**EDAX** ®
Smart Insight

# Diffraction Peak Removal Software

Since the optical configuration of a direct excitation EDXRF spectrometer is in essence the same as that of a fixed position X-ray Diffractometer, Bragg diffraction peaks will be generated from crystalline sample materials, some of which may be directed towards the detector system. The effect becomes more prominent where the sample is a single crystal, such as a gemstone. Bragg diffraction peaks are superimposed upon the sample matrix's analyte energy spectrum with a peak position where the X-ray wavelengths ($\lambda$) satisfy Bragg's law for a fixed geometry ($\theta$) between the X-ray source, suitably oriented crystallographic planes of appropriate interatomic spacing (**d**) within the sample and the detector.
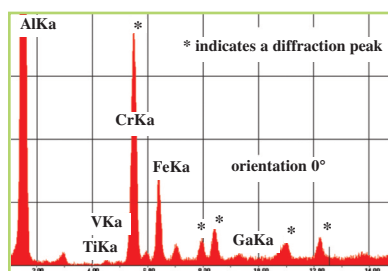
**Bragg's Law:**     $n\lambda = 2d \sin \theta$



Figure 1. EDXRF measurement of Ruby measured at reference orientation, 0 degrees. Diffraction peaks are denoted by * marks.

Bragg diffraction peaks can yield information about crystal structure, such as crystal phase and atomic spacing, but can also interfere with X-ray fluorescence peaks of the crystal's elemental analyte spectrum. For example, the above spectrum in Figure 1 was acquired from the "table" of a cut natural ruby with an orientation arbitrarily defined as 0° with respect to the X-ray source and detector.

The elements Ti, V, Cr, Fe and Ga are of particular interest in the analysis of natural rubies, as they can be used to identify the geological source of a ruby. The region of Cr(K) is obscured in this spectrum by a diffraction peak.

By reorienting the ruby (e.g. rotating about the table's normal with respect to the X-ray source/detector), other planes within the matrix may be brought into a position to satisfy the fixed geometry (i.e. $\theta$ in Bragg's law), but now for different wavelengths/energies. The result is that the diffraction peaks appear at different locations along the spectrum's energy scale. Planes parallel to the table surface are unaffected but, for the geometry of the EDXRF system in this example, planes in or close to this orientation cannot contribute to any observed diffraction peaks.

Observe the following two spectra in Figure 2 and Figure 3 where the ruby has been reoriented by 90° and 180° degrees with respect to the initial position. Notice how, for example, the apparently high $CrK_\alpha$ analyte peak



Figure 2. EDXRF measurement of Ruby measured at 90 degrees.

disappears when the sample's orientation is changed from the original 0° position, indicating the initial diffraction peak interference.
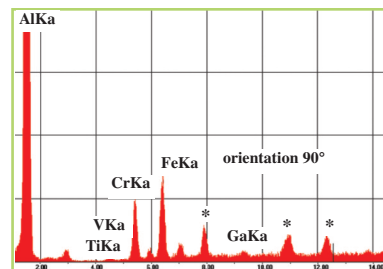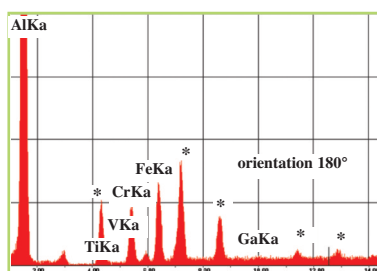


Figure 3. EDXRF measurement of Ruby measured at 180 degrees.

In each spectrum, different regions of the spectrum are affected by the presence of diffraction peaks. If the goal is to evaluate the spectra for fluorescent elemental peak intensities (the intended purpose of an EDXRF spectrometer), it should be possible to remove the diffraction peaks by an appropriate recombination of the various spectra collected at each orientation. An example of such processing can be seen in the spectrum below, which is the result of such a recombination of the previous three spectra acquired at orientations 0°, 90° and 180°.

The methodology can be successful as long as each element or each region of the spectrum is not affected by diffraction peak interference in at least one of the original component spectra. Since five-fold symmetry is nonexistent, spectra acquired at 72°



Figure 4. Recombination of EDXRF spectra at 0, 90 and 180 degrees which eliminates the interfering diffraction peaks in Ruby measurement.

rotational intervals (a maximum of five) should maximize the probability of generating a spectral data set which fulfills this criterion. Of course, if the region of interest is not the entire spectrum, then a successful result is generally possible with fewer spectra as was seen in Figure 4.
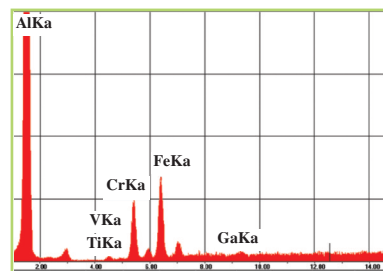
This method was developed by Mr. Alan Seaman and is available in Orbis Vision Software.

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