LEXS: Analysis of Nitride Layers on Steel

APPLICATION NOTE–WDS

Introduction

The EDAX LambdaSpec LEXS is a wavelength dispersive X-ray spectrometer which features X-ray optics designed for parallel beam operation. The high collection optics provide high efficiency for light elements, especially B, C, N and O, with a total range of operation from 80eV to 2400eV.

Application

In order to understand the distribution and composition of the nitrides in steel, high precision X-ray data collection for quantification, X-ray maps and line scans is required.

Spectrometer Conditions

For this application, the LEXS WDS spectrometer was employed. A boron nitride standard was measured (shown in Figure 1) along with a few pure elements to enable quantitative analysis of the nitride unknowns to be calculated. All samples were measured at an accelerating voltage of 10 kV and a beam current between 20 to 25 nA. A tungsten filament SEM was used with a Keithley Pico ammeter to monitor the beam current. EDS spectra were acquired from the samples, along with the WDS data. The simultaneous data acquisition of EDS and WDS information would enable the possibility of using EDS data for some of the higher atomic number samples; however this was not done in this report. Both detectors had a takeoff angle of 35 degrees. The working distance on the instrument used was 17mm. (This distance is specific to the instrument, and could be much smaller if the SEM chamber allows it.)

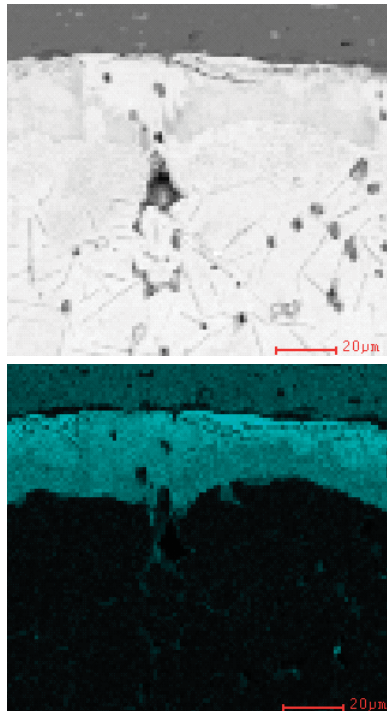

Figure 2: BSE image and N K X-ray map of the layer.

Nitride Measurements

Using the WDS system, the nitride layer on steel was measured and a nitrogen X-ray map was acquired from the layer and the substrate.

Figure 2 shows the SEM image (BSE image) and the nitrogen K X-ray map. The top layer is an Al coat, the bright middle layer is the nitride layer, and the bottom is the substrate. The image is a 128x100 pixel image with a dwell time of 200 msec/pixel. The layer shows different levels of N, perhaps from different phases within the layer. X-ray data was acquired from a spot on the lower part of the nitride layer (which appeared darker), and from the middle, (where the N map appeared brighter). The collected spectra are shown in Figures 3 and 4.

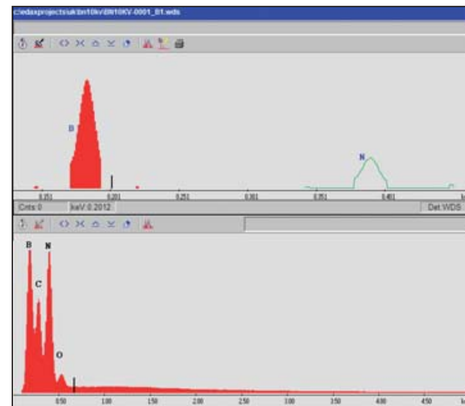

Figure 1: Spectra of BN, the WDS (top) shows the peaks of B K and N K.

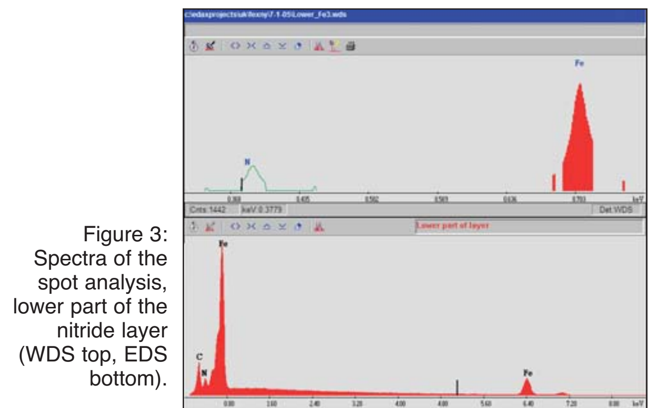

Figure 3: Spectra of the spot analysis, lower part of the nitride layer (WDS top, EDS bottom).

EDAX

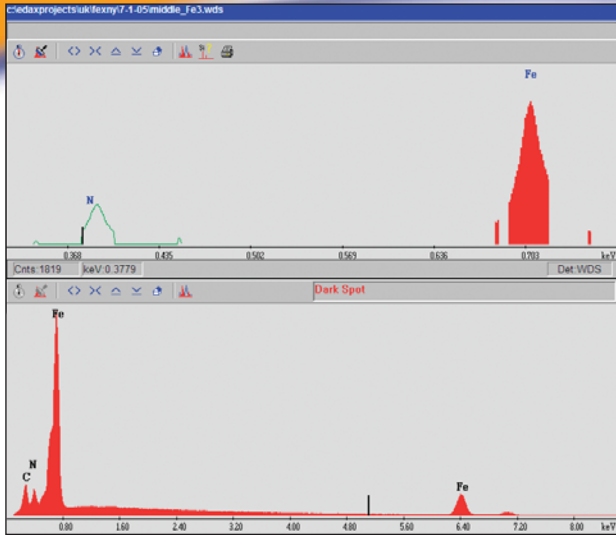

Figure 4: Spectra of the spot analysis, middle part of the nitride layer.

Using the boron nitride standard, the two spectra shown above were quantified; the results in Tables 1 and 2 show the lower nitrogen concentration in the darker part of the nitride layer and the higher concentration in the brighter part of the layer.

Element	Wt %	At %	K-Ratio	Z	A	F
N K	7.81	25.63	0.0502	1.205	0.5331	1.0014
FeL	90.32	74.37	0.8311	0.9799	0.939	1
Total	98.133	100				

Table 1: Lower spot analysis.

Element	Wt %	At %	K-Ratio	Z	A	F
N K	9.32	28.97	0.0609	1.201	0.5435	1.0014
FeL	91.14	71.03	0.8324	0.9767	0.9351	1
Total	100.467	100				

Table 2: Middle spot analysis.

To finalize the analysis of the nitride sample, a line scan was acquired across the various layers in the sample. The resultant line profile is shown in Figure 5, illustrating the various intensity levels for nitrogen across the layers.

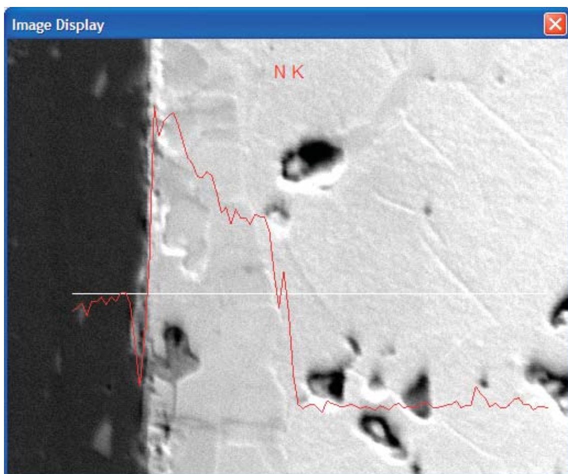

Figure 5: N K WDS line scan across the nitride layer.

Conclusion

The extremely high performance of the optics enables the LEXS to be ideally suited for light elements from Be to S. The system is capable of producing various data sets enabling the material characterization scientist to fully understand the composition and chemical trends across layers such as the nitride layer examined.

The LEXS' high performance for nitrogen is most useful for calculating concentration levels within the layers. An X-ray map of the whole area of interest and line scan profiles illustrate the nitride intensities across the various layers.

Spectrometer

The LambdaSpec is a uniquely designed compact parallel beam X-ray spectrometer (PBS), with high sensitivity and excellent resolution as two main design criteria. The spectrometer uses flat diffractors and has the added advantage of having a unique leak free detector and gas system. The compact design enables the LambdaSpec to fit on most electron microscopes (and in most cases in the EDS port, if necessary). The LambdaSpec provides improved quantification in terms of accuracy and detection limit and is the perfect complimentary tool for an EDS system. To improve ease of use the software is fully integrated and has been designed to enable the LambdaSpec to be operated as an EDS system. In fact, the LambdaSpec is a WDS system that thinks and acts like an EDS system.

LEXS

The LEXS has been optimized for low energy microanalysis by utilizing the unique high collection optics (HCO), which capture the highest count rates available, providing rapid X-ray analysis at the best resolutions obtainable. The LEXS is specifically designed for low energy X-ray microanalysis, providing high peak-to-background ratios and resolutions of less than 20eV for X-ray energies below 2.5keV. It is ideal for resolving such overlaps as Si Kα from W or Ta Mα and N Kα from Ti Lα, with count rates sufficiently high enough so that speed of analysis is not a constraint of the system.

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