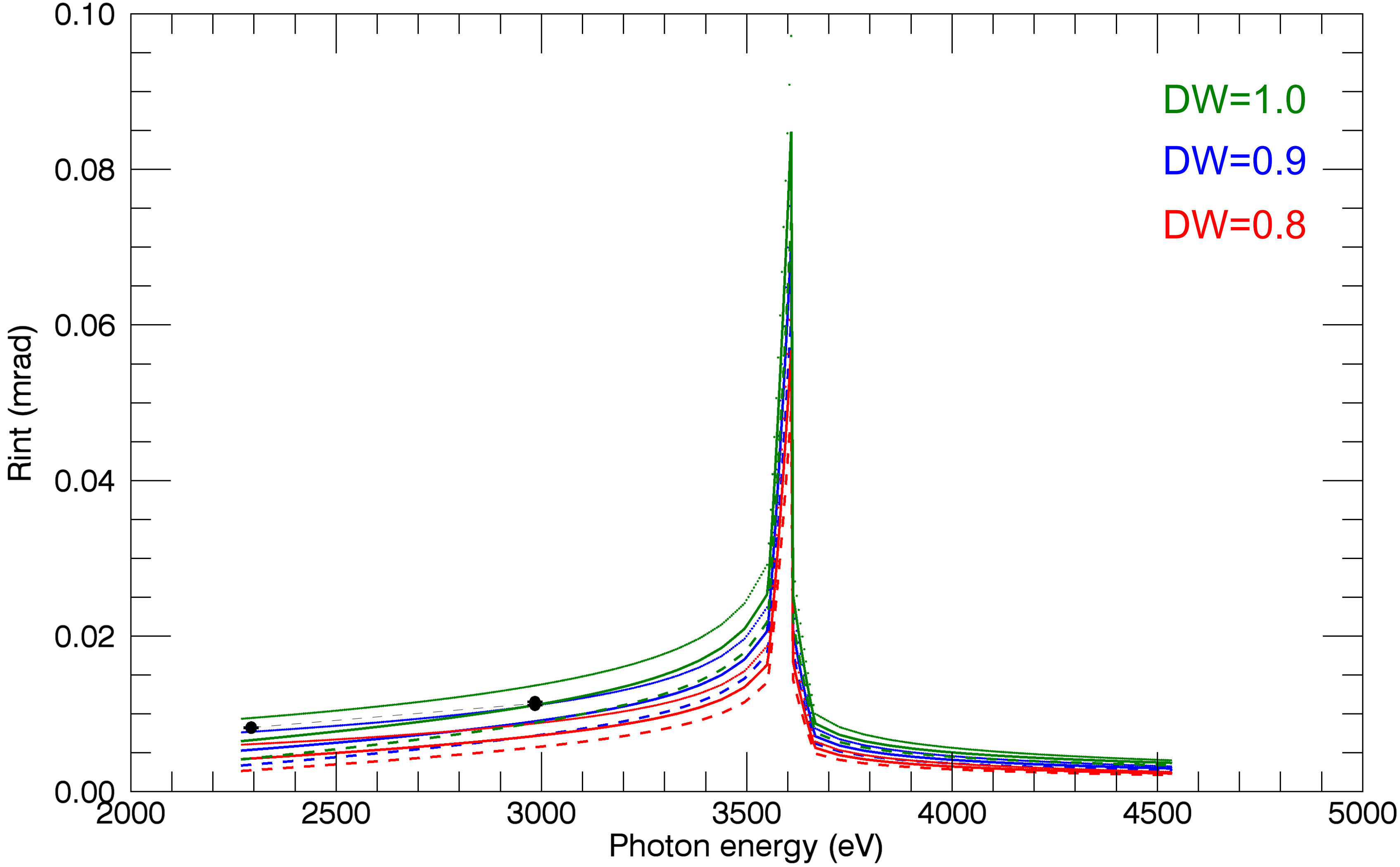
9in – 2nd order



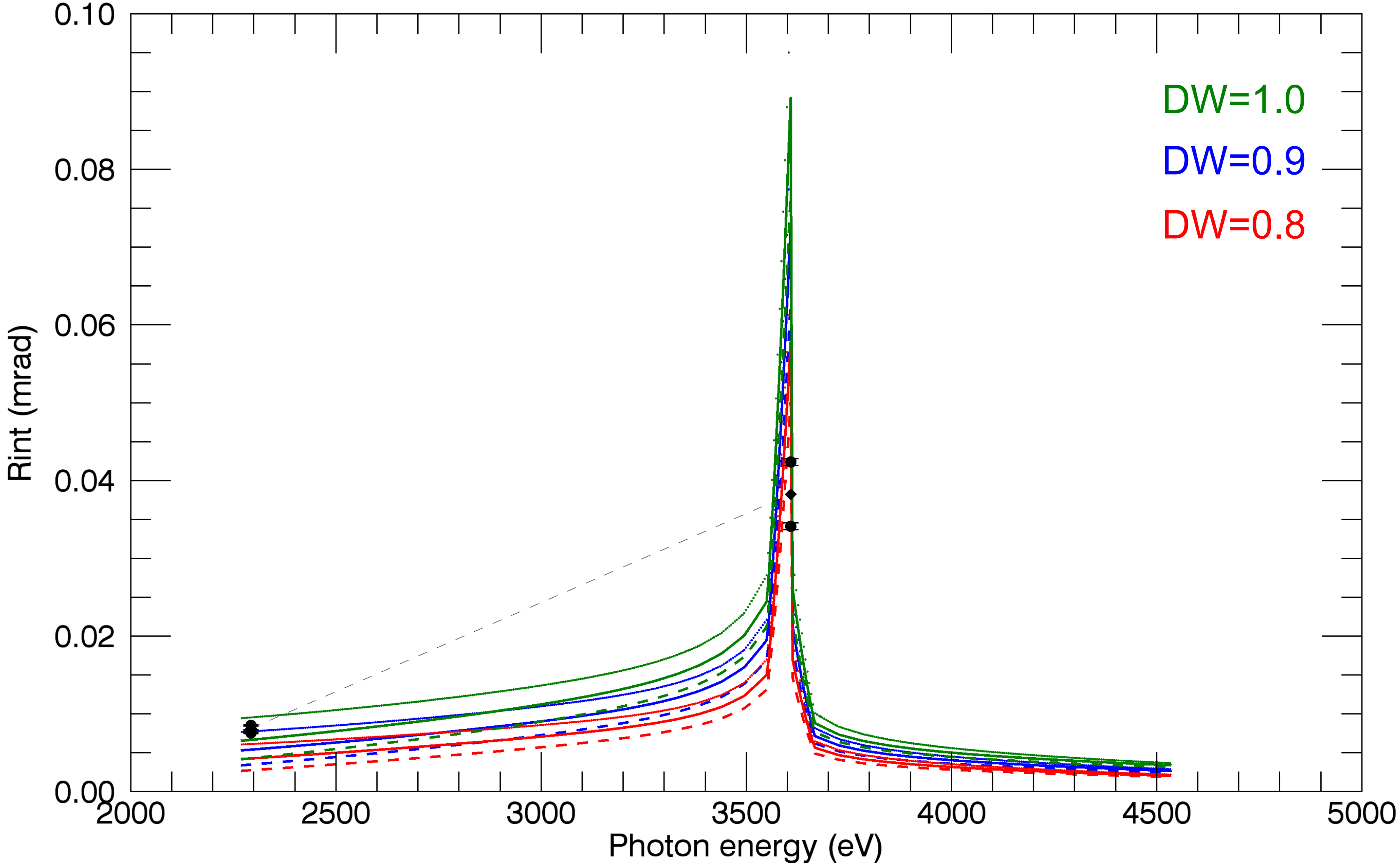
6in – 2nd order



4in – 2nd order



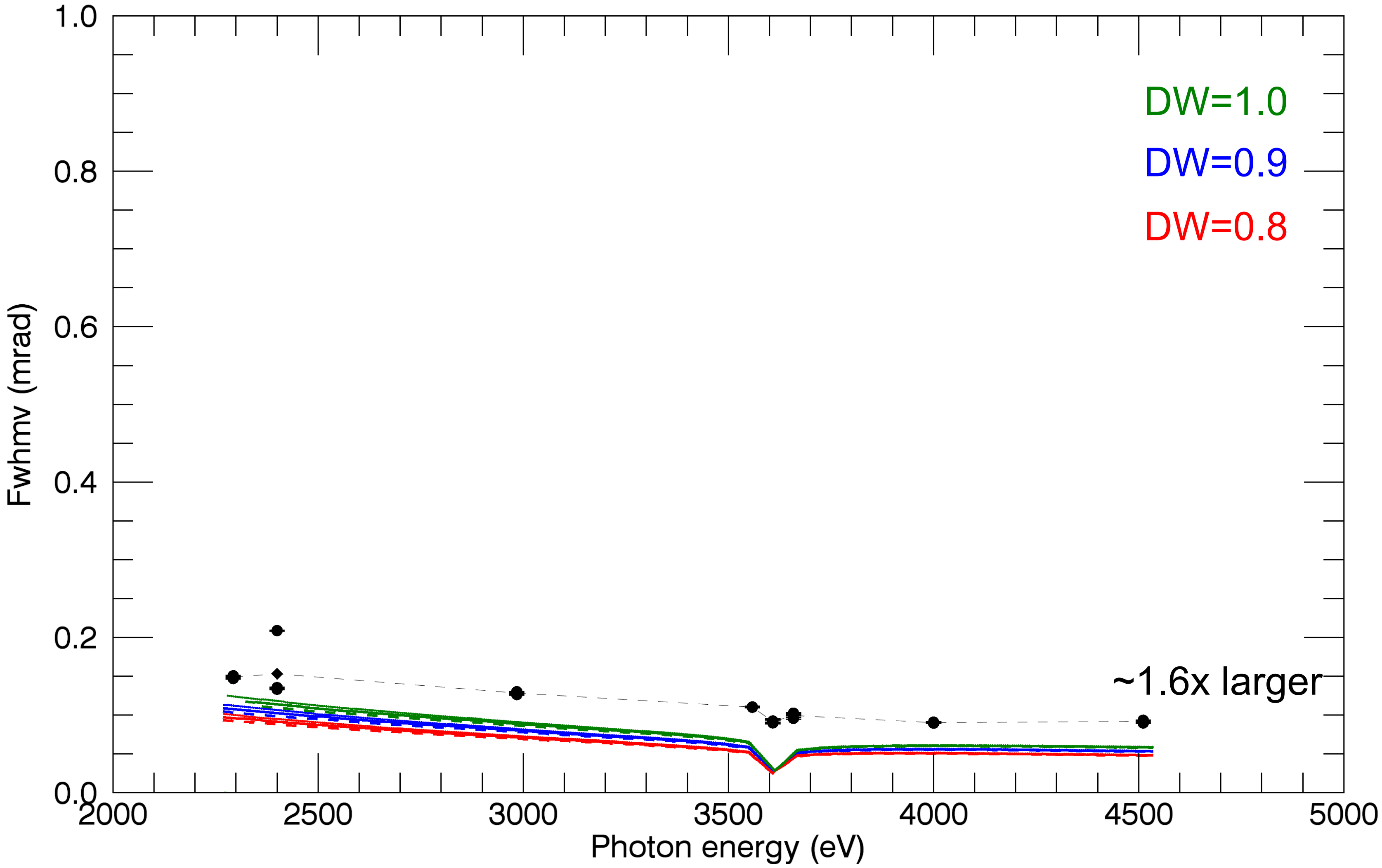
2in – 2nd order



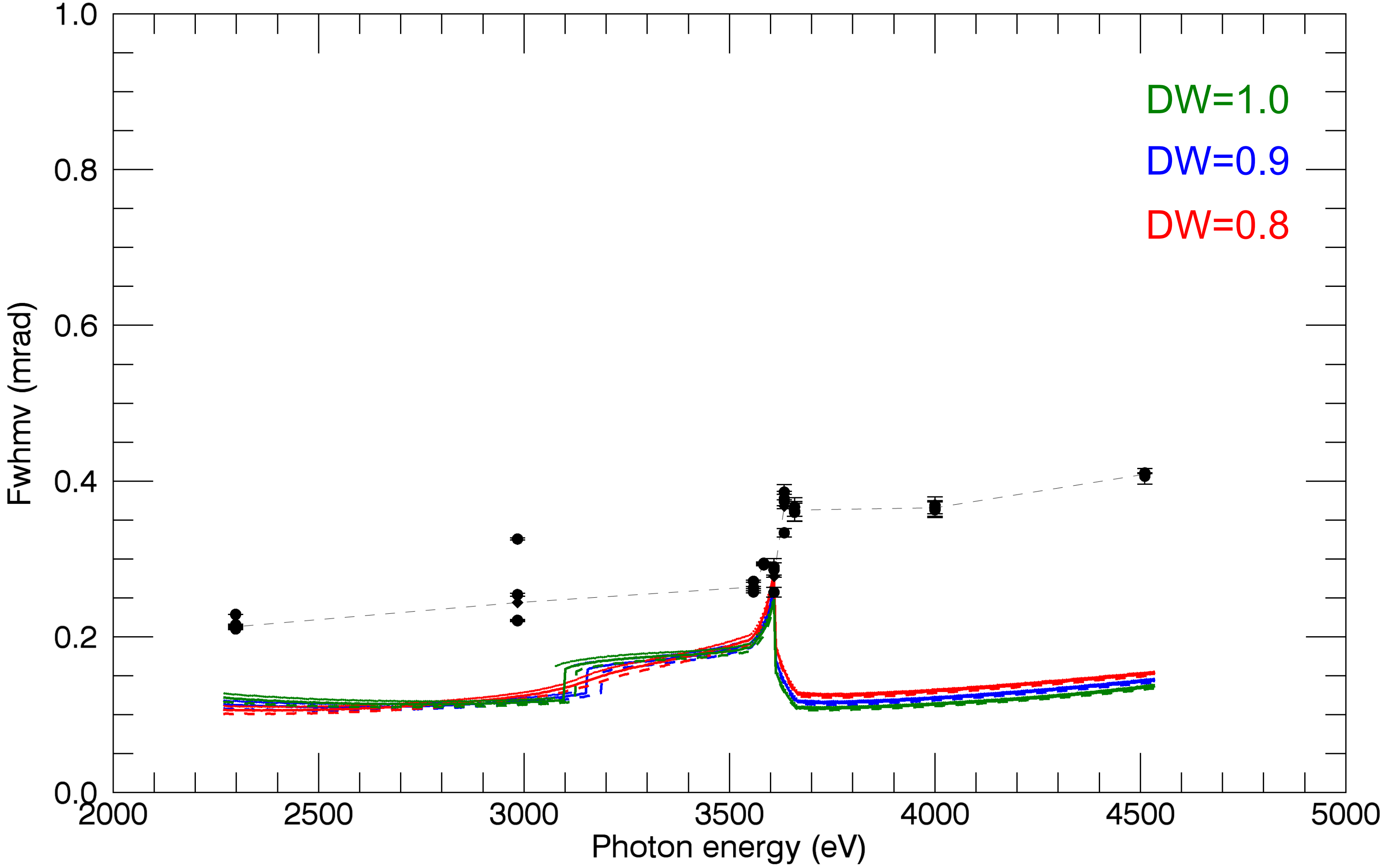
Rocking curve FWHM

1st order

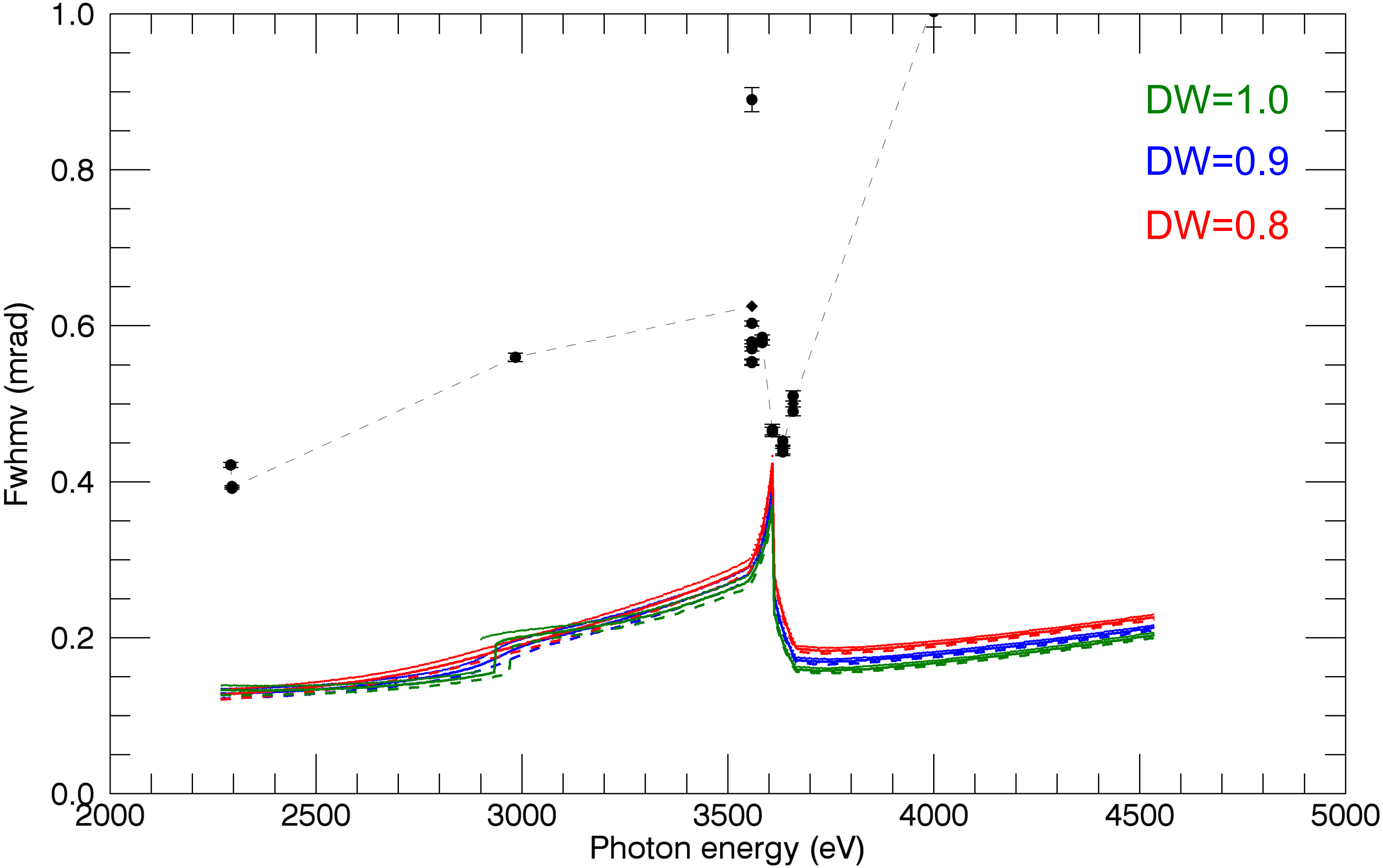
FLAT – 1st order



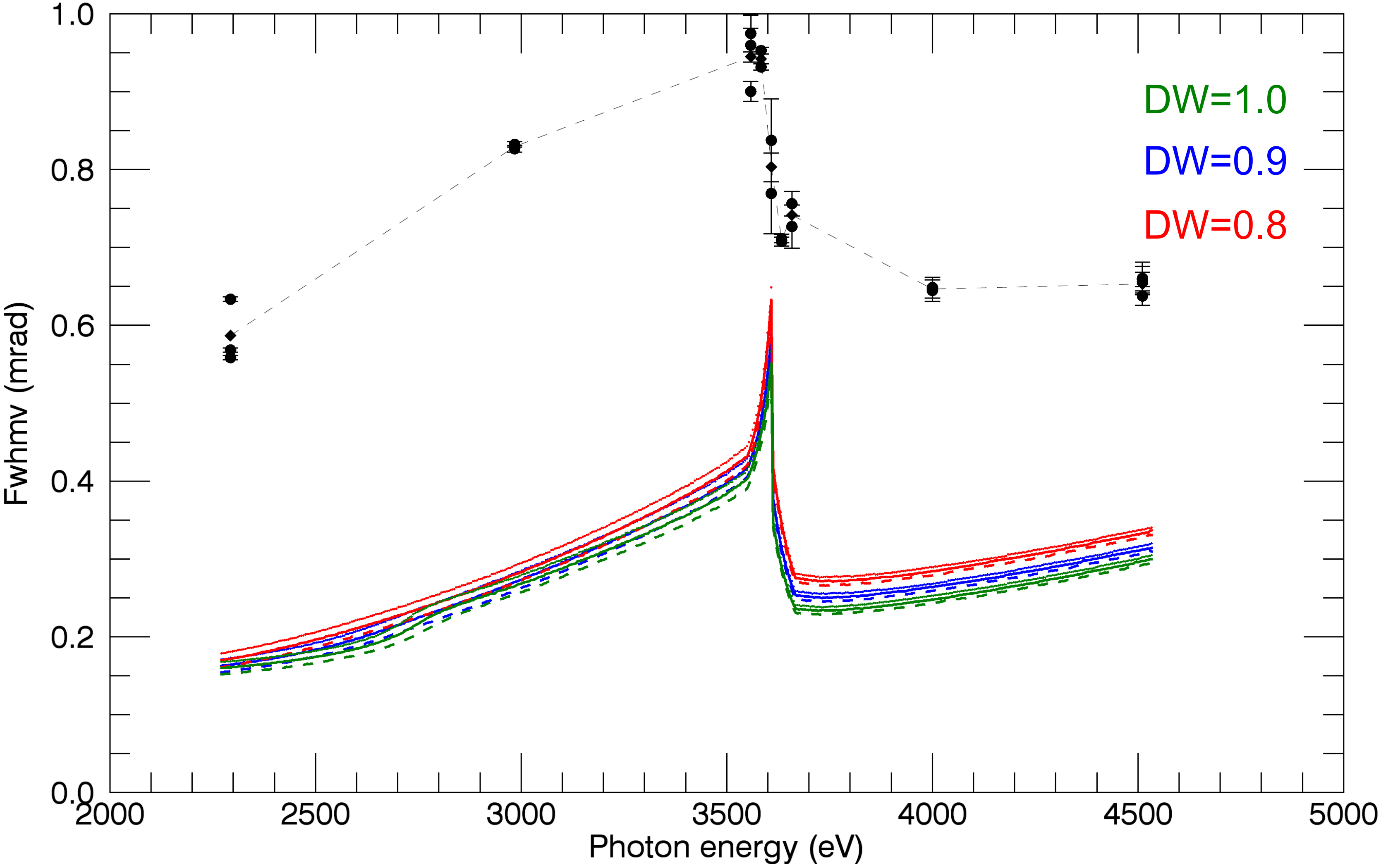
9in – 1st order



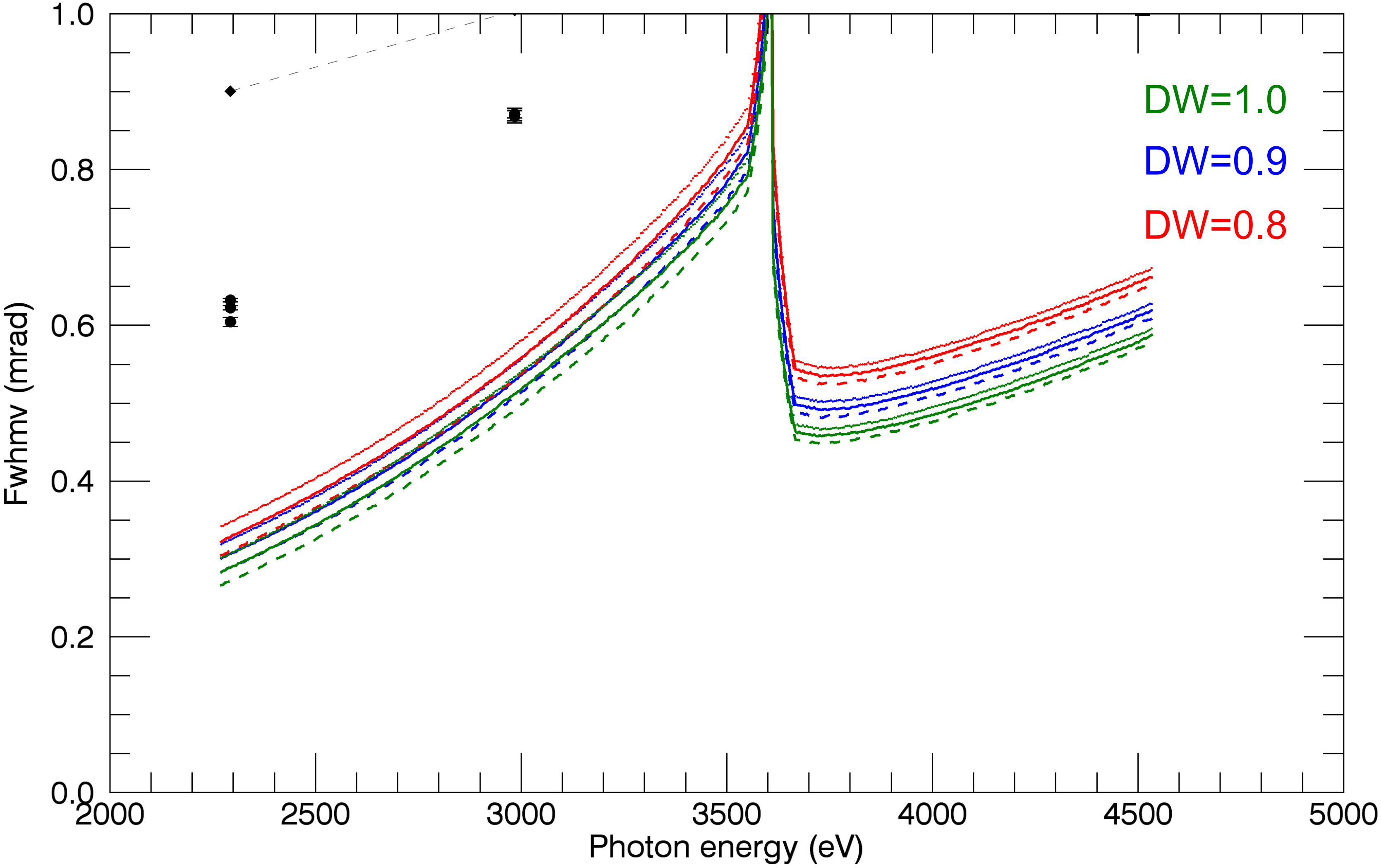
6in – 1st order



4in – 1st order



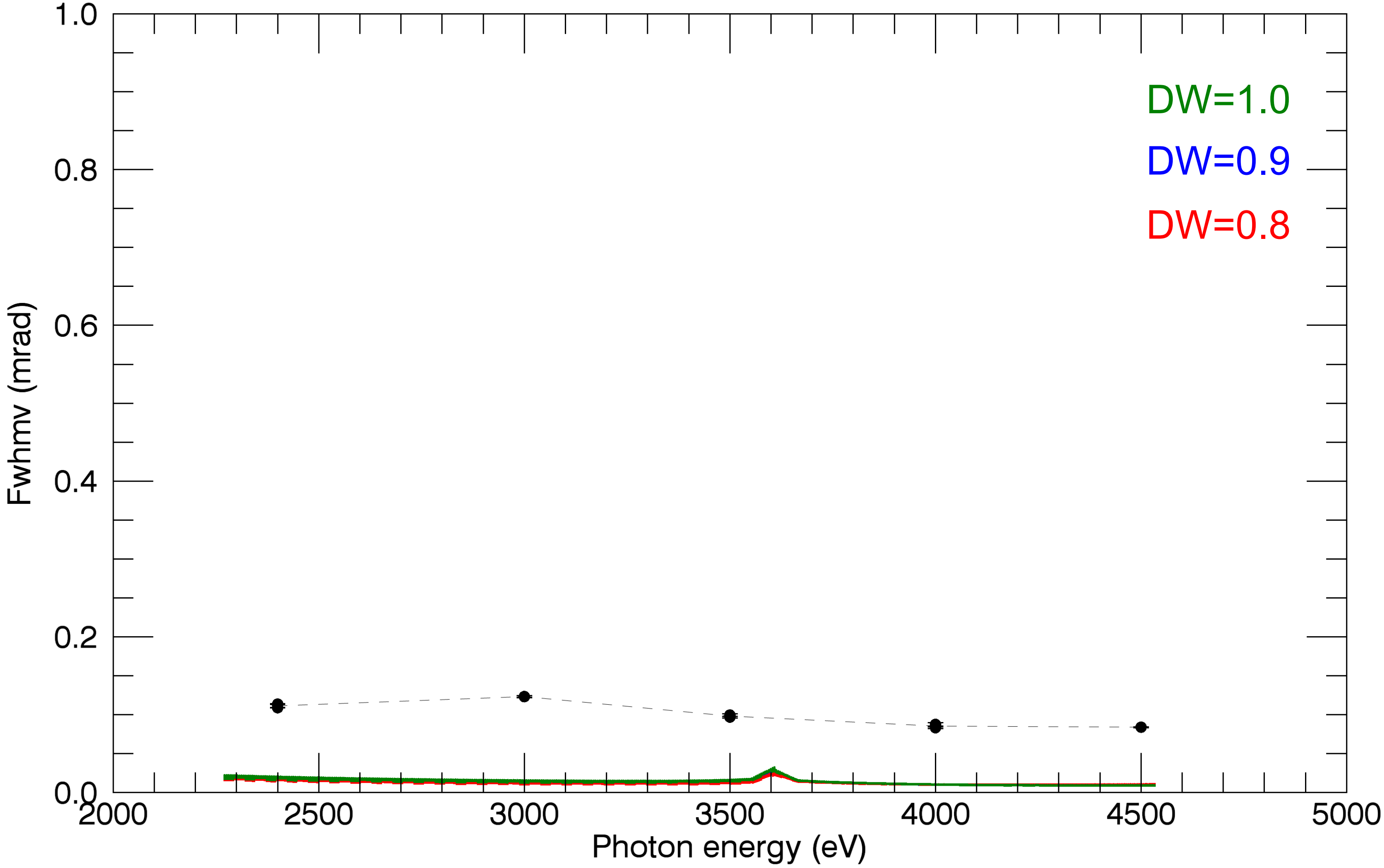
2in – 1st order



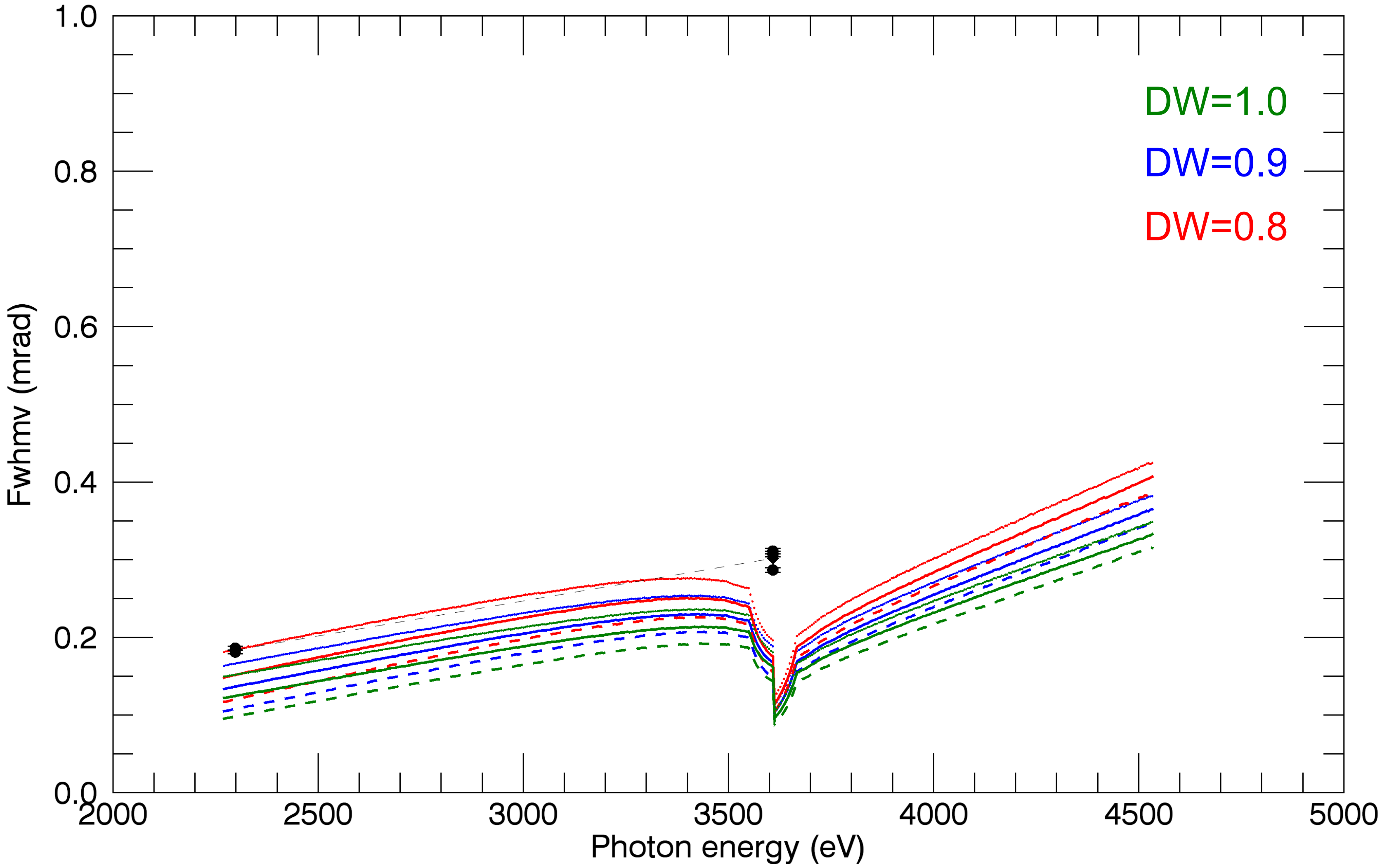
Rocking curve FWHM

2^{nd} order

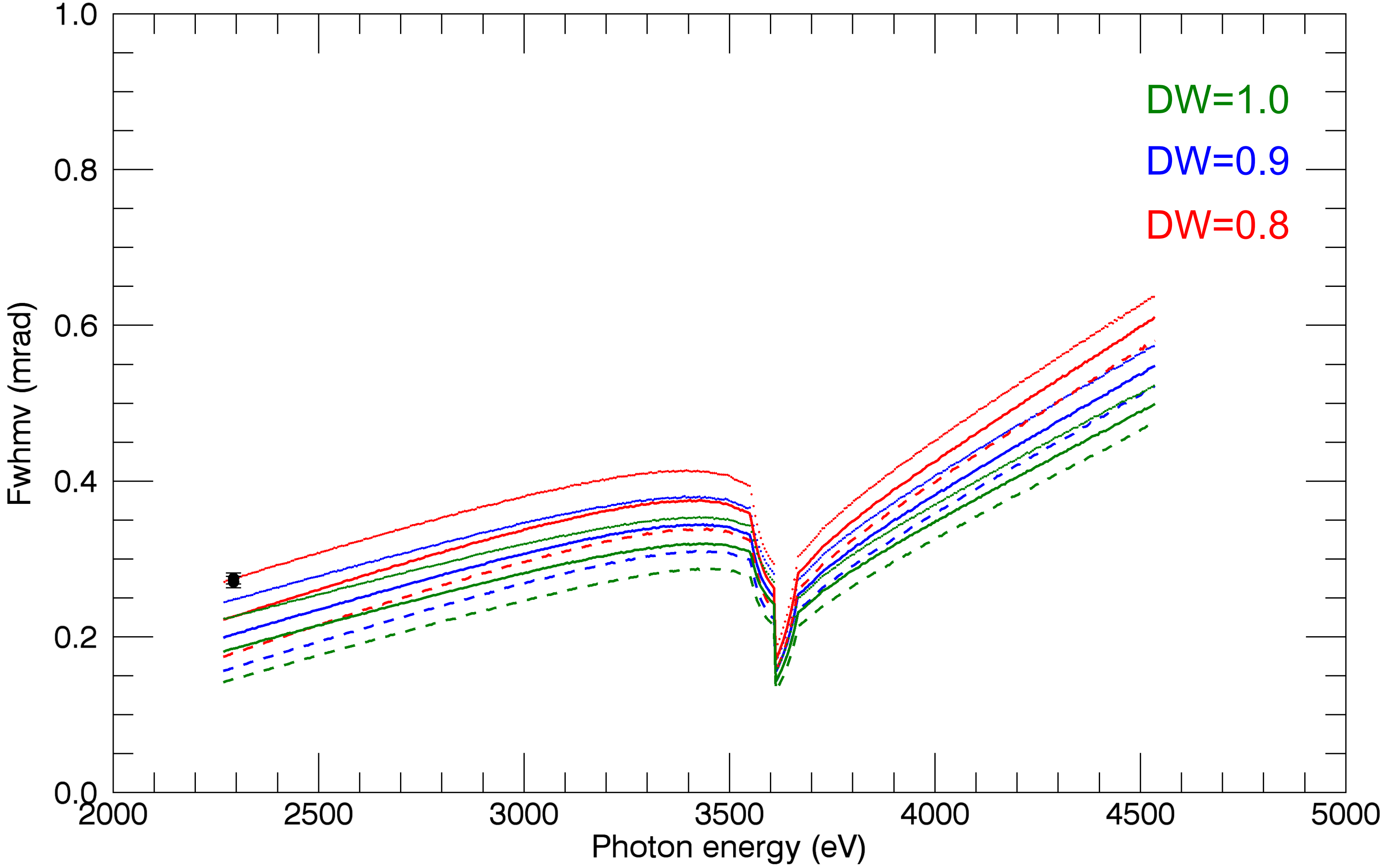
FLAT – 2nd order



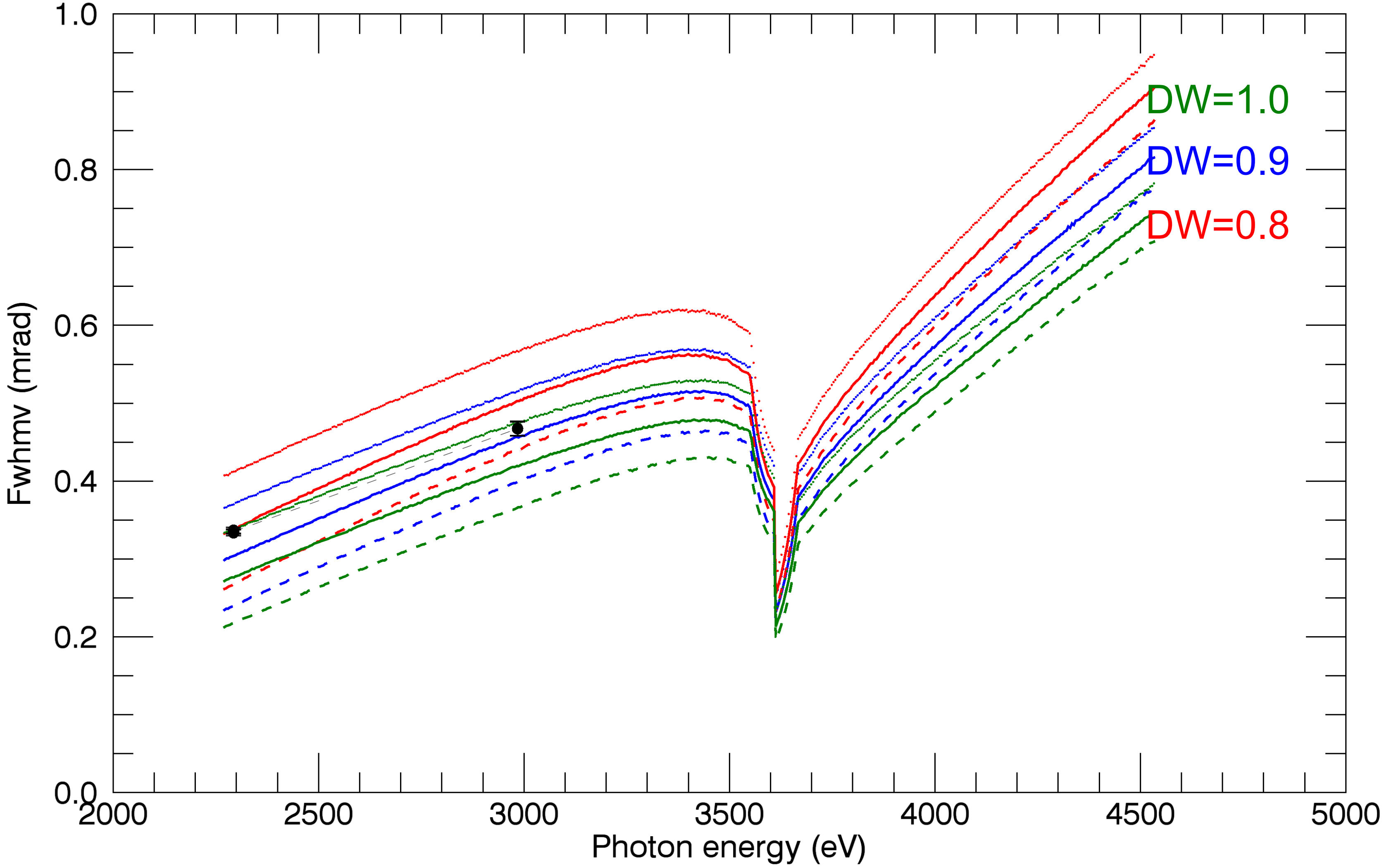
9in – 2nd order



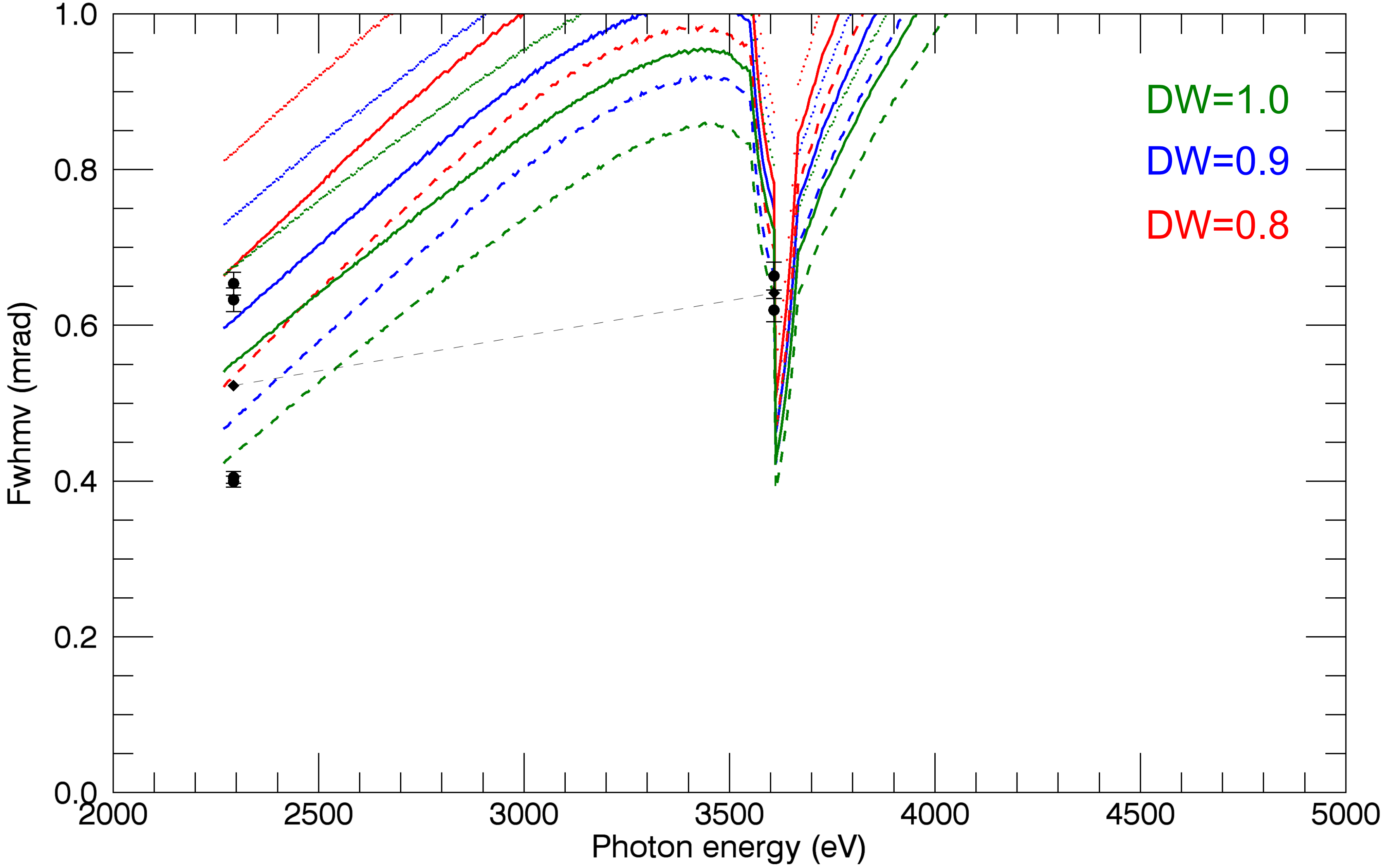
6in – 2nd order



4in – 2nd order



2in – 2nd order



Contributions to rocking curve width

Sandia National Laboratories

1) crystal rocking curve
2) beam size at crystal (depends on angle of incidence)
3) crystal bending
4) intrinsic energy width
5) misalignments

Width Voigt components:

Lorentzian (measured)

$$w_L = w_{L,xtl} + w_{L,\mathrm{intrinsic}}$$

Gaussian (measured)

$$w_g^2 = w_{g,xtl}^2 + \left[\left(\alpha d_p + 1.22\frac{r\lambda}{d_p}\right)\frac{1}{R_c \sin\theta}\right]^2 + w_{g,\mathrm{intrinsic}}^2$$

diffraction

effective beam size

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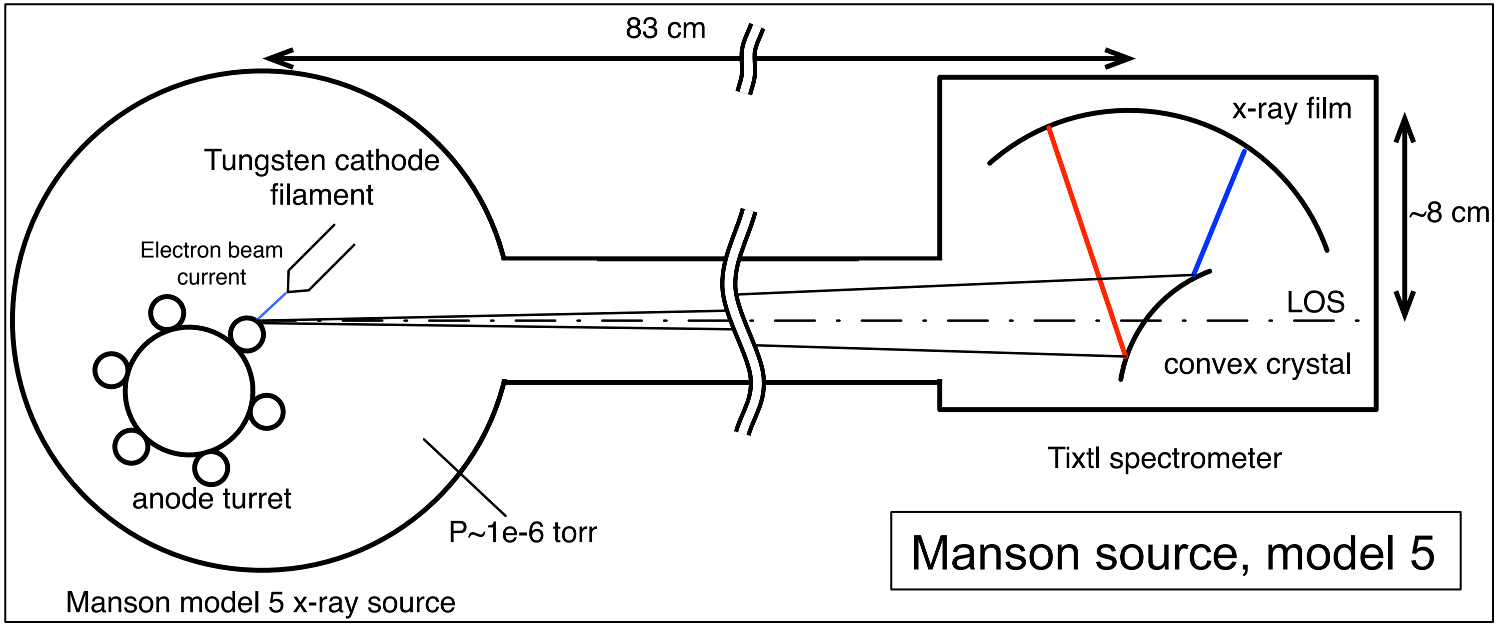
diffraction

effective beam size

The unknowns can be searched using under this large dataset to perform a forward deconvolution

2) Using the un-calibrated Manson source to measure crystal spectral resolution and shape in KAP

The rocking curve shape and width in 1st order can be measured with a conventional x-ray source*



x-ray film

mag~0.3

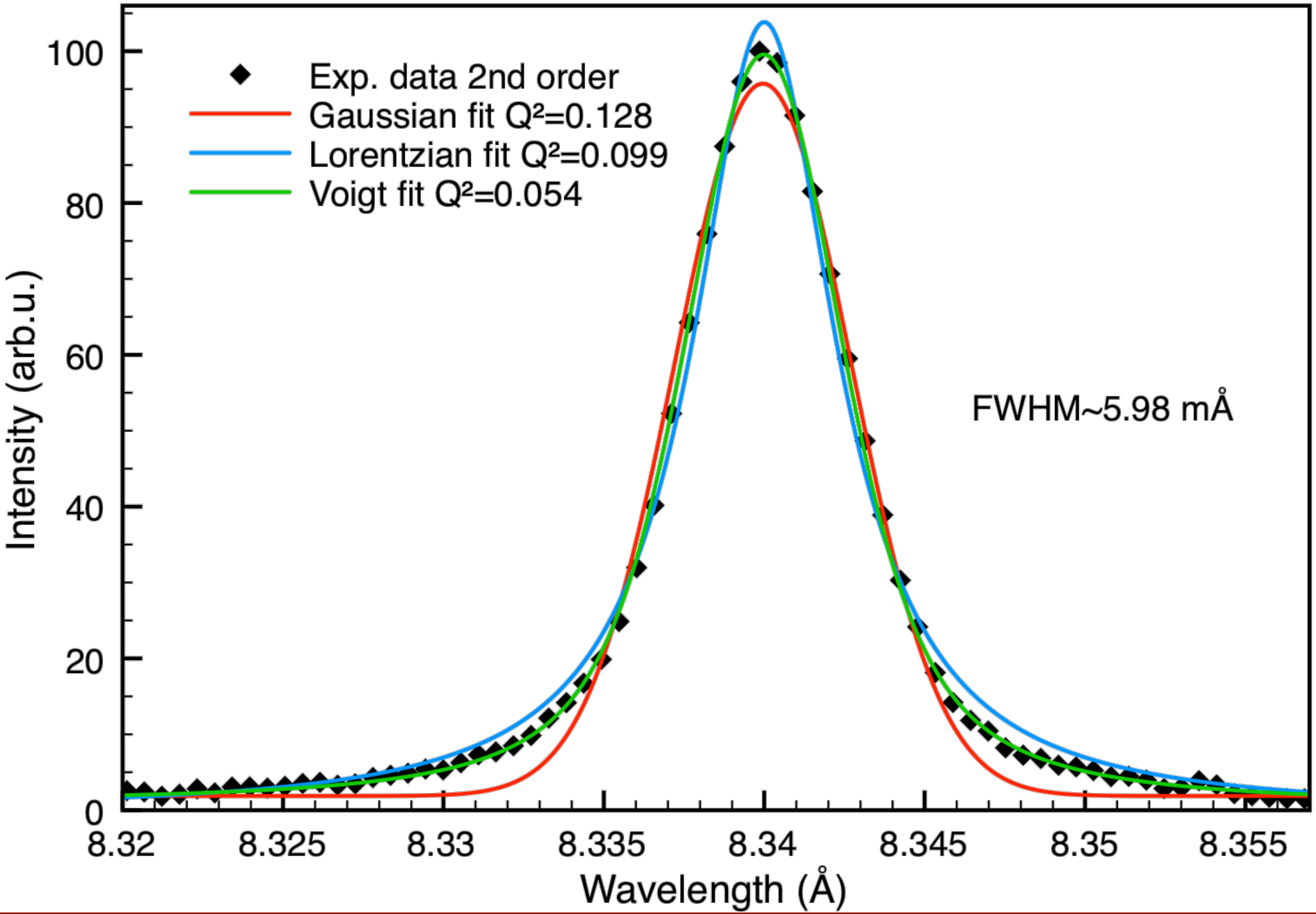
source size~200μm

Methodology

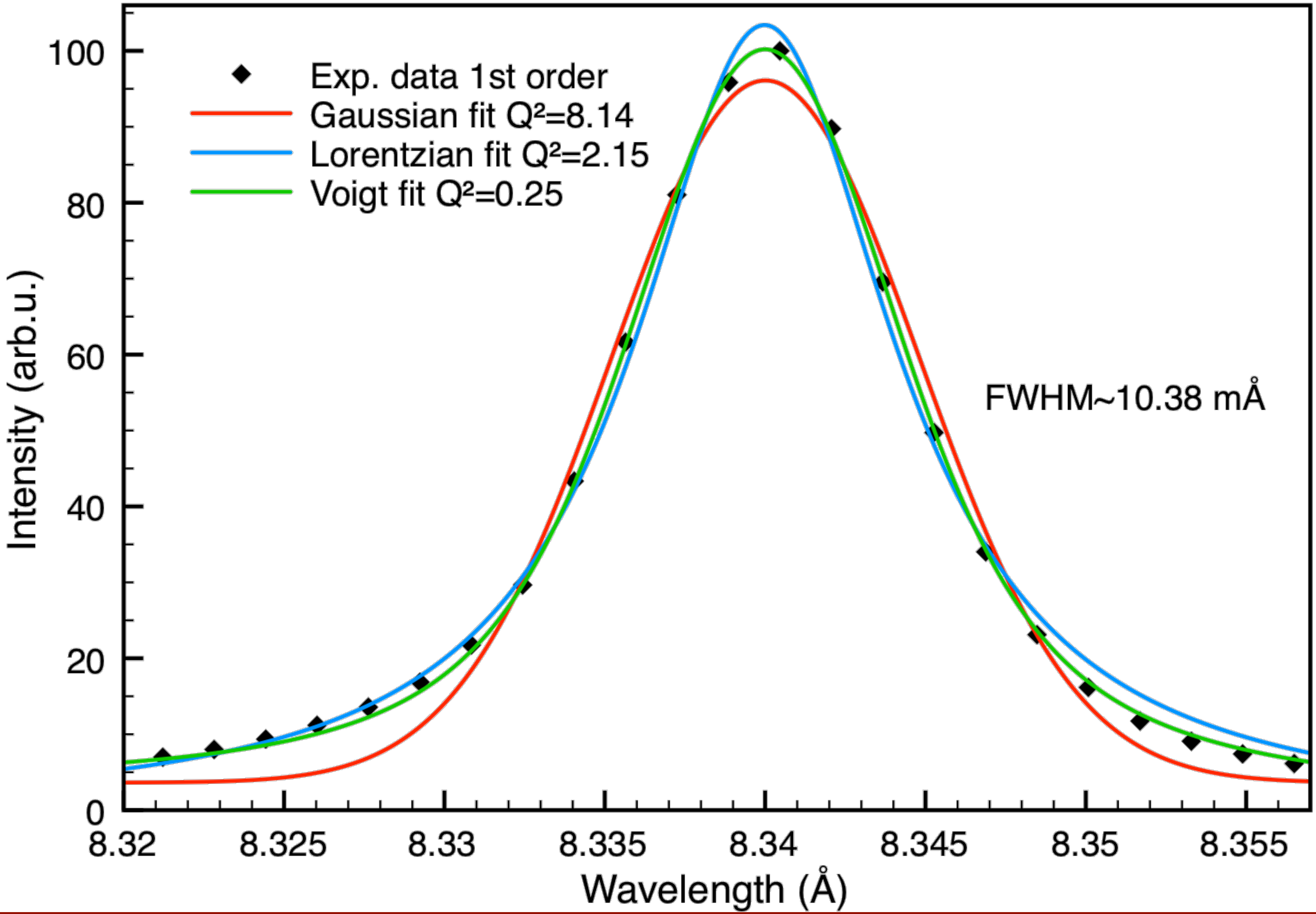
- The detected spectral line (characteristic line) is made of the intrinsic line (at the source) convolved with crystal rocking curve.
-The intrinsic line shape and width is measured with a high resolution setup (here 2nd order measurement)
-The 1st order shape and width are obtained by deconvolution of the 2nd order measurement

*G. Loisel *et al*, RSI, 2012

KAP 6in - Al Kα in 2nd order



KAP 6in - Al Kα in 1st order



KAP 6in results for Al, Mg and Si Kα lines

Element / K α wavelength	2^{nd} order width (mÅ)	1^{st} order width (mÅ)	Crystal broadening (mÅ)	Crystal 1^{st} order resolving power
Si / 7.126 Å	5.87 (a=0.67)	9.72 (a=0.83)	6.24 (a=0.67)	1142±73
Al / 8.34 Å	5.98 (a=0.88)	10.38 (a=0.99)	6.64 (a=0.77)	1256±73
Mg / 9.89 Å	7.03 [13] (a =0.94)	11.81 (a=1.04)	7.47 (a=0.13)	1415±348 [13]

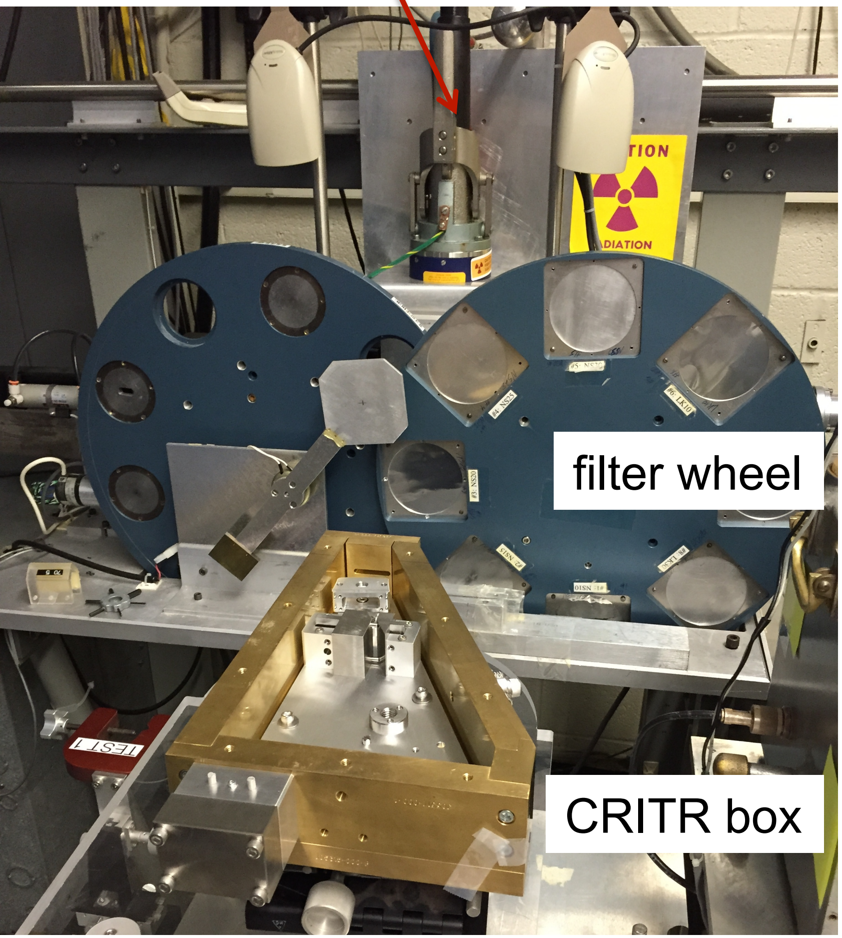
The found crystal profiles are Voigt profiles with RP=1100

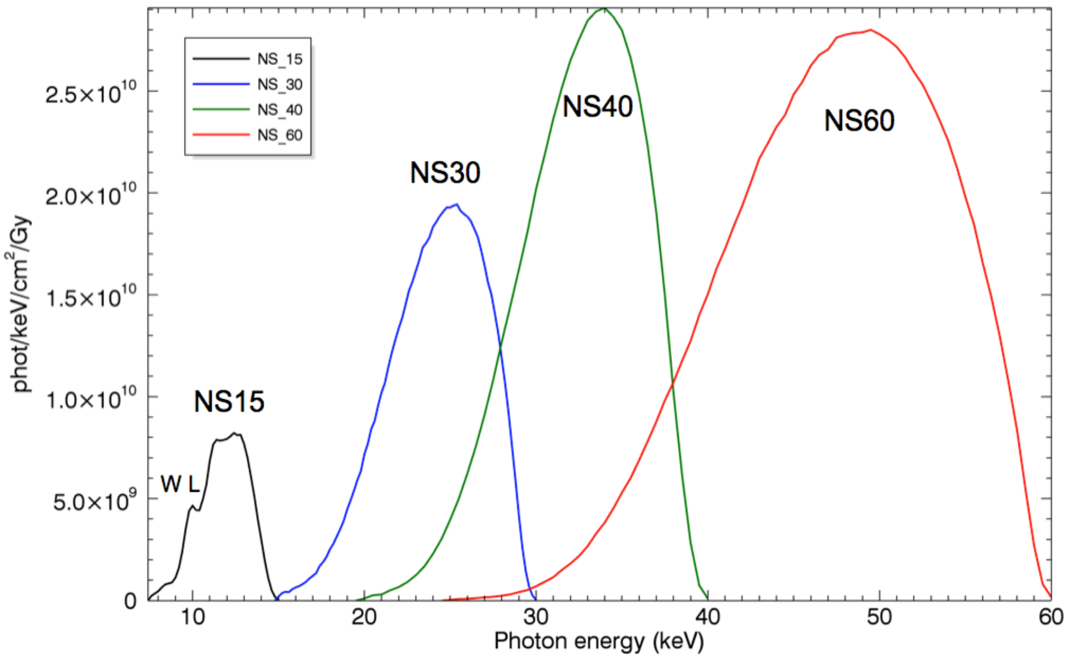
NB: the convolution of a Voigt (RP=1100) is similar to a Gaussian (RP=700)

3) Using the NIST absolutely calibrated KERMA facility to measure crystal efficiency in Quartz transmission

Absolute calibration setup @ NIST KERMA facility

100kV tube, W anode



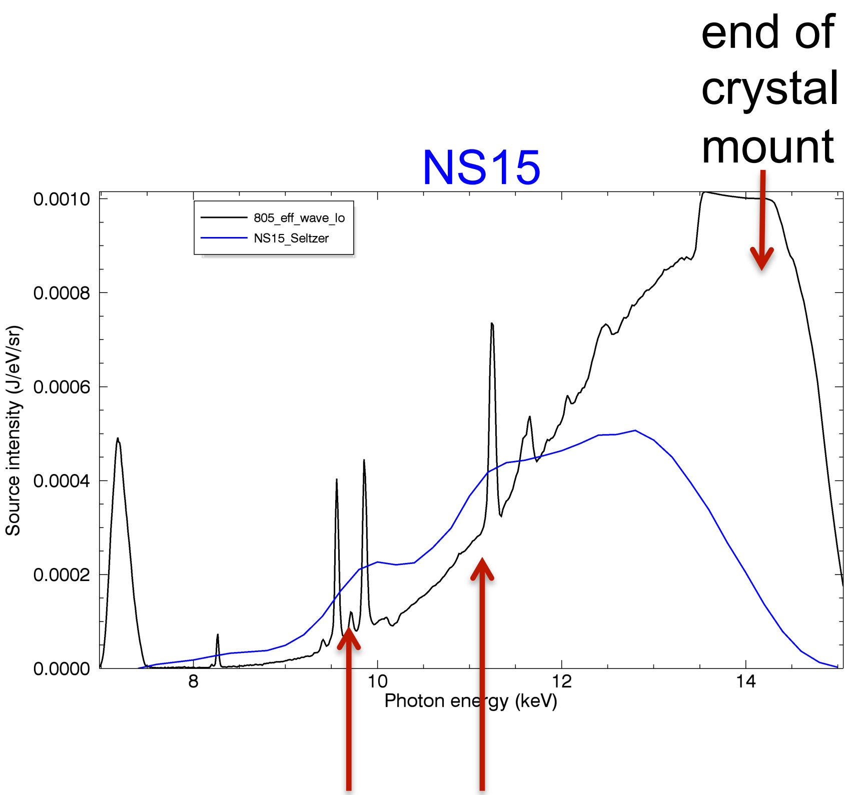


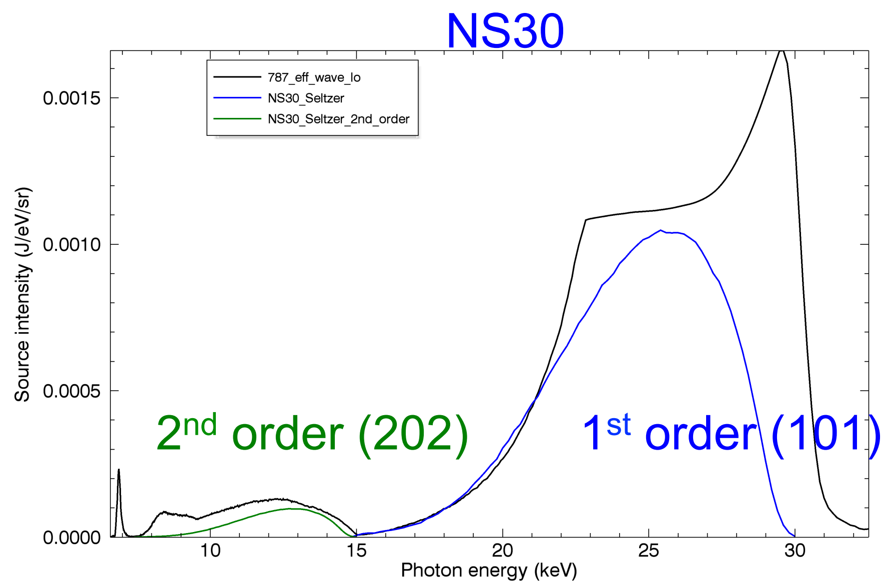
Beam qualities (NS) used for the calibration are international standards with 4% relative uncertainty

End-to-end instrument calibration

101 data

XOP calc rescaled with a flat **4.7** ratio





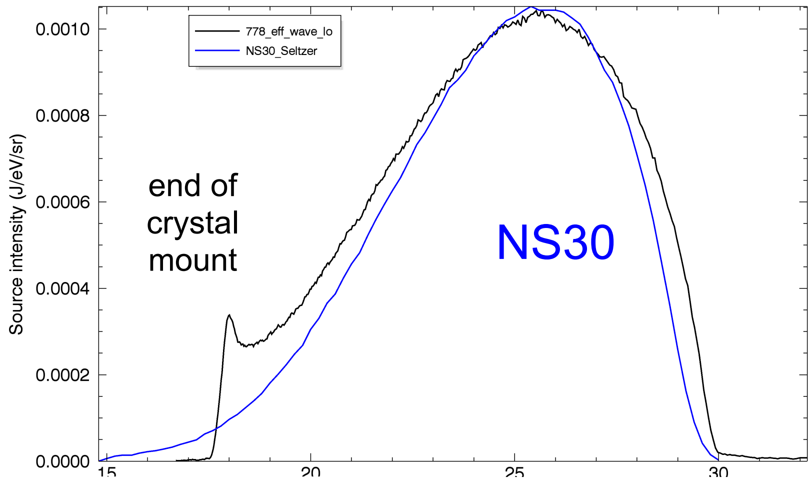
2nd/1st ratio ~10 is in reasonable agreement

The NS resolution should be used to convolve the crystal dispersed spectrum

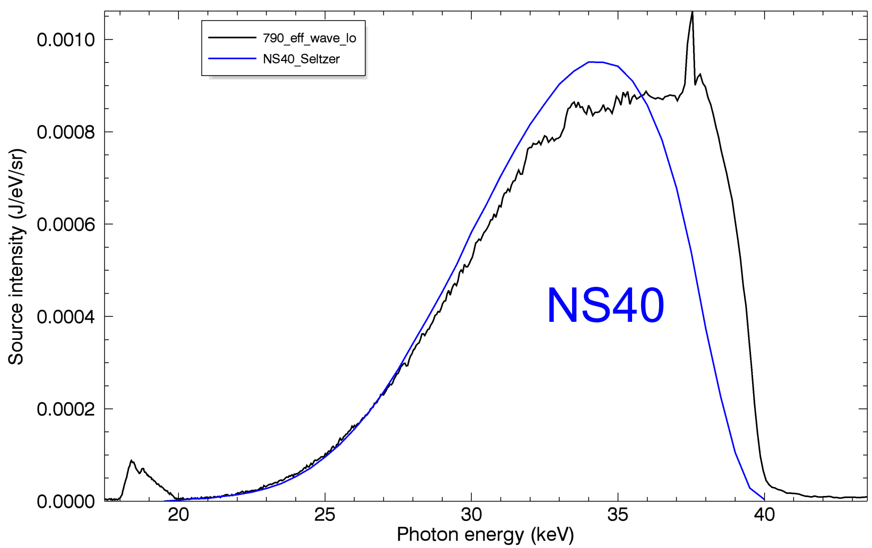
End-to-end instrument calibration

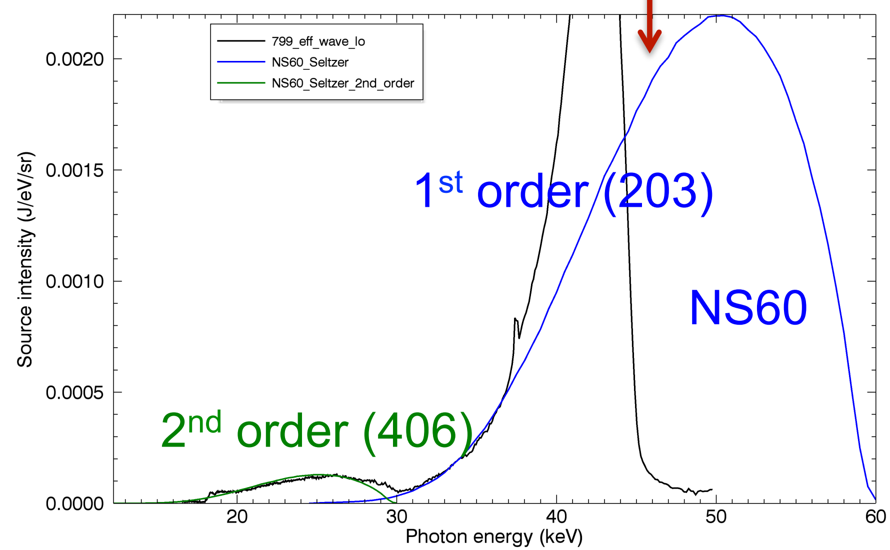
203 data
crystal 203-1-1

XOP calc rescaled with a flat **5.9** ratio



crystal mount clipping





$2^{nd}/1^{st}$ ratio ~20 is correct

Goals and results of the SNL crystal calibration work

Sandia National Laboratories

Goals

1) Measure accurate rocking curve of bent crystals to help the understanding of measurements taken on Z (opacity, photoionized plasmas, non-thermal emission, liner spectra…)
   e.g The iron opacity measurements on Z require integrated reflectivity, crystal resolution, and 2^{nd} to 1^{st} order reflectivity ratio (2^{nd} order correction below 950eV)

2) Evaluating absolute source intensities, instrumental broadening, plasma line-widths.

3) Develop intuition and a path to model crystal performance that could be used in a larger parameter space (various crystal curvatures, geometries, orders, photon energy…)

Results

1) We accurately measured integrated reflectivities for a set of KAP crystal curvatures (flat, 2,4, 6 and 9in) and diffraction orders (1^{st}, 2^{nd} and 3^{rd})
2) Integrated reflectivities show good agreement with XOP multilamellar model with a set of Debye-Waller factors (temperature factors) depending on crystal curvature and order of diffraction. It is possible that they could also depend on photon energy but the present data is too limited to be conclusive.
4) Width for 2^{nd} and 3^{rd} order agree relatively well with XOP multilamellar model
5) Widths in 1^{st} order are systematically measured higher than any calculation due to extra instrumental broadenings, this might be solved through deconvolution.
6) Width and spectral shape in 1^{st} order are measured with a conventional x-ray source
7) Crystal efficiencies were measured to high accuracy using NIST calibrated KERMA source