Background

Coating, or plating, is the deposition of a metal on an object's surface (usually also made of metal) to give this object specific properties: coatings can be decorative, prevent corrosion, improve solderability, increase hardness, reduce friction and wear, and more.

For example, silver (Ag) is commonly used in the electronics industry as a coating on copper components to increase their corrosion resistance, their operating temperature, and improves their high frequency transmission characteristics. Silver is also used for the decorative coating of cutlery, trophies and medals.

To ensure that the thickness of the coating applied is sufficient to give parts and components the desired properties, without wasting material by applying too thick a layer, plating companies need to control their process and the final products. The requirement for a rapid, simple analysis (carried out by non-laboratory staff) on site makes field-portable energy-dispersive X-ray fluorescence (EDXRF) spectrometry the ideal analytical technique.

Instrumentation

XRF is a widely used analytical technique for the determination of coating thickness. It provides reliable and rapid analysis (results are available in seconds), is a non-destructive technique (no need to scrap samples after analysis), and can be used by any operator with minimal training.

While benchtop XRF is commonly used to measure relatively small components, handheld XRF offers truly portable analysis, making it ideal for the testing of large and/or heavy parts.

The optimised combination of a high performance X-ray tube and Oxford Instruments' large area silicon drift detector (SDD) delivers the speed and performance needed for the control of coating thickness. With no requirement for a power source or bench space, the **X-MET**8000 delivers reliable results all day, every day. Its ruggedness (IP54 rated) and long battery life (up to 10-12 hours use on a single battery charge) make it the tool of choice for incoming goods or inventory inspection, and for process and quality control, even in harsh environments.





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X-MET8000 Series for rapid, on-the-spot coating thickness measurement

Application Note 11

Sample presentation and measurement

There is no sample preparation required. The user simply places the nose of the analyser on the part to be measured, and presses the trigger to start the analysis. Initial results are displayed on the analyser's large (4.3") integrated touchscreen within 2 seconds, and are updated until the end of the measurement. A typical analysis time for coating thickness measurement is 10-20 seconds.

For a representative thickness result over the surface of a large component, the operator can either setup the **X-MET'**s moving average function, where the average result is updated while the nose of the analyser is slid on the sample surface. Or the user can define how many analyses to use to calculate the average coating

thickness on the sample.

The **X-MET**8000's **integrated camera** can be used to accurately position the **X-MET**'s nose on the sample. The image can also be saved for future reference or inclusion in analysis reports.

For small samples measurements, Oxford Instruments also has the perfect solution:



Camera picture

- The light stand and safety shield fit in the analyser's compact transport case, for maximum flexibility and portability. It is ideal for the analysis of fasteners, other small pieces, and sample cups
- The benchtop stand allows to test larger parts with awkward shapes in total safely. Its use does not require mains power, and it can be folded to be stored away when needed.

Single or multiple elements plating solutions can also be measured. Once a calibration is derived, it is simply a question of pouring the solution into a sample cup fitted with a thin film (e.g. Mylar), and measure it using the light stand and safety shield.





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Performance and results

A simple empirical calibration was created for each application shown below, by measuring 2 samples with known coating thickness and 1 pure sample made of the coating material to establish the relationship between coating thickness and X-ray signal. Each sample was measured for 20 seconds.

The tables below show the accuracy and precision for a series of coatings applications carried out on the **X-MET**8000 using reference standards. The **X-MET**'s precision was derived from 5 repeat analyses of each sample.

Cr over Fe		
Known thickness	Average thickness	Precision (95% confidence)
5.3µm (210µin)	5.3µm (210µin)	0.02μm (0.8μin)
7.9µm (311µin)	7.8µm (307µin)	0.05μm (2.0μin)
12.4µm (487µin)	12.4µm (487µin)	0.07μm (2.8μin)

Ni over Fe		
Known thickness	Average thickness	Precision (95% confidence)
2.7μm (95μin)	2.6µm (102µin)	0.02μm (0.7μin)
10.3µm (407µin)	10.5µm (414µin)	0.10μm (3.9μin)
19.9µm (782µin)	19.9µm (783µin)	0.24µm (9.5µin)

Zn over Fe		
Known thickness	Average thickness	Precision (95% confidence)
3.0µm (120µin)	2.9µm (114µin)	0.02μm (0.6μin)
14.6µm (574µin)	14.9µm (588µin)	0.05μm (2.0μin)
19.2μm (756μin)	19.2µm (754µin)	0.13µm (5.0µin)

Ag over Cu		
Known thickness	Average thickness	Precision (95% confidence)
7.5µm (294µin)	7.4µm (290µin)	0.04μm (1.8μin)
12.1µm (477µin)	12.1µm (477µin)	0.10μm (3.8μin)
28.6μm (1,125μin)	28.9µm (1,138µin)	0.13μm (5.2μin)

Sn over Cu		
Known thickness	Average thickness	Precision (95% confidence)
4.8µm (189µin)	4.7µm (184µin)	0.07μm (2.6μin)
16.7µm (659µin)	16.6μm (655μin)	0.12µm (4.9µin)
27.3μm (1,075μin)	28.3µm (1,114µin)	0.32μm (12.7μin)

Note: the difference in μ in values for the same μ m value is due to additional decimal places being used for the calculation of averages and precision.

The maximum thickness that can be measured depends on the material and the analyser's components geometry (angles and distances between X-ray tube, sample surface and detector). Examples of maximum measurable thicknesses for the **X-MET**8000 Series are:

Coating	Substrate	Maximum coating thickness (μm)
Ag	Cu	50
Sn	Cu	50
Zn	Fe	35
Ni	Fe	20
Cr	Fe	15

Ordering Information

Minimum required:

- X-MET8000 Expert (software 2.1 or above). Includes
 the compact, waterproof, rugged carrying case, a wrist
 strap and lanyard, 2 batteries, a battery charger, a USB
 cable to connect to a PC/laptop, 5 replacements windows,
 and the user manuals.
- Empirical calibration software
- Known samples (provided by user)

Optional:

- Light stand and safety shield, for the analysis of small samples. Included in some packages
- Benchtop stand, for the safe measurement of awkward shapes
- Window-shield, for maximum protection of the analyser against small and sharp samples
- Sample cups and thin film, for the analysis of solutions

Summary

Once calibrated (a procedure that can be carried out by the user in minutes), Oxford Instruments' **X-MET**8000 provides accurate and repeatable coating thickness analysis for a wide variety of applications. The **X-MET**'s ease of use and ruggedness make it an ideal tool on the shop floor for the incoming inspection of parts or components, as well as for process and quality control.

The versatility of the calibration software also enables the analysis of plating solutions (single and multi-elements), ensuring the rapid monitoring of the plating baths composition.

With results being available on the **X-MET**'s large integrated screen in seconds, decisions to accept/reject a part or modify the plating process can be made on the spot, maximising productivity and savings costs.



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