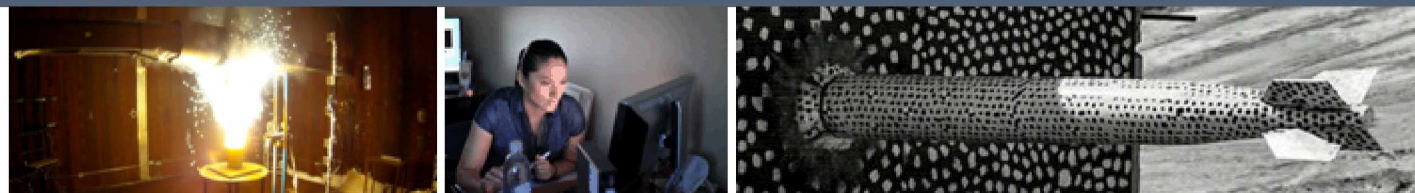


# Analysis of sample KAP crystals from INRAD



PRESENTED BY

Greg Dunham, October 22, 2019

Meeting with INRAD



Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

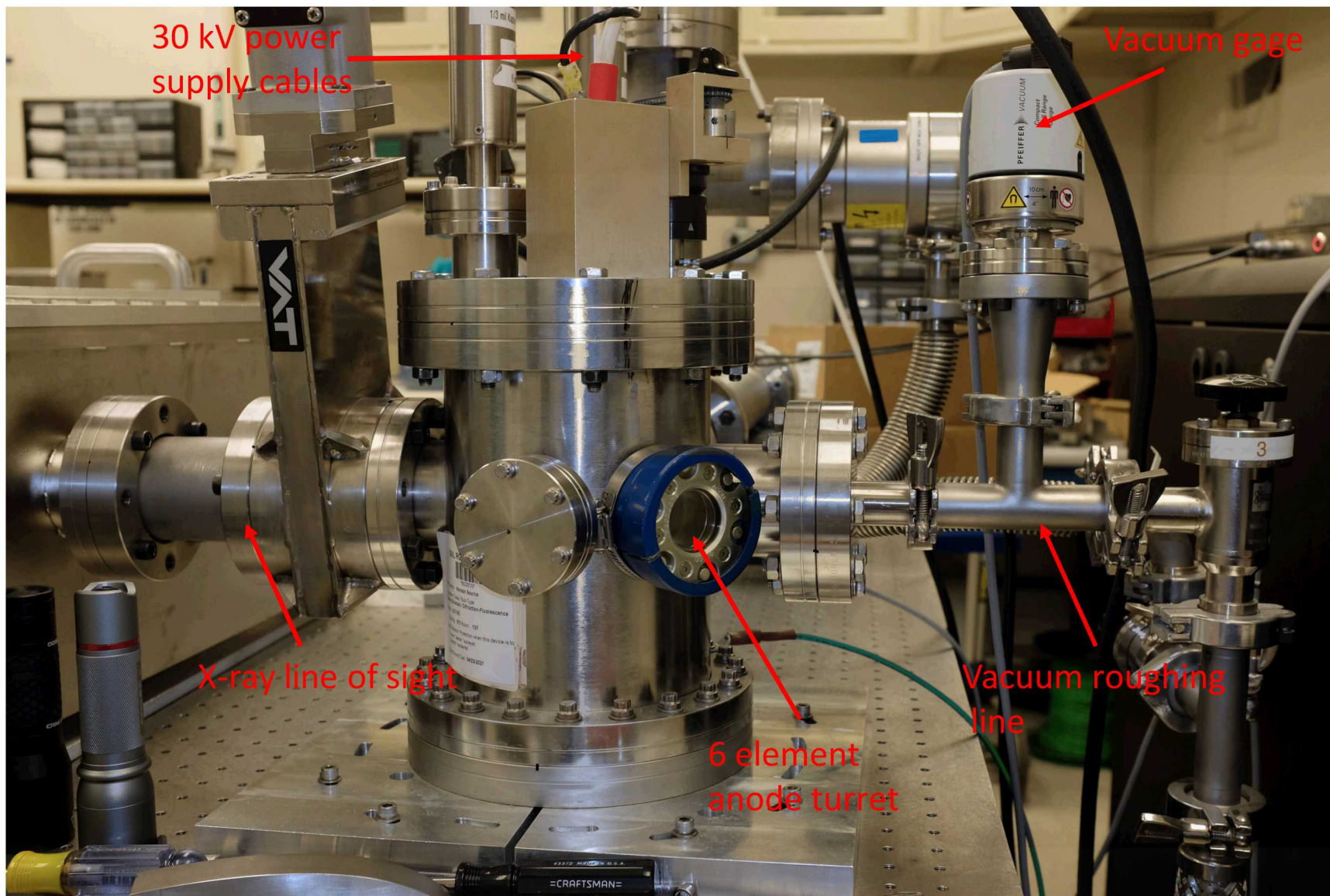
# Outline

- Samples from INRAD
- Measurement technique for calibrations
- Images and data
  - Full images, detail image, and a vertical lineout
  - Horizontal lineout, line fitting and width measurements
  - Data table showing line widths and resolution
- Sample calibration dataset from nominal KAP crystals used for opacity measurements and other campaigns
- Summary

# INRAD supplied KAP crystal samples

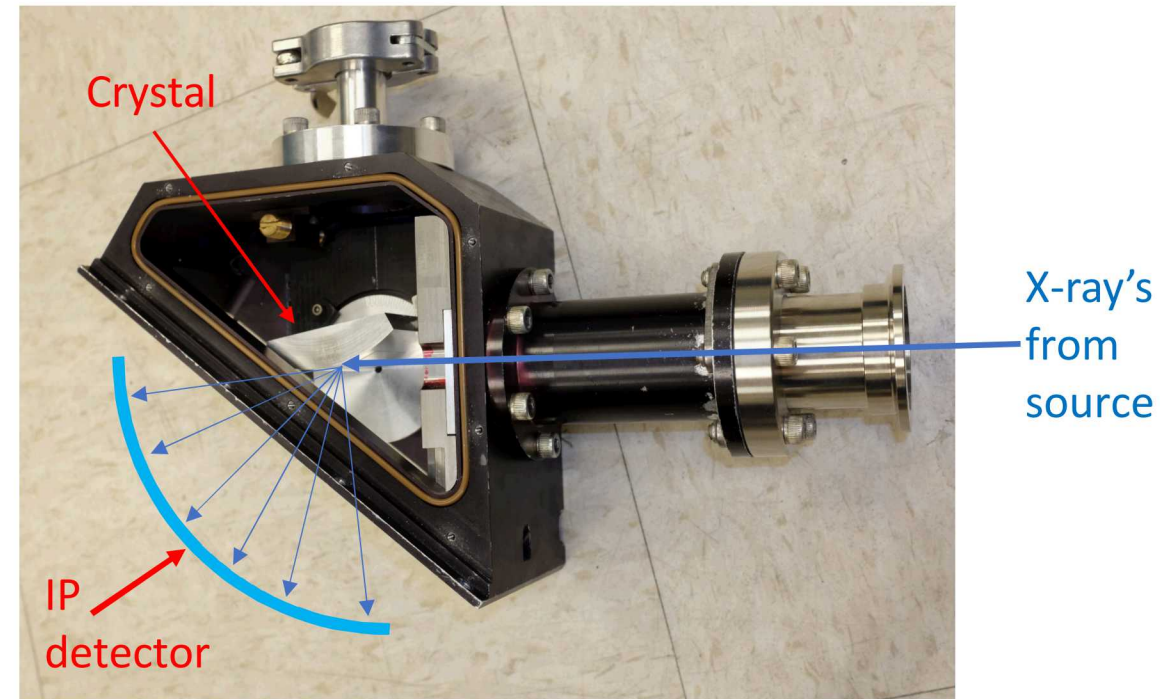
- Two 4" bend radius convex KAP crystals
  - 6482-4-1 & 6482-5-2
- Two 6" bend radius convex KAP crystals
  - 6482-6-1 & 6482-7-2
- Two mounting techniques were used with samples of each in both sizes.

# Manson Source – 6 anode, 30 kV x-ray source



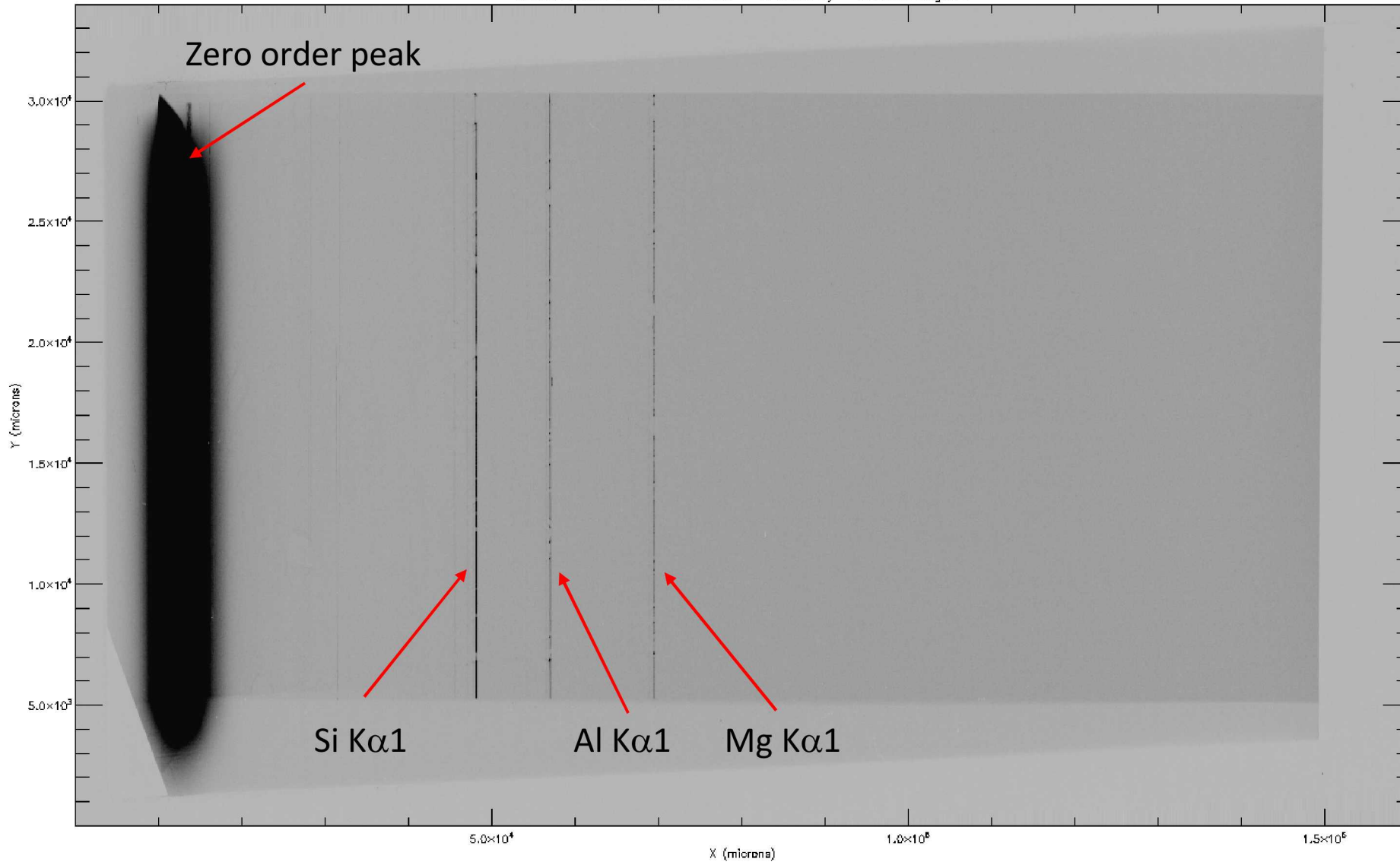
# Calibration data obtained using x-rays from a Manson source.

- Typical calibration setup
  - Mg, Al and Si anodes are excited to generate characteristic K-shell emission spectra at 1.254 keV, 1.487 keV, and 1.74 keV, respectively.
  - Crystals are installed in a time-integrating 1D-space resolving spectrometer (TIXTL).
  - Expose signal onto image plate (Fuji TR IP) for approximately 1 hour for each anode.
  - Scan data on an image plate scanner.
  - Analyze to determine qualitative and quantitative measurements of crystal quality and performance.



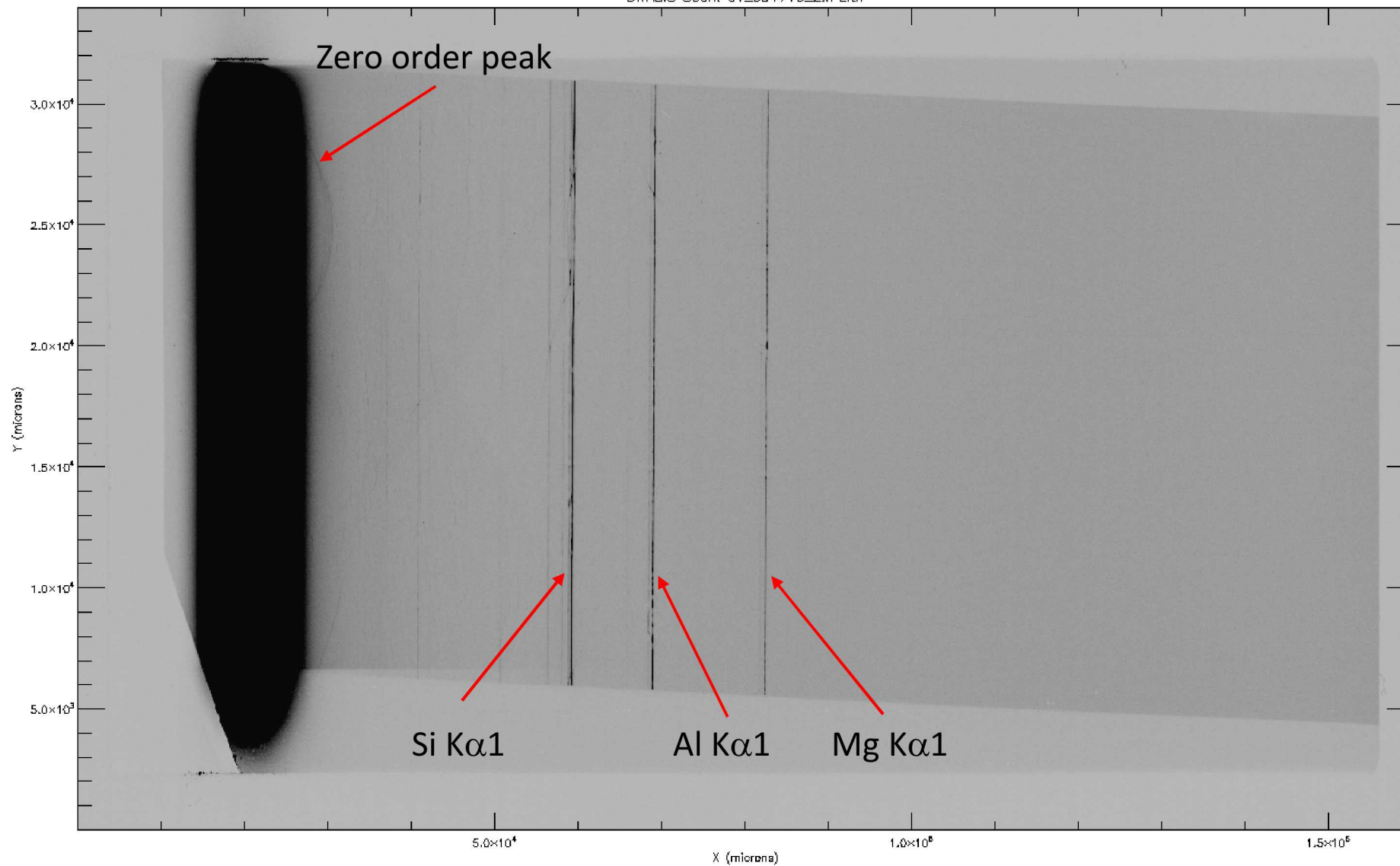
# 4" KAP 6482-4-1 (INRAD)

DITABIS Scan: c1\_090319.IPL.tif - Rotated by -1.00000 degrees

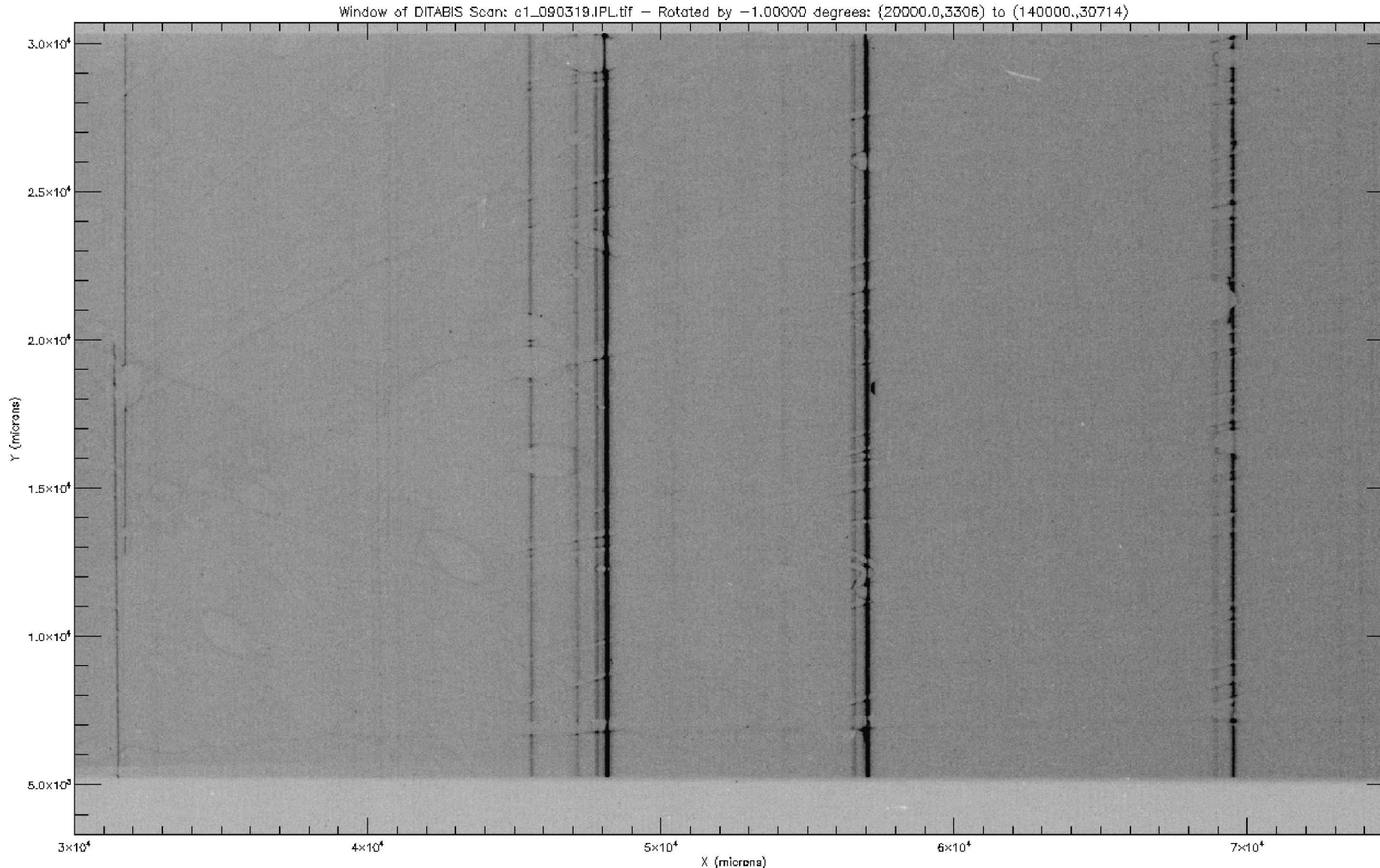


# 4" KAP 6482-5-2 (INRAD)

DITABIS Scan: e1\_091719\_2.IPL.tif



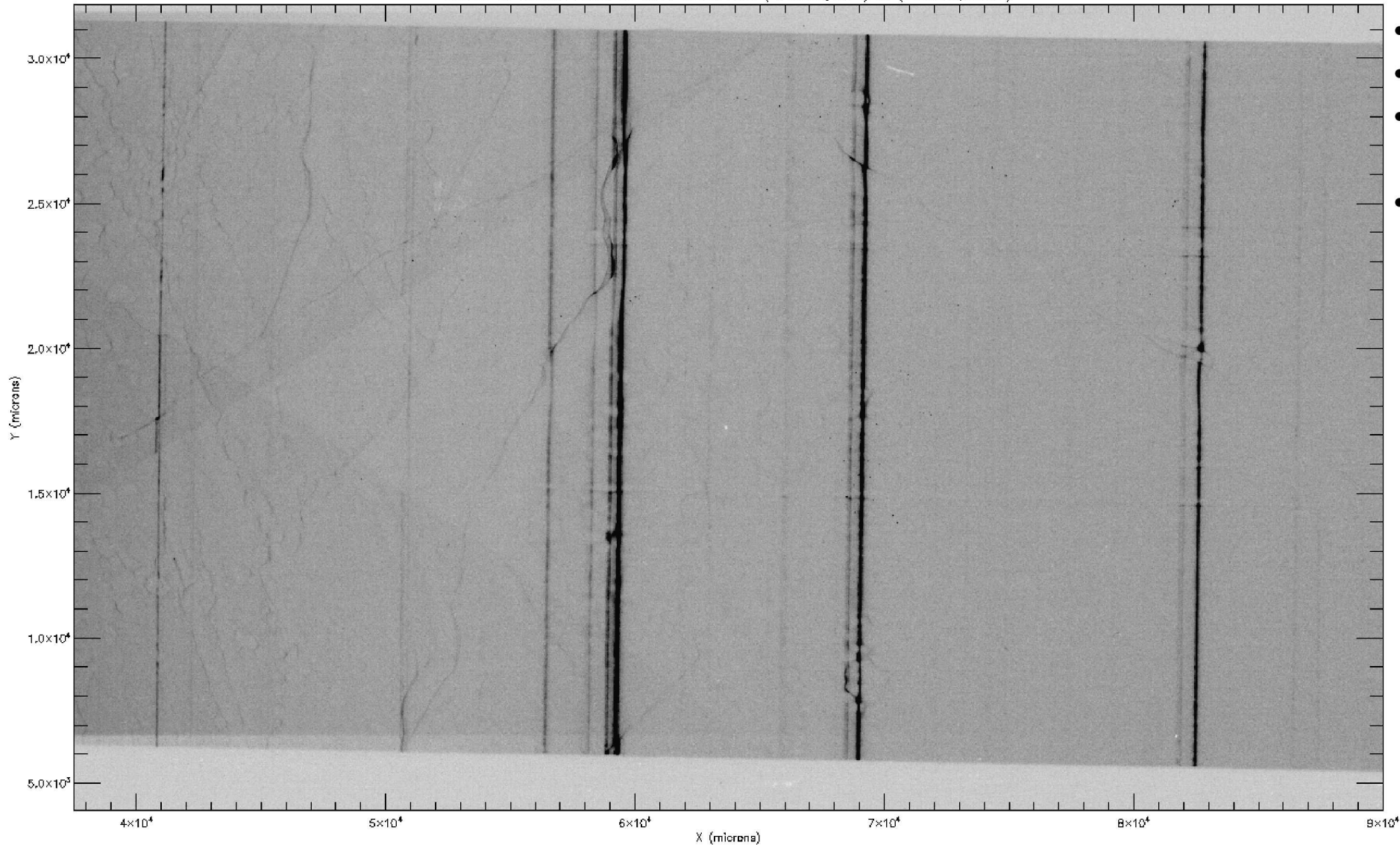
# 4" KAP 6482-4-1 Detail



- Spectral lines are straight
- Bubbles are noticeable throughout image
- Higher order reflections create a cross pattern
- Lines are broken causing intensity variations
- Off-diagonal features extend across image

# 4" KAP 6482-5-2 Detail

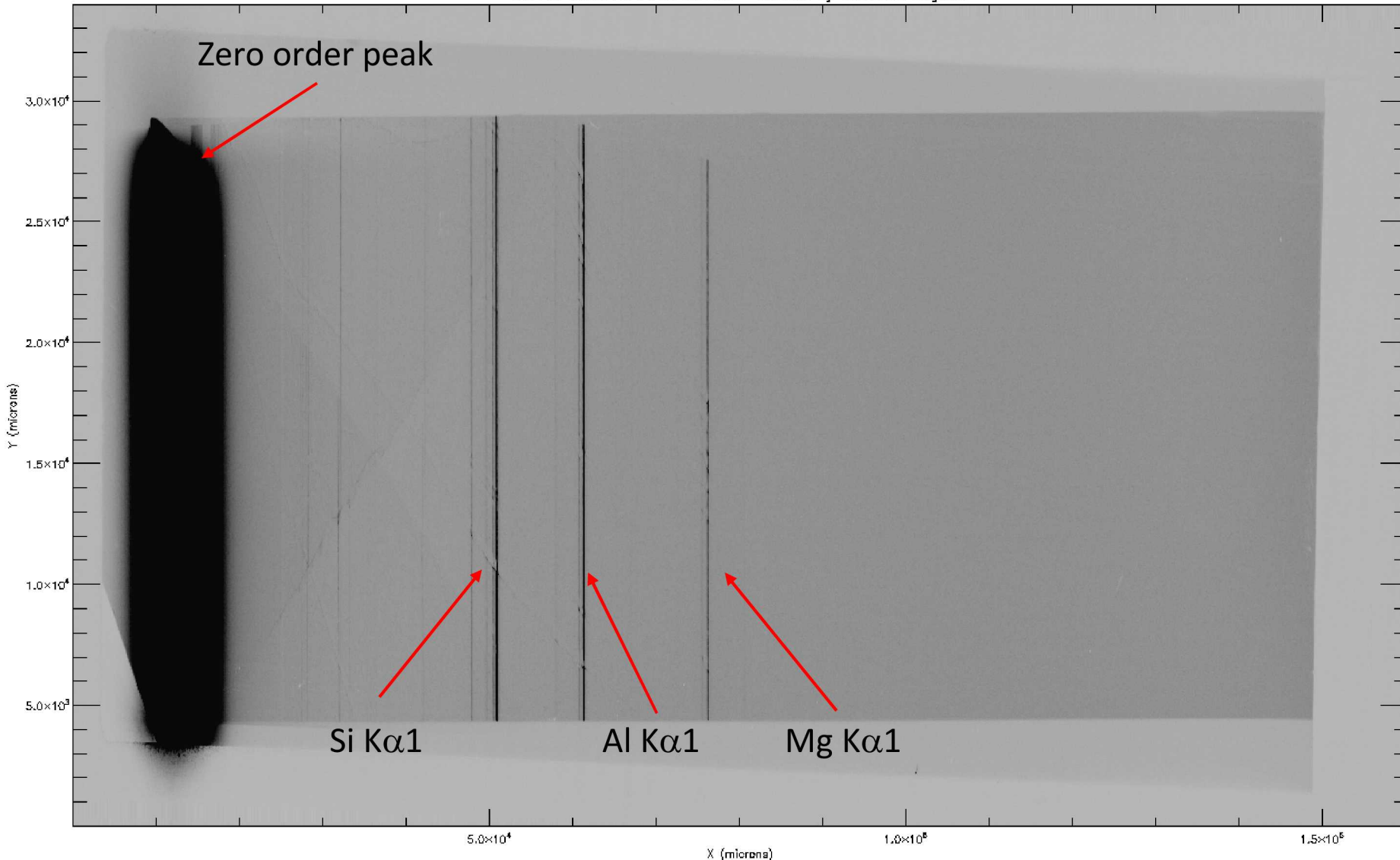
Window of DITABIS Scan: e1\_091719\_2.IPL.tif: (27000,0,4041) to (140000,31862)



- Spectral lines not as straight
- Numerous wavy features
- Higher order reflections create a cross pattern
- Lines are broken causing intensity variations

# 6" KAP 6482-7-2 (INRAD)

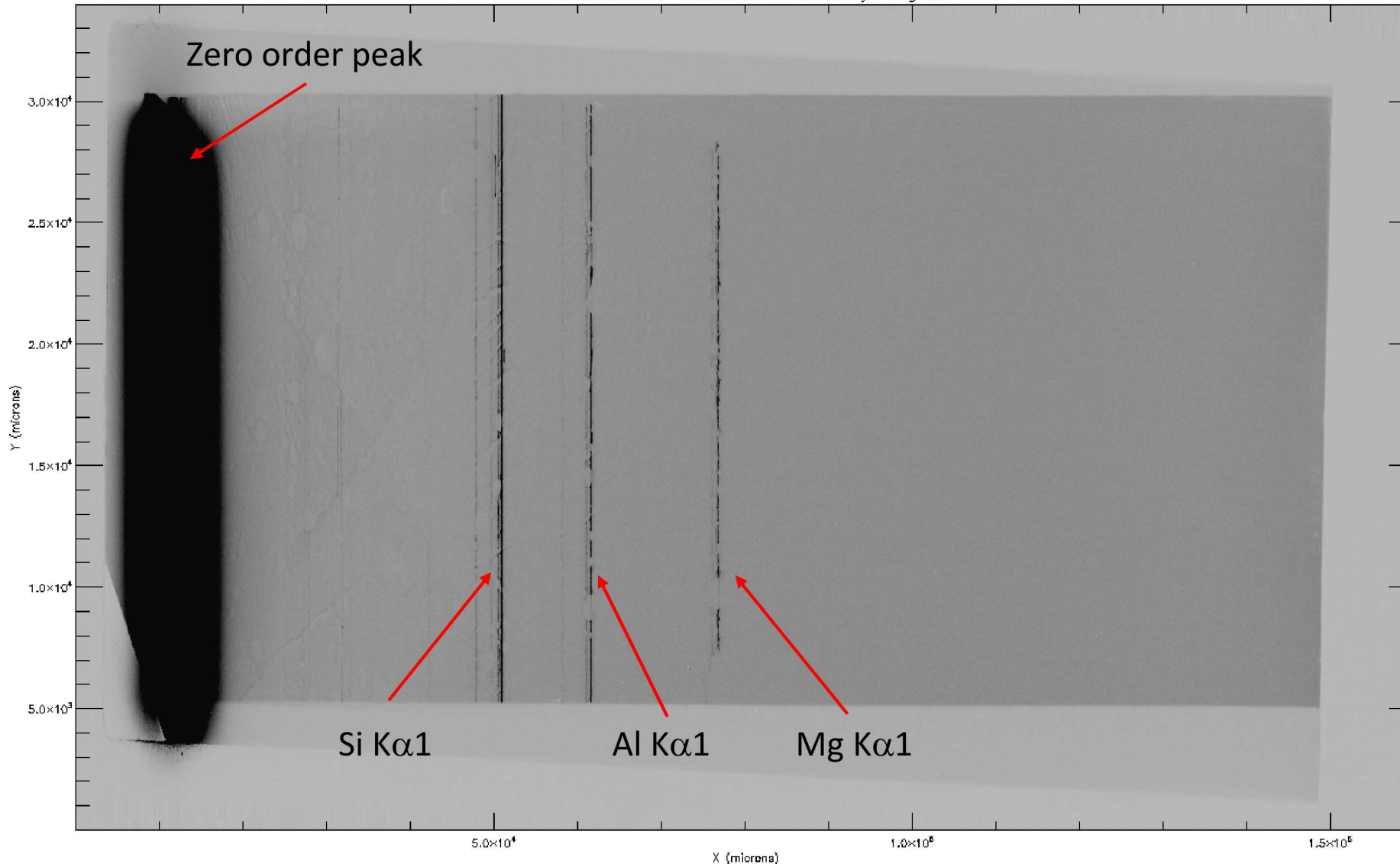
DITABIS Scan: c1\_101019.IPL.tif - Rotated by 0.800000 degrees



- Qualitatively cleaner than 4" crystals
- Higher order reflections create a cross pattern
- Crystal planes appear to truncate at higher  $\lambda$
- Spectral lines are straight

# 6" KAP 6482-6-1 (INRAD)

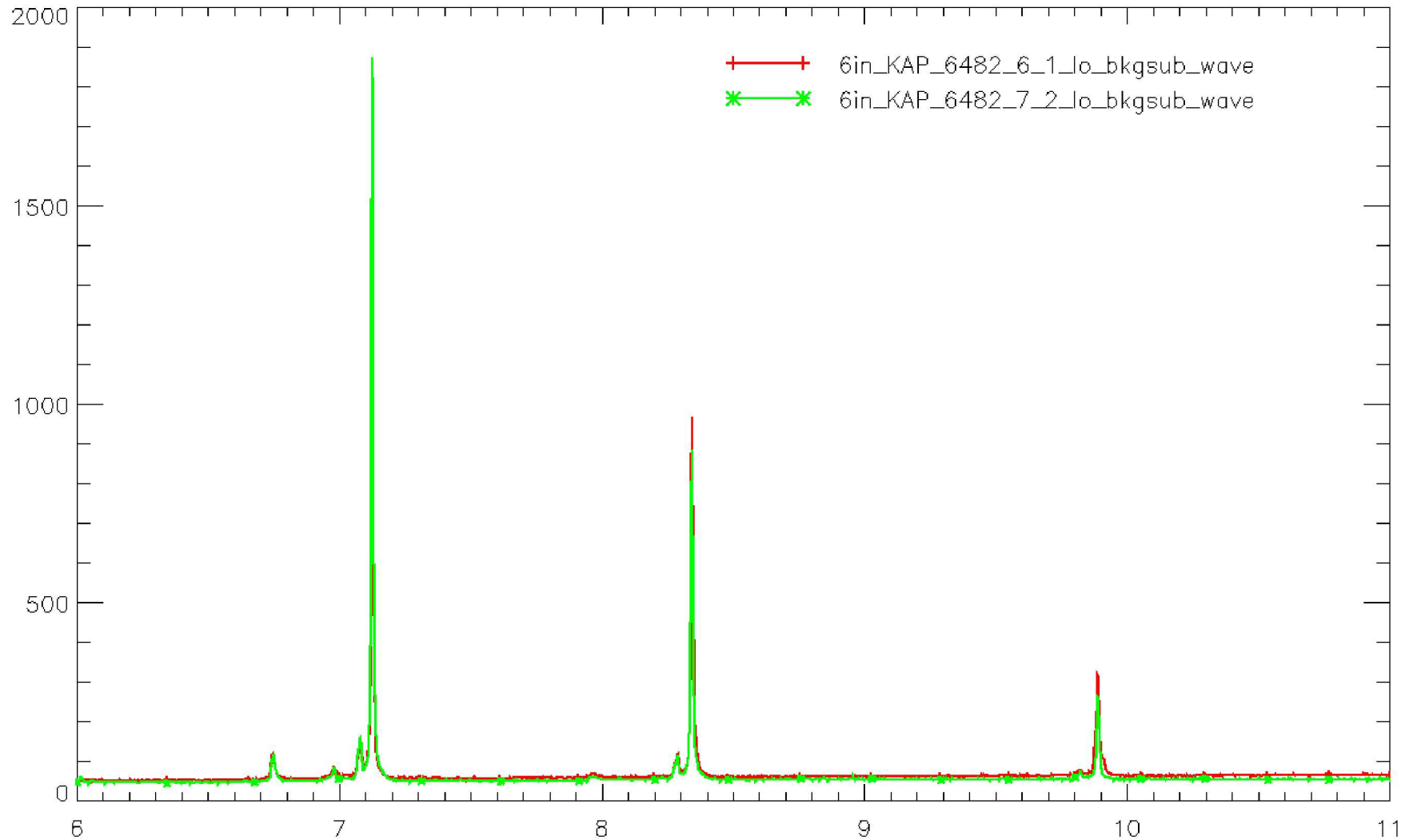
DITABIS Scan: c1\_101619.IPL.tif - Rotated by 1 degree



- Qualitatively similar level of imperfections
- Higher order reflections create a cross pattern
- More pronounced crystal planes truncation at higher  $\lambda$
- Spectral lines are straight

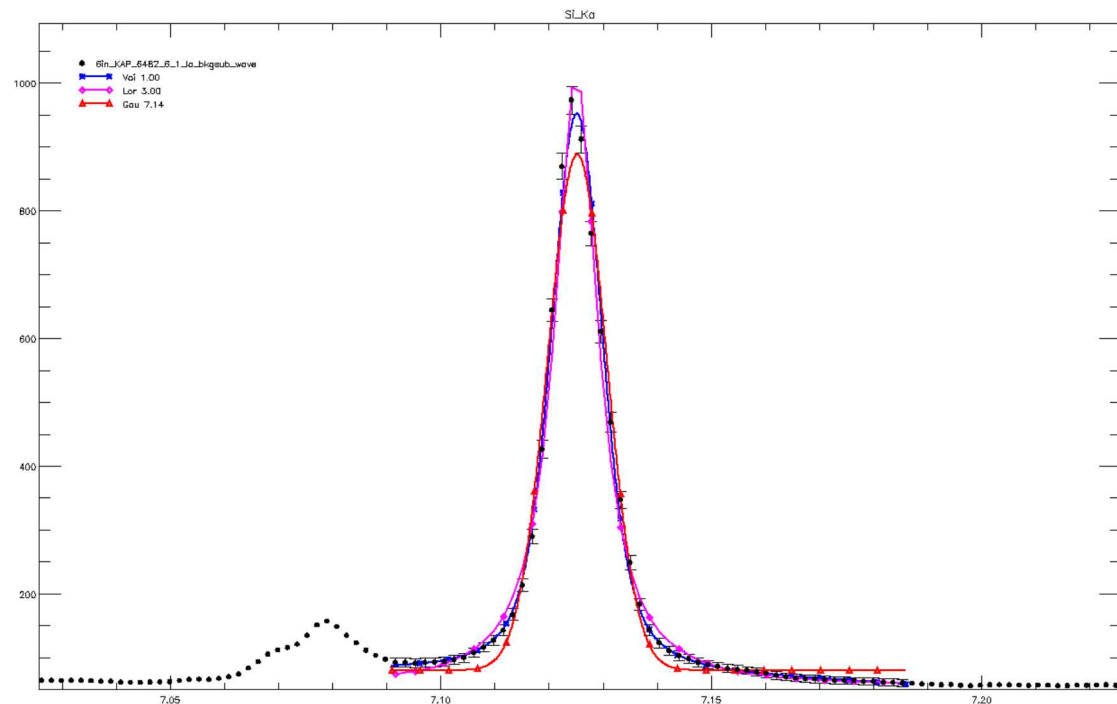


# Lineout through central region



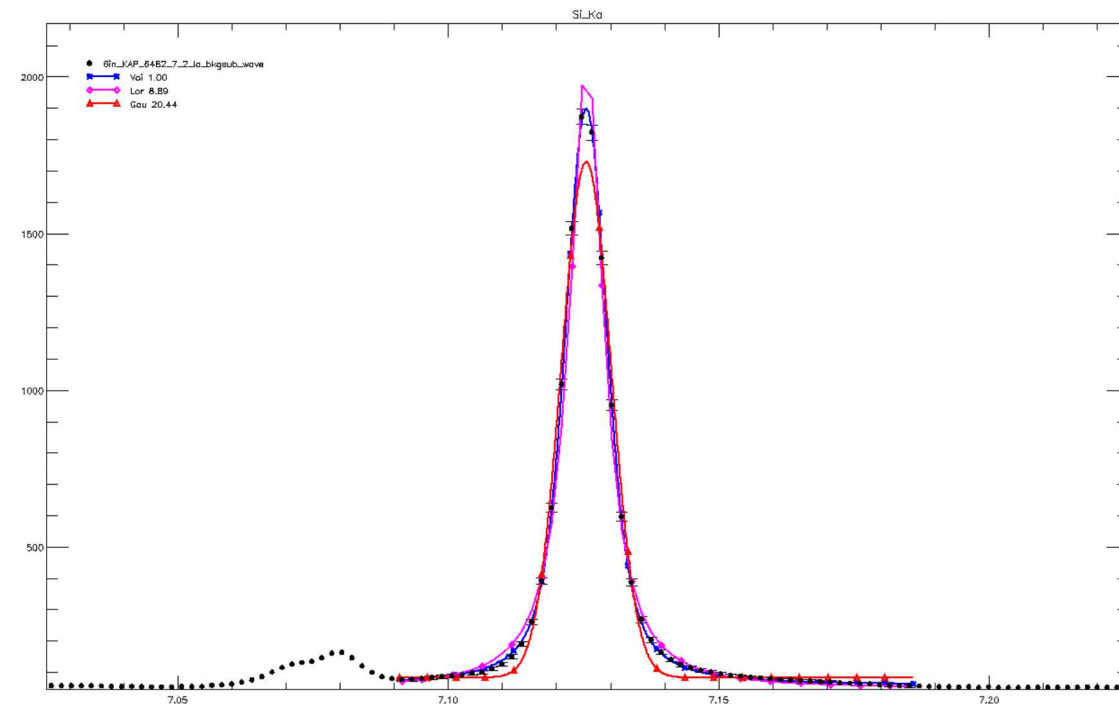
# Line fits to Si K $\alpha$ 1

6" KAP 6482-6-1



Voigt fwhm (mAng.), error , error (%)	11.30774	0.59720	5.28136
Lorentzian FWHM FL (mAng), error , error (%)	5.25531	0.54500	10.37046
Gaussian FWHM FG (mAng), error , error (%)	8.13868	0.49408	6.07076

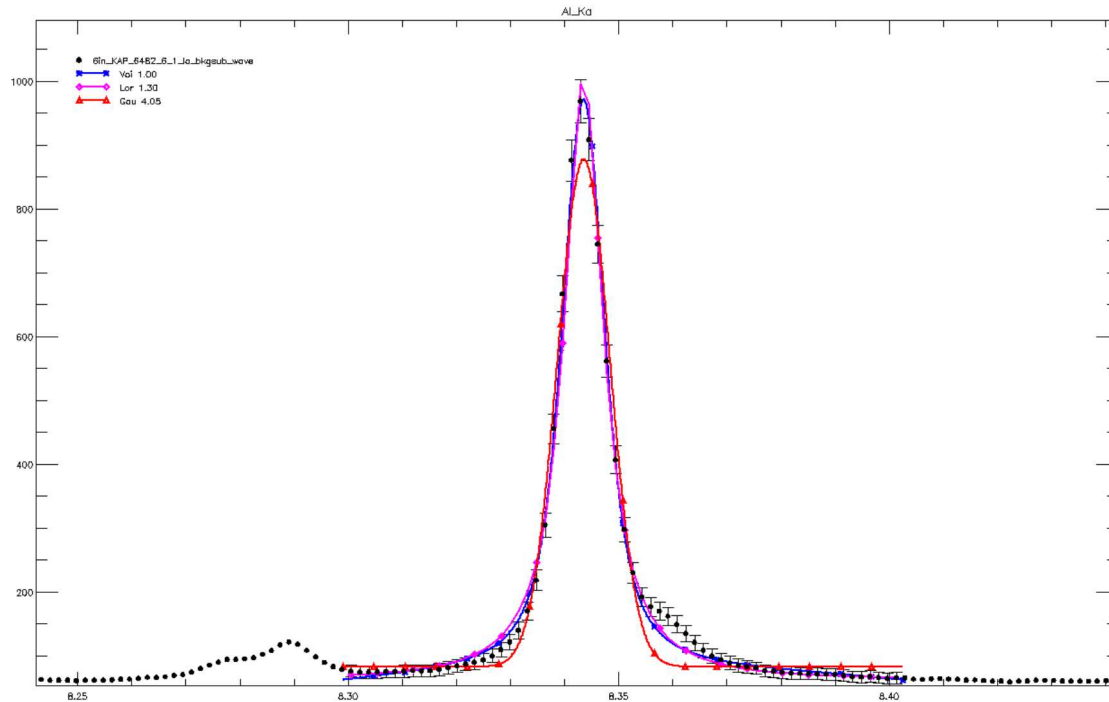
6" KAP 6482-7-2



Voigt fwhm (mAng.), error , error (%)	9.28261	0.21658	2.33318
Lorentzian FWHM FL (mAng), error , error (%)	4.99229	0.16775	3.36028
Gaussian FWHM FG (mAng), error , error (%)	6.19218	0.19459	3.14251

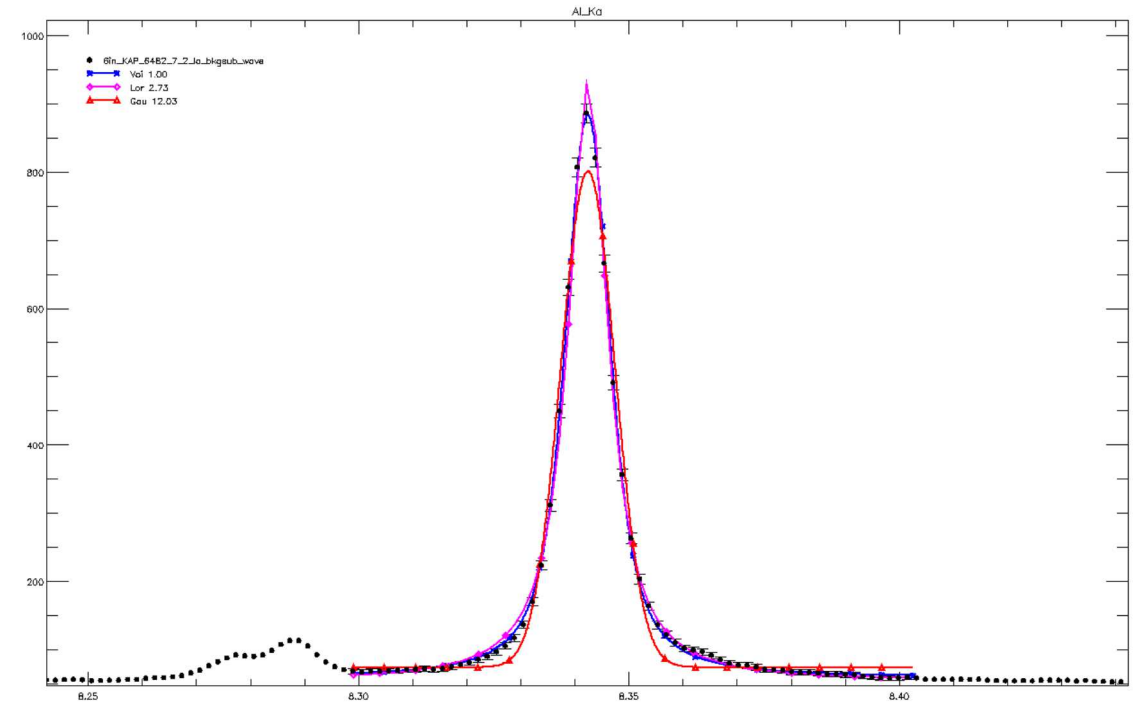
# Line fits to Al K $\alpha$ 1

6" KAP 6482-6-1



Voigt fwhm (mAng.), error , error (%)	9.48205	0.85788	9.04736
Lorentzian FWHM FL (mAng), error , error (%)	6.21853	0.67775	10.89894
Gaussian FWHM FG (mAng), error , error (%)	5.43511	0.78103	14.37016

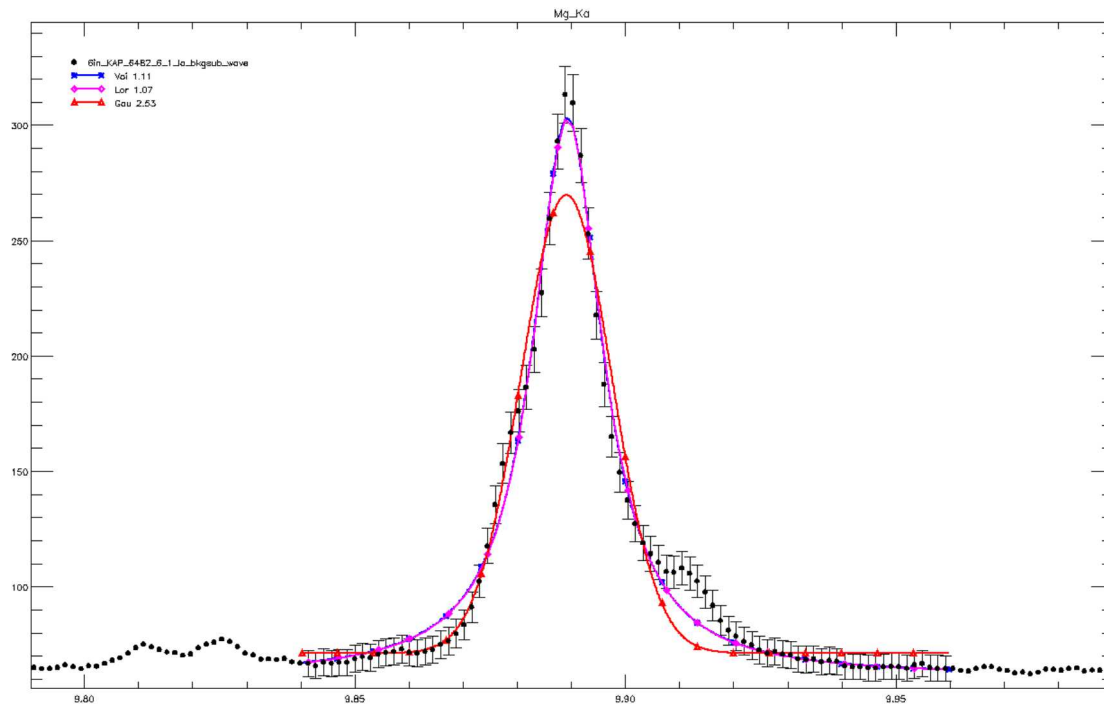
6" KAP 6482-7-2



Voigt fwhm (mAng.), error , error (%)	9.93867	0.32972	3.31756
Lorentzian FWHM FL (mAng), error , error (%)	6.67366	0.23868	3.57643
Gaussian FWHM FG (mAng), error , error (%)	5.56254	0.31508	5.66438

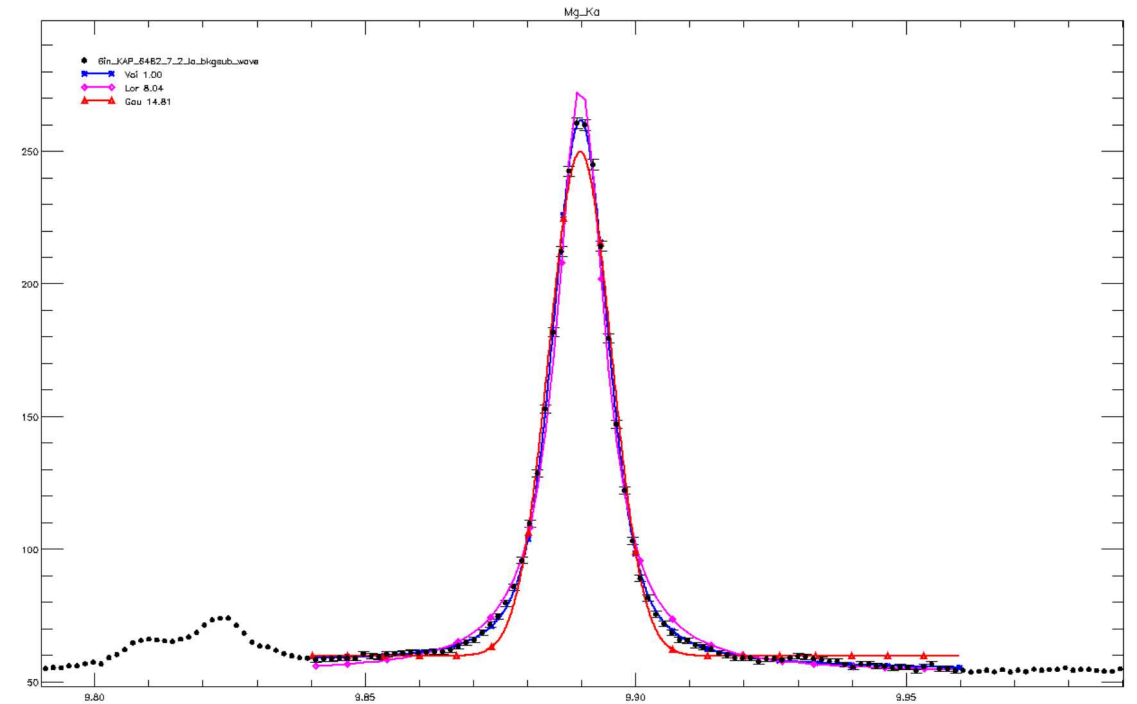
# Line fits to Mg K $\alpha$ 1

6" KAP 6482-6-1



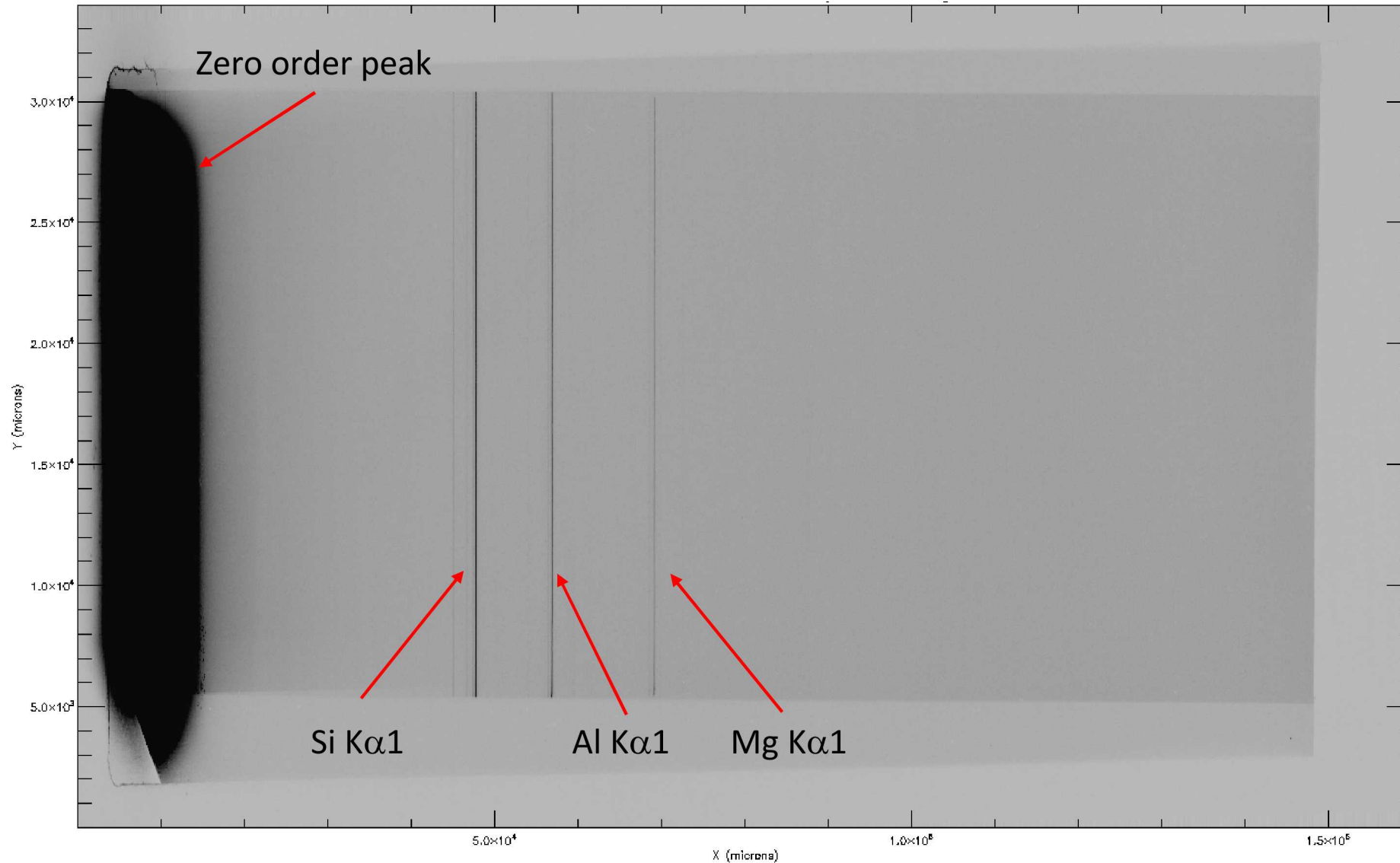
Voigt fwhm (mAng.), error , error (%)	15.74677	0.00005	0.00029
Lorentzian FWHM FL (mAng), error , error (%)	15.72980	0.00003	0.00022
Gaussian FWHM FG (mAng), error , error (%)	0.49805	0.00042	0.08526

6" KAP 6482-7-2



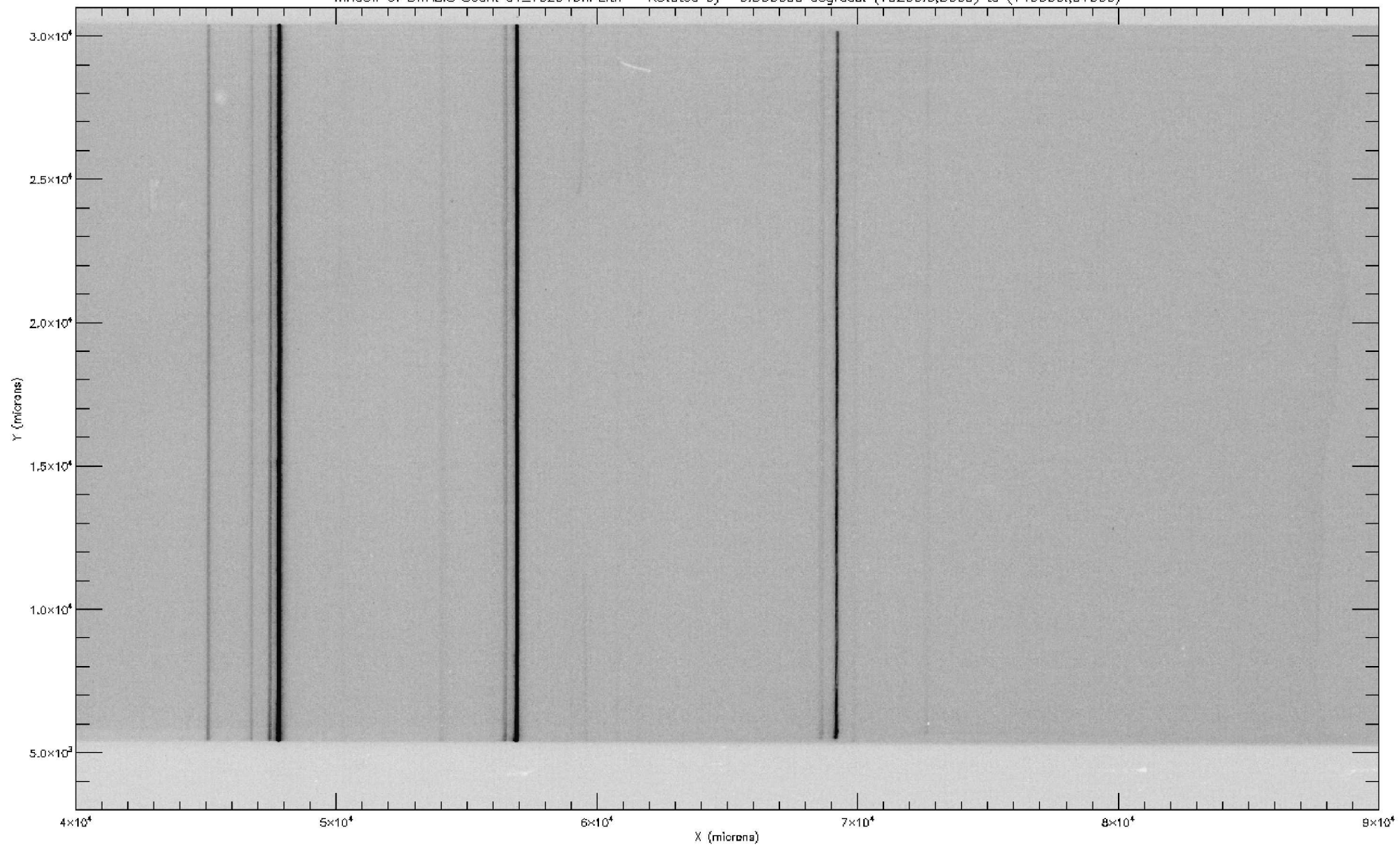
Voigt fwhm (mAng.), error , error (%)	12.28170	0.23901	1.94607
Lorentzian FWHM FL (mAng), error , error (%)	6.55803	0.20325	3.09930
Gaussian FWHM FG (mAng), error , error (%)	8.22793	0.20540	2.49642

# 2" KAP crystal used on Z experiments (from usual supplier)

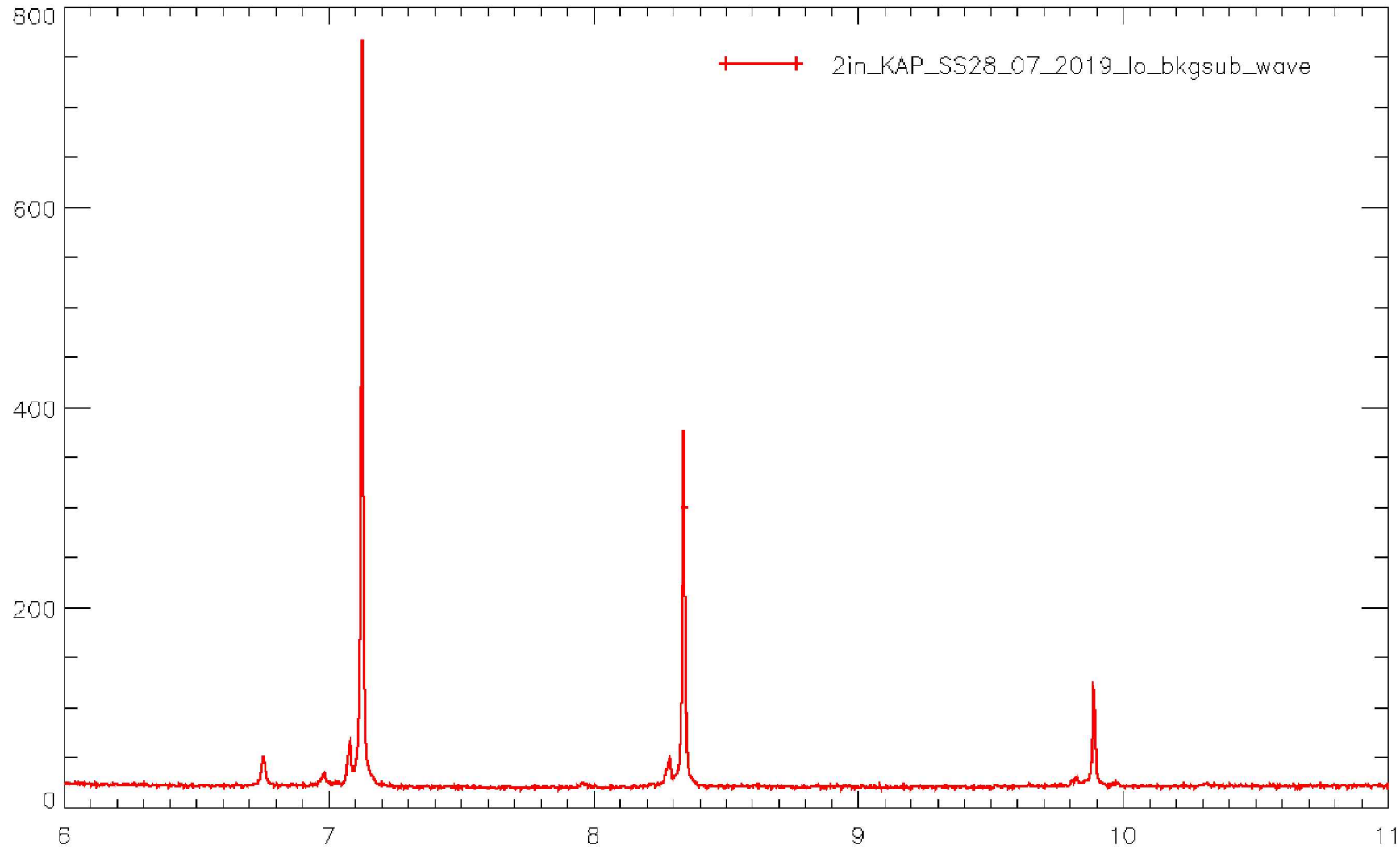


# 2" KAP SS28 07 2019 Detail

Window of DITABIS Scan: c1\_102919.IPL.tif - Rotated by  $-0.500000$  degrees: (15200.0,3000) to (140000.,31000)



# 2" KAP SS28 07 2019 Detail Lineout



# Line widths in milli Angstroms from Voigt fits

Crystal ID	6482-4-1	6482-5-2	6482-6-1	6482-7-2	SS28-07-2019
Type	4" KAP	4" KAP	6" KAP	6" KAP	2" KAP
Si K $\alpha$ 1	10.203 $\pm$ 0.166	10.123 $\pm$ 0.284	11.291 $\pm$ 0.539	9.283 $\pm$ 0.217	9.565 $\pm$ 0.233
Al K $\alpha$ 1	10.786 $\pm$ 0.183	11.186 $\pm$ 0.303	9.444 $\pm$ 0.749	9.939 $\pm$ 0.330	10.380 $\pm$ 0.258
Mg K $\alpha$ 1	12.163 $\pm$ 0.475	11.366 $\pm$ 0.359	15.986 $\pm$ 0.054	12.282 $\pm$ 0.239	11.345 $\pm$ 0.480

## Resolution (E/ $\delta$ E)

Crystal ID	Line	6482 4 1	6482 5 2	6482 6 1	6482 7 2	ss28 07 2019
Type	Energy (keV)	Resolution	Resolution	Resolution	Resolution	Resolution
Si K $\alpha$ 1	1.740	698	704	630	768	745
Al K $\alpha$ 1	1.487	773	746	880	839	803
Mg K $\alpha$ 1	1.254	813	870	628	805	872

# Summary

- Crystal resolution is on par with nominal crystals used routinely at Z
- Spectral lines are straight showing acceptable waviness
- Broken (discontinuous) spectral lines detract from quality creating variations of 2x or more in intensity in the space resolving direction over a single spectral feature
- Circular features from bubbles or contaminants are too numerous
- Many issues, possibly due to crystal quality, substrate preparation, environmental cleanliness, or mounting technique, render current KAP samples unsuitable for quantitative spectroscopy

# Cleanroom Definition

Class	Maximum particles/m <sup>3</sup> <sup>a</sup>						FED STD 209E
	≥0.1 μm	≥0.2 μm	≥0.3 μm	≥0.5 μm	≥1 μm	≥5 μm	equivalent
ISO 1	10 <sup>b</sup>	<sup>d</sup>	<sup>d</sup>	<sup>d</sup>	<sup>d</sup>	<sup>e</sup>	
ISO 2	100	24 <sup>b</sup>	10 <sup>b</sup>	<sup>d</sup>	<sup>d</sup>	<sup>e</sup>	
ISO 3	1,000	237	102	35 <sup>b</sup>	<sup>d</sup>	<sup>e</sup>	Class 1
ISO 4	10,000	2,370	1,020	352	83 <sup>b</sup>	<sup>e</sup>	Class 10
ISO 5	100,000	23,700	10,200	3,520	832	<sup>d,e,f</sup>	Class 100
ISO 6	1,000,000	237,000	102,000	35,200	8,320	293	Class 1,000
ISO 7	<sup>c</sup>	<sup>c</sup>	<sup>c</sup>	352,000	83,200	2,930	Class 10,000
ISO 8	<sup>c</sup>	<sup>c</sup>	<sup>c</sup>	3,520,000	832,000	29,300	Class 100,000
ISO 9	<sup>c</sup>	<sup>c</sup>	<sup>c</sup>	35,200,000	8,320,000	293,000	Room air