



FIRST Newsletter

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S8 TIGER – Enhancing Quality Control of Tool Steels with High-Performing WDXRF

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Introduction

Tool steels are iron-based alloys that are particularly well-suited to be made into tools. Their suitability comes from their distinctive hardness, resistance to abrasion and deformation, and their ability to hold a cutting edge at elevated temperatures. As a result, tool steels are used in the shaping of other materials.

With a carbon content between 0.5% and 1.5%, tool steels are manufactured under carefully controlled conditions to produce the required quality. The presence of carbides in their matrix plays the dominant role in the qualities of tool steel. The four major alloying elements in tool steel that form carbides are tungsten, chromium, vanadium and molybdenum. The rate of dissolution of the different carbides into the austenite form of the iron determines the high temperature performance of steel. Typically, the manganese content is kept low to minimize the possibility of cracking during water quenching.

There are six groups of tool steels: water-hardening, cold-work, shock-resisting, high-speed, hot-work, and special purpose. The choice of group to select depends on cost, working temperature, required



surface hardness, strength, shock resistance, and toughness requirements. The more severe the service condition (higher temperature, abrasiveness, corrosiveness, loading), the higher the alloy content is. Therefore the amount of carbides required for the steel is greater. The elemental composition must be controlled tightly in order to achieve a maximum product quality, while not spending too much on expensive alloying metals.

Sequential wavelength dispersive X-Ray fluorescence spectroscopy (WDXRF) is an excellent method for testing these types of samples. Sample preparation can be very straight forward as the only requirements are that the sample can fit into the spectrometer and that the analytical surface is polished to a mirror finish. The high degree of accuracy and precision and the speed at which samples can be analyzed by WDXRF means that quality control can be done almost in “real-time,” thus ensuring that the quality of the product leaving the production line is of the highest possible quality. This lab report outlines the advantages of the **S8 TIGER** WDXRF spectrometer with its specific benefits for this analytical task.



Figure 1: S8 TIGER WDXRF spectrometer

Instrumentation

The fast and accurate analysis of tool steels has been performed on the WDXRF spectrometer S8 TIGER. With the direct loading setup in the S8 TIGER, accurate and always reproducible sample positioning in the beam path is ensured in contrast to other conventional spectrometers. The optimal excitation of all relevant elements is based on 4-kW X-ray excitation ranging from 20 kV for very light elements, 27 kV and 50 kV for the medium range, and 60 kV for heavy elements. The current ranges from 5 mA to 170 mA maximum. The switch between the different power levels and kV settings is seamless and instantaneous without compromising the analytical precision.

The most important advantage of WDXRF versus other technologies is its optimal spectral resolution. The S8 TIGER incorporates latest developments in analyzer crystals, which are added to the system configuration for the analysis of tool steels. With the curved germanium crystal, the resolution and intensity is enhanced for the elements P, S and Cl with an increased reflectivity and focusing of the radiation towards the detector. The basis for the excellent results, analyzing speed and analytical

precision for the alloying elements is the XS-400. It covers the same elemental range as the LiF200, but adds at least 35% intensity compared to the standard crystal. This saves more than 10% measurement time when analyzing tool steels with the same precision and detection limits. For optimal resolution, the LiF 220 crystal is used for the remaining elements.

Measurement Conditions

The measurement conditions chosen for this application are summarized in Table 1.

Table 1: Measurement Conditions

	Line	kV	mA	Filter	Crystal	Collimator
Si	K α	30	135	None	PET	0.46
S	K α	30	135	None	Ge (Curved)	0.23
P	K α	30	135	None	Ge (Curved)	0.46
Mn	K α	40	101	None	LiF220	0.23
Fe	K α	40	101	Al, 500 μ m	LiF220	0.12
Ni	K α	40	101	None	XS400	0.23
Cr	K α	40	101	Al, 12.5 μ m	LiF220	0.23
Mo	K α	50	81	Al, 800 μ m	LiF220	0.23
Co	K α	40	101	Al, 100 μ m	XS400	0.23
W	L β	40	101	None	LiF220	0.23
V	K α	40	101	None	LiF200	0.23

When choosing measurement conditions, care was given to ensure that the best signal to noise ratio was achieved, while still maintaining good overall count rates. This ensures that the application is both sensitive (in terms of limits of detection) and efficient (in terms of measuring times).

Figure 2 shows a scan of the Mn-K α line for a sample containing approximately 3 wt% Mn. The scan shown in blue uses the LiF(220) crystal with no primary beam filter, and achieves a count rate of approximately 110 kcps with a background intensity of approximately 2 kcps. This gives a peak to background ratio of 55:1.

The scan in orange shows the same sample, measured with the LiF(220) crystal but with an aluminum primary beam filter, 200 μ m thick. Here, the background has been reduced to around 500 cps, but the intensity for the peak has dropped to 34 kcps. The peak to background ratio here is 85:1.

The scan in red shows the same sample, measured this time with the XS400 crystal and an aluminum primary beam filter, 200 μm thick. Using this special structure crystal, the peak intensity observed is

approximately 125 kcps, but the background is still reduced (by the beam filter) to around 500 cps. This gives a peak to background ratio of 250:1.

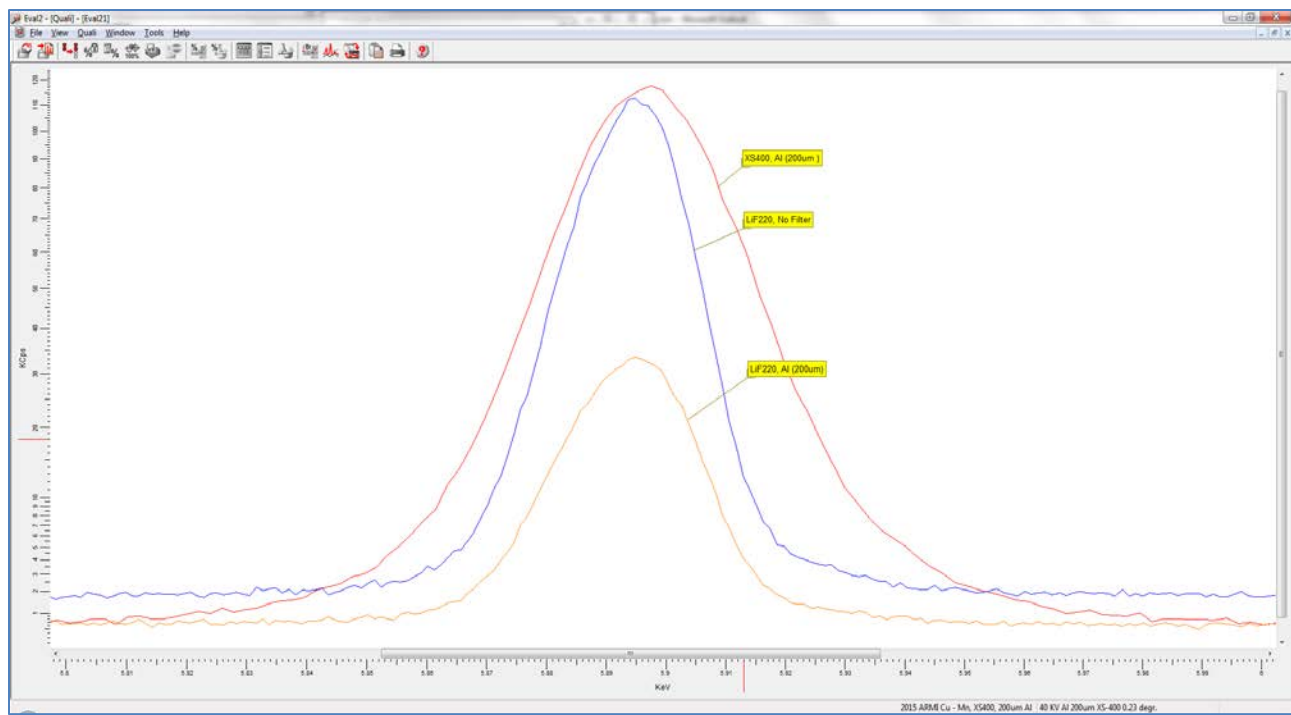


Figure 2: Sample containing 3 wt% Mn

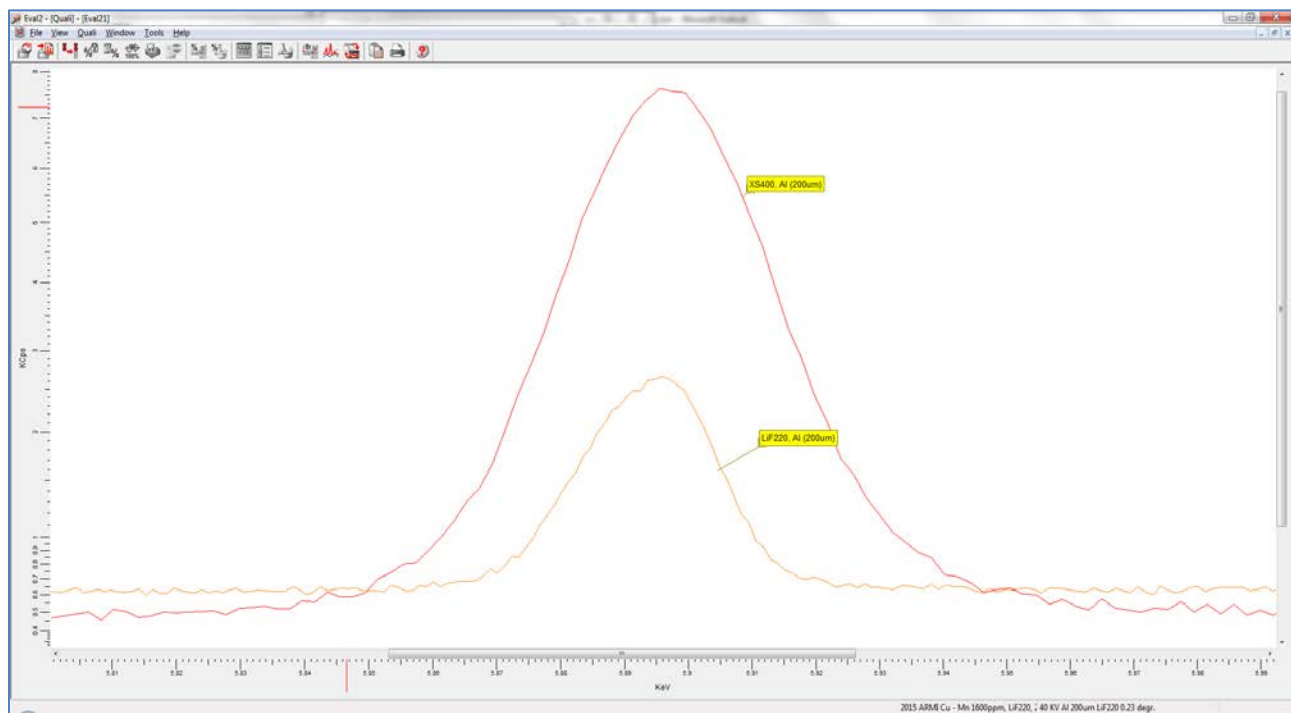


Figure 3: Sample containing 0.16 wt% Mn

For easily detectable concentrations of metals such as Mn, this sort of increase in intensity is not so critical, but at lower concentrations, as shown in Figure 3, this could be very important in ensuring accurate trace level determination.

In Figure 3, the scan using the LiF(220) crystal (orange) gives a peak to background ratio of 4:1, whereas the scan using the XS400 gives a peak to background ratio of 15:1.

With good count rates such as these, it is possible to shorten the measuring time of each sample (and thus increase the overall throughput of samples in the lab) while still maintaining good accuracy and precision. SPECTRA^{plus} is capable of managing of this for you. You can simply specify the level of counting statistical error and the maximum counting time, for each element, you deem as acceptable. If the statistical error in the count rate is minimized to the level you stipulate before the maximum counting time is reached, the spectrometer will automatically move on to the next element.

Results

Table 2 shows the standard deviation figures obtained for the calibration lines. It is not possible to measure Silicon accurately using this set of standards, as they were polished using a grinding machine with Silicon Carbide papers. So much of the signal is from residual Silicon on the surface of the samples, rather than from the actual sample itself. Phosphorus is also not well characterized in these standards, so the results are not reliable.

Table 2: Standard Deviations for Calibration Lines

	S	Mn	Ni	Cr	Mo	Co	W	V
St. Dev (wt%)	0.009	0.011	0.008	0.10	0.019	0.032	0.49	0.037

The calibration curves for V, Mo and Cr are shown in Figures 4 -6. The white boxes are showing the raw intensities for each reference sample. Based on the matrix correction using the fundamental parameter model and overlay correction the calibration fit is almost perfect.

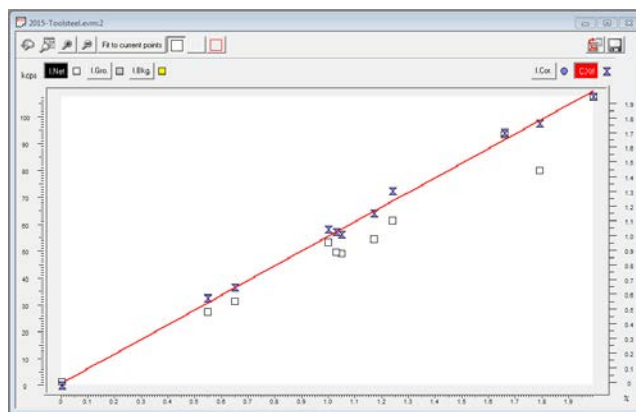


Figure 4: Regression line for V

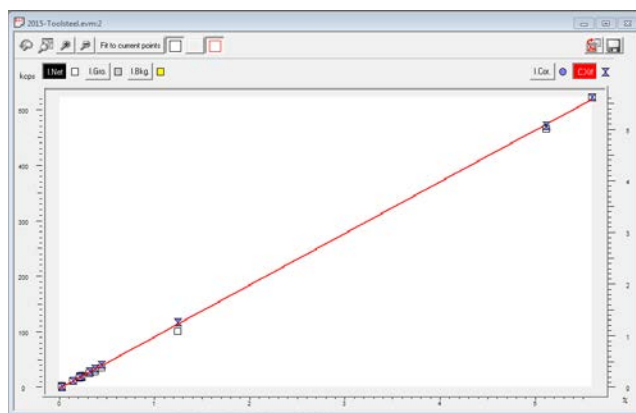


Figure 5: Regression line for Mo

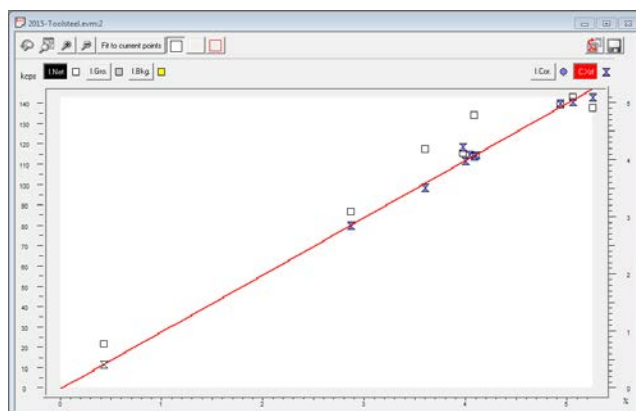


Figure 6: Regression line for Cr

A repetition test of 20 measurements of sample HS8 (certified concentrations are shown in Table 4) was performed to demonstrate the accuracy and precision of the instrument. For this repetition test, the sample was loaded and unloaded between measurements.

Table 3: Repetition Test Data (Intensities, kcps)

	S	Mn	Ni	Cr	Mo	Co	W	V
MIN	6.25	10.72	13.70	119.62	935.14	728.94	30.53	71.16
MAX	6.31	10.78	13.80	120.19	939.48	735.21	30.89	72.01
AVERAGE	6.28	10.75	13.75	119.91	936.59	732.47	30.67	71.55
ST.DEV	0.02	0.02	0.03	0.17	0.98	1.29	0.08	0.32
%RSD	0.31	0.20	0.18	0.14	0.10	0.24	0.27	0.44

Table 4: Repetition Test Data (Concentrations, wt%)

	S	Mn	Ni	Cr	Mo	Co	W	V
MIN	0.030	0.288	0.158	3.640	9.882	8.980	1.910	1.302
MAX	0.031	0.290	0.159	3.659	9.933	9.310	1.940	1.317
AVERAGE	0.031	0.289	0.158	3.649	9.899	9.030	1.922	1.309
ST.DEV	0.000	0.001	0.000	0.005	0.011	0.021	0.008	0.006
%RSD	0.839	0.263	0.259	0.149	0.116	0.230	0.423	0.432
CERTIFICATE	0.03	0.28	0.16	3.55	9.47	9.04	1.81	1.2
Abs. Diff	0.001	0.009	-0.002	0.099	0.429	-0.010	0.112	0.109

In total, 11 lines are measured in this method (see Table 1). The maximum measurement time for each element was set at 30 seconds and a counting statistical error optimization factor of 0.3% was specified for each element. The average measuring time across the repetition test was 5 minutes, so a time saving per sample of 30 seconds.

Summary

With the optimal system configuration the S8 TIGER provides a fast, accurate and precise analysis of tool steel samples for quick quality control. With the enhanced sensitivity for each element due to optimal instrument settings and the automatic time-optimizing measurement method, a high sample throughput and fast time-to-result is guaranteed.

By adding the TouchControl interface to the S8 TIGER, ease of use in night- and day-shift is guaranteed making the analysis of production test samples as easy as 1, 2, 3.

The S8 TIGER WDXRF spectrometer is making an intelligent move in order to achieve an optimum sample throughput, while maintaining the analytical precision: For each particular element a defined precision limit and maximum measurement time is set. When analyzing a sample, each element peak is measured automatically until the defined precision limit or the maximum measurement time is achieved. Therefor each element is only analyzed as long as needed, saving valuable measurement time for the next sample. This maximizes the sample throughput on the S8 TIGER substantially.