

# WDS Explained

Wavelength Dispersive X-ray Spectrometry (WDS) uses the characteristic X-rays generated from a sample bombarded with electrons to identify the elemental constituents comprising the sample. It generates a spectrum in which the peaks correspond to specific X-ray lines. Measurement of peak intensities can be used to provide a full quantitative analysis.

## Advantages of WDS in the SEM

- Excellent Spectral Resolution
- Separation of overlapping peaks
- Detection and quantification of minor and trace elements

## Principles of WDS

Whereas Energy Dispersive Spectrometry (EDS) acquires the full range of energies simultaneously, wavelength dispersive spectrometry (WDS) acquires a spectrum sequentially over a range of wavelengths.

The WD spectrometer is fitted on a port of the SEM, usually at an identical take off angle to the ED detector. Inside the spectrometer, analysing crystals with a specific lattice spacing (1) are used to diffract characteristic X-rays from the sample into the detectors (5).

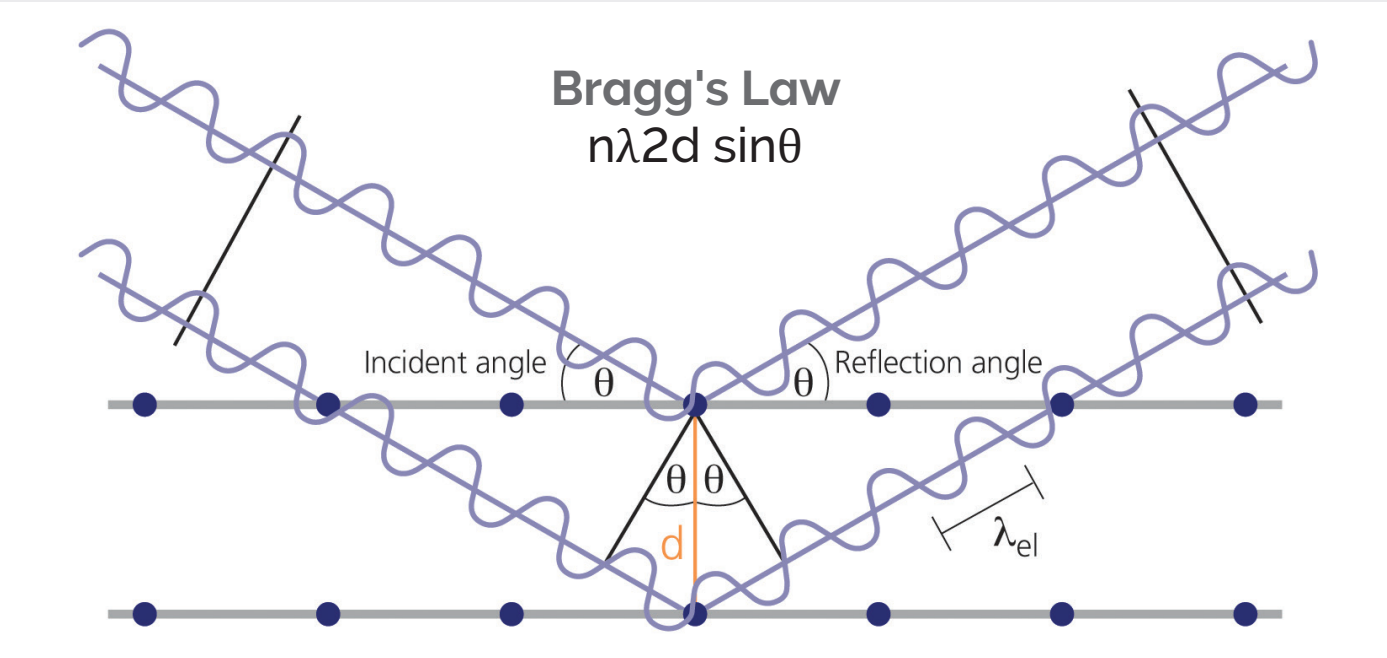
The wavelength of the X-rays diffracted into the detector is selected by changing the position of the analysing crystals and detectors with respect to the sample. This selection is done according to Bragg's Law (see below).

The diffracted X-rays are focused to a point just in front of the detector where they pass through a slit (4). The width of this entrance slit determines the peak resolution seen in the spectrum. Once the X-rays have been collimated by the slit, they are detected by one or two gas proportional X-ray counters set in series (5).

Following detection, the Wave spectrometer electronics measures the signals and determines the energy of each X-ray measured. A spectrum of intensity against energy is then created. Note the significantly improved resolution and peak to background of the WDS spectrum (blue) compared to the EDS spectrum (yellow).

## Bragg's Law

Bragg's Law ( $n\lambda = 2d\sin\theta$ ) determines the angle required to detect a characteristic X-ray.  $\lambda$  is the wavelength of the characteristic X-ray;  $d$  is the lattice spacing of the diffracting crystal; and  $\theta$  is the angle between the X-ray and the crystal. Characteristic X-ray detection only occurs when Bragg's Law is met. The position of the crystal changes every time another element is to be measured. A number of different crystals are provided as no single crystal will be able to diffract all the wavelengths required.



## AztecWave

AZtecWave combines the unique power of WDS to resolve X-ray peaks and quantify minor and trace elements with the speed and flexibility of EDS. It uses real-time input from EDS and SEM to optimise WDS set-up, guaranteeing optimal performance and accurate results for all levels of user expertise. By combining WDS and EDS together in this way, the composition of elements at all levels of concentration are accurately determined with only a single WDS spectrometer mounted on the SEM.

Element	Si	Mn	P	Co	Ti	Cr/Fe/Ni
Published reference values (wt. %)	0.59	0.90	0.011	0.022	0.30	-
Technique used	EDS	EDS	WDS	WDS	EDS	EDS
Average (wt.%)	0.614	0.961	0.009	0.021	0.303	Rem.
1σ	0.027	0.077	0.002	0.003	0.262	
# analysis points	6	6	6	4	6	

Crystal	2d (nm)	Energy Range (keV)	Element Range (K Line)	Type	Optimised Element
LSM200	19.7	0.07 – 0.22	Be to B	Johann	B
LSM-80N	7.8	0.17 - 0.56	B to O	Johann	C
LSM80E	7.8	0.17 - 0.56	B to O	Johann	N
LSM60	6.0	0.22 – 0.73	C to F	Johann	O
TAP	2.575	0.52 – 1.70	O to Al	Johansson	
PET	0.8742	1.54 – 4.99	Si to Ti	Johansson	
LiF (200)	0.40267	3.33 – 10.84	Ca to Ge	Johansson	
LiF (220)	0.28473	4.712 – 15.33	V to Y	Johansson	

## Crystals

The wavelength of the X-rays diffracted into the detector is selected by varying the position of the analysing crystal with respect to the sample. 4 - 6 crystals are supplied with each spectrometer depending on the intended application. Each has a specific lattice spacing that diffracts a range of characteristic X-rays from the sample into the detector according to Bragg's Law (see Table).

## Rowland Circle

To maintain the correct geometrical relationship between specimen, crystal and detector for the full range of diffracted angles, it is necessary to maintain all three on a circle (known as the Rowland circle). Movement is accomplished by a mechanical goniometer which moves the crystal and detector so that correct diffraction conditions are maintained.

## Spectrometer Geometry

If a spectrometer is mounted with the Rowland circle vertical (typical of dedicated microprobes), it is necessary to position the sample height very accurately to maintain diffraction geometry. In the SEM, accurate vertical positioning is not critical because the spectrometer is mounted with the Rowland circle inclined. Thus the sample remains on the Rowland circle over a large range of sample heights, X-ray intensity does not change, and the correct height is easily achieved using just the electron image.

## Entrance Slit

Located in front of the detector, the entrance slit is used to remove unwanted non-characteristic X-rays. The width of the slit optimises the X-ray peak widths and helps to maximise the performance of the spectrometer for any given X-ray line.

## Detectors

With the X-ray beam focused by the crystals in a WDS, proportional counter detectors offer sufficient resolution to be used as the X-ray detectors. When the X-ray enters the detector through a thin window, it is absorbed by atoms of the counter gas. Photo-electrons are ejected, and these produce an electrical pulse which is proportional to the energy of the X-ray causing the event in the first place. Wave spectrometer has two proportional counters working together to optimise count rates for all elements from Be - Pu. A flow proportional counter optimises low energy count rates while a sealed proportional counter optimises the high energy count rates.

