Oxford Instruments portable XRF analyser X-MET8000...

Application Note 13

...for the rapid, non-destructive analysis of electroless nickel coatings on steel

Electroless nickel plating is an auto-catalytic chemical reaction used to deposit a layer of nickel-phosphorus (NiP) alloy on a substrate. Unlike electroplating, the process does not require to pass an electric current through the solution to form a deposit. The coating thickness varies typically from 1 to 40 µm depending on the application. The phosphorus (P) content in the NiP alloy can range from 2 wt% (low phosphorus) up to 14 wt% (high phosphorus).

Electroless nickel plating provides an even layer regardless of sample shape. The resulting coating provides excellent corrosion and wear resistance, as well as hardness, improved solderability and brazability properties. Its biggest limitation is its relatively high cost (waste treatment cost due to regular chemical renewal). Electroless nickel coating is used in many industries such as automotive (e.g. gear assemblies and bearings), electronics (e.g. connectors and printed circuit boards), petrochemical (e.g. oil field valves), in the manufacturing of computers (hard disk drives), and many more. A consistent NiP coating thickness and composition guarantee the good functionality of the coated components. One common measurement technique involves assessment of the nickel layer by simply cross-sectioning the sample. This can lead to inaccurate results because of sample preparation variability from one technician to another, while introducing unnecessary material waste and labour.

The **X-MET**8000 **Expert** is a handheld X-ray fluorescence (XRF) analyser providing fast, accurate and non-destructive determination of NiP coating composition and thickness on steel. The combination of a high performance 50kV X-ray tube and Oxford Instruments' high resolution silicon-drift detector (SDD) provides faster analysis and lower limits of detection.

The **X-MET**8000's simple "point and shoot" operation and lightweight design make it the ideal tool for in-line analysis during the production and manufacturing of components, as well as for the inspection of incoming goods. With a battery life of up to 12 hours, the **X-MET**8000 can be



used all day on a single battery charge. Its light weight (1.5kg) makes it truly portable, so there is no need to take samples to the laboratory. The analysis is simply done by placing the **X-MET**'s "nose" directly against the sample, and pressing the trigger to start the analysis. Results are displayed on the **X-MET**'s large screen within a few seconds. Users can store up to 100,000 results, and create reports at the press of a button.



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Empirical Calibration

For best accuracy, an empirical calibration for the determination of P content and NiP coating thickness on steel was developed using six standard reference materials. These were flat, covered the measurement window, and were measured twice for 60 seconds, rotating them between measurements to get an average reading over their surface. A region of interest for phosphorus was used to derived a linear calibration for the determination of phosphorus content in NiP. The NiP coating thickness was determined by absorption method (absorption of Fe X-rays by the NiP coating). The calibration performance summary is presented in Table 1.

	P % NiP µm	
Calibration range	3.5 - 11.1	5.0 - 22.4
Standard error of calibration	0.5	1.2

Table 1: Calibration performance summary

Instrument Precision and Accuracy

In order to determine the instrument precision and accuracy for this application, two samples that were not included in the calibration were measured ten times. The repeats were carried out using 30 seconds measurement time. The data is presented in Table 2.

	Sample 1		Sample 2	
	P, %	NiP, µm	P, %	NiP, µm
Minimum	3.1	7.5	8.6	20.6
Maximum	3.3	8.5	9.5	20.7
Average	3.2	8.1	9.1	20.6
Given	3.4	8.6	8.7	21.3
Standard deviation	0.06	0.3	0.3	0.04

Table 2: Repeat measurements

Conclusion

The results for this application demonstrate that the **X-MET**8000 **Expert** provides repeatable and accurate results within seconds for the analysis of NiP coating on steel. The analyser enables the user to make rapid decisions in terms of quality control, as well as parts acceptance or reject during incoming inspection.

Ordering Information

X-MET8000 **Expert** with calibration software (P/No. 54-4106250). Customer to provide the calibration standards.



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