APPLICATION NOTE

Measuring Metal Coating Thickness at Line

Using the Thermo Scientific Niton XL5 Plus XRF Analyzer

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Introduction

Metal coatings are applied on all kinds of items made of metals, alloys and plastics either for decorative purposes or to optimize the physical and chemical properties of their surface (corrosion, wear and heat resistance, hardness, electrical conductivity, adhesion, solderability or lubricity). Over coating can significantly increase the cost of manufacturing, but under coating can cause dramatic product failure. That is why controlling the coat weight or the coating thickness is essential in metal finishing, fabrication, automotive and aerospace industries.

Among available technologies for measurement of metal coat weight, X-ray fluorescence (XRF) spectrometry has numerous advantages: it is non-destructive and element specific, and it delivers very accurate results.[1] Nevertheless, for certain elements XRF cannot measure metal layers that are too thick or purely organic coatings. For many years, this was addressed by bringing the specimen to be analyzed inside or close to the analyzer chamber so it could be analyzed by benchtop XRF. Measuring coating thickness on large and heavy parts was impractical without cutting samples. This limitation can be overcome using handheld XRF.

The Thermo Scientific™ Niton™ XL5 Plus, the smallest, lightest and most powerful tube-based handheld XRF analyzer, accurately measures the coat weight or coating thickness of metals in seconds. The non-destructive analysis is done directly at-line for process control or on-site for finished products. Operators can analyze large

The Niton XL5 Plus Handheld XRF Analyzer

and heavy parts directly, increasing productivity and improving efficiency. Having to cut samples and bring them to the lab belongs to the past.

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Instrument Features & Analytical Method Set Up

The Niton XL5 Plus is an advanced handheld energy dispersive XRF analyzer with:

- A proprietary miniaturized 5W (5-50kv/5-500µA) Ag-anode x-ray tube
- A large silicon drift detector with graphene window for enhanced light element detection
- ProGuard detector protection to prevent window puncture
- A macro camera to take sample image for better record keeping
- A micro camera to visualize and document the analyzed area
- A standard spot size of 8 mm to analyze large areas and compensate for micro heterogeneities
- An optional 3 mm small spot collimator to analyze small sized samples.

Using a proprietary fundamental parameter (FP) based algorithm, the Niton XL5 Plus analyzer is versatile and handles an infinite number of situations without having to calibrate the analyzer using reference standards.[1,2] The Niton XL5 Plus analyzer measures the coat weight or coating thickness of up to 4 layers over one substrate. The substrate can be either defined as metal, alloy, plastic or wood while the layers can be defined as pure element, alloy or compound (via pseudo element).

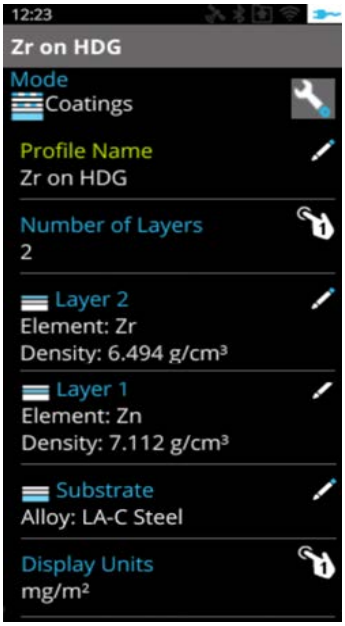

Figure 1: excerpt of a user-defined profile (analytical method)

The analytical method development for coatings analysis with the Niton XL5 Plus analyzer is simple and intuitive. As seen in figure 1, the user selects the number of layers and enters the composition of layers and substrate using a list of elements or available alloy libraries according to AISI/ASTM, DIN or GB standards. This analytical method is then stored in a profile and the instrument is ready to be used. The Niton XL5 Plus analyzer allows operators to configure, store and call as many profiles as needed.

Capabilities and examples of application of the Niton XL5 Plus Coatings Mode

Table 1 illustrates capabilities of the Niton XL5 Plus Coatings Mode with coated materials used for various purposes in different industries and applications. The list of examples is not exhaustive - any combination of metal coating can be analyzed using the Niton XL5 Plus analyzer - provided the composition of the coating layer(s) and the substrate are not identical and provided the thickness of the coating layers does not exceed a critical value, called infinite thickness.[1,2,3] The infinite thickness is reached when the signal from the substrate or a layer is totally absorbed by an intermediate layer or the layer itself.

Results of analysis from three real-world example are shown in Figure 2. All three results were obtained out of the box, without any empirical adjustments based on coating reference samples.

The first example is the analysis of a samples made of copper used in electrification products. The product is coated with silver to increase the electrical conductivity at high frequency. This is a simple coating system of a mono-elemental layer over a pure metal. Figure 2a shows analysis results obtained with the Niton XL5 Plus analyzer of 12.88 µm vs. the 13 µm measured in a laboratory, which constitutes excellent accuracy.

The second example is the analysis of a zirconium conversion coating over hot-dip galvanized steel used in the automotive industry, among others. It is a two-layered coating system consisting of a relatively thick layer of zinc that protects steel from rusting and a second layer of zirconium to prevent so-called white rusting of zinc; by avoiding the formation of zinc hydroxide, the layer containing zirconium ensures a better adhesion of paint layers. The layer of zirconium is very thin, not exceeding the equivalent of several atomic layers. The measured value for zirconium

Measurement Capability	Coating Example	Purpose or Improved Property	Industries, Application
Single mono-elemental layer over pure metal* or a non-metal substrate	Ag / Cu Zn / [Steel] Cd / [Steel] Cu / [Plastic]	Electrical conductivity Corrosion resistance Corrosion resistance Metal coating primer	Electrification products Various metal fabrication Aerospace, Military Various metal fabrication
Multiple mono-elemental layers over a pure metal* or a non-metal substrate	Au / Ni / Cu/ [Zn] Cr/ Ni / Cu / [Plastic] Ti / Zn / [Steel] Zr / Zn / [Steel]	Decorative Decorative Corrosion resistance Corrosion resistance	Accessories and apparel Fabrication, fixtures Fabrication, automotive Fabrication, automotive
Single mono-elemental layer over alloys	Cr / [Stainless steel] Rh / [White gold] Rh / [Brass] Ti / [Al alloy] Zr / [Al alloy] Cr (VI) / [Alloys**]	Wear and corrosion resistance Decorative Decorative Corrosion resistance, paint primer Corrosion resistance, paint primer Corrosion resistance, paint primer	Various metal fabrication Jewelry Accessories Fabrication, Automotive Fabrication, Automotive Various metal fabrication
Multiple layers over alloys	Cr /Ni/ [Brass]	Decorative	
Alloys as coatings over pure metals	NiP / [Steel] NiP / [Al] ZnNi / [Steel]	Wear and corrosion resistance Wear and corrosion resistance Corrosion resistance	General fabrication General fabrication Automotive, Aerospace
Alloys as coatings over alloys	NiP / [Kovar]	Wear and corrosion resistance	Glass sealing
Compound layers using tracer elements	P_2O_5 / [Steel] TiN / [Tool steel]	Corrosion, paint primer Hardness, wear resistance	Fabrication, Automotive Cutting tools

Table 1: Measurement capability examples and applications of Coatings Mode on the Niton XL5 Plus analyzer
*In many cases for electroplated items, low alloy steel can be approximated as pure iron **Al, steel, Cu, Zn alloys

(40.24 mg/m^2) is very close to the lab value (38 mg/m^2) whereas the measured value for zinc is also close to the expected value.

The third example is an electroless nickel coating over Kovar alloy. Kovar alloy is used in industry when a reliable glass-to-metal seal is needed. The alloy contains about 29% nickel, 17% cobalt, a few other elements at levels below 1%, and a balance of iron. Electroless a nickel is a nickel-phosphorous alloy (NIP) that is chemically applied on a substrate to increase wear and corrosion resistance of the surface. This type of sample is relatively complex to analyze because it is an alloy coated over another alloy as substrate with both alloys containing high concentrations of nickel. Also, in this case, the analytical results (18.58 µm) are very close to the certified values (20.0 µm).

From the simplest to the most complex coating samples, the Niton XL5 Plus Coatings Mode is exceptionally versatile and delivers accurate results "out of the box" without any adjustment. When desired, accuracy of the measurement can be further improved using reference samples (standards or in-house characterized reference materials) in a type standardization process.

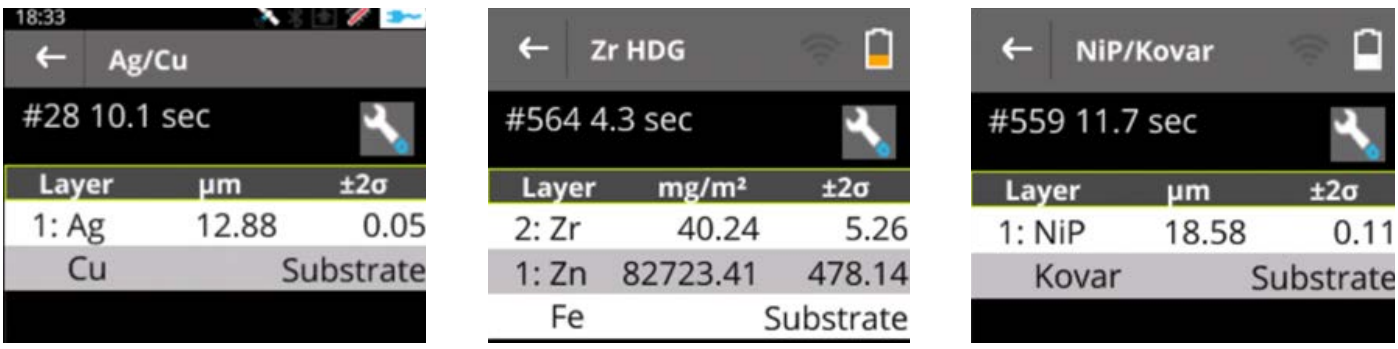

Figure 2: Examples of measurements obtained "out of the box" using Niton XL5 Plus Coatings Mode

a) Silver over Copper.
Lab value for Ag: 13 µm

b) Zirconium over galvanized steel.
Lab value for Zr: 38 mg/m^2.
Expected Zn value: ca. 80 g/m^2

c) Electroless Ni coating (NiP) over Kovar™ alloy. Certified thickness of NiP: 20 µm

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Conclusion

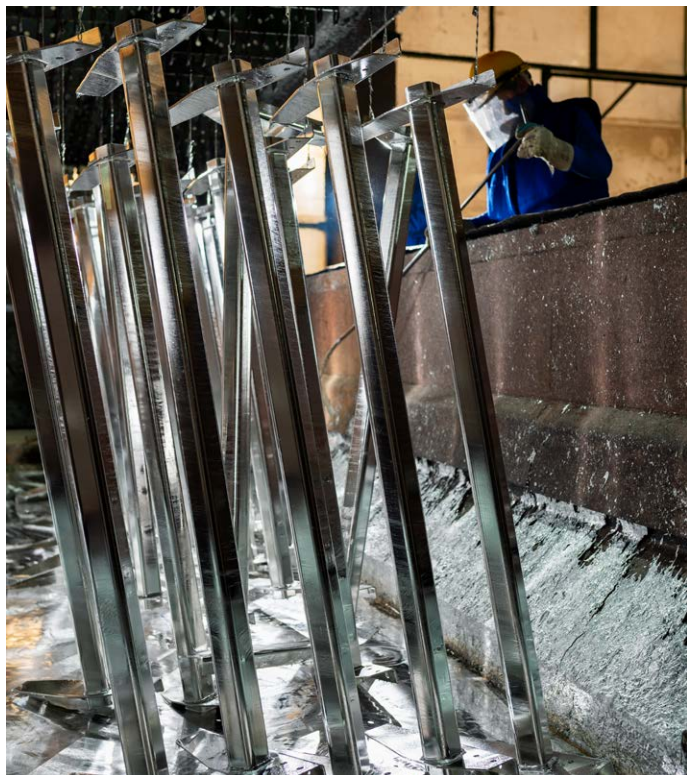
To ensure customer coating specifications are met, quality control at-line, online or during final product inspection is a necessity. The Niton XL5 Plus analyzer assists the operator to:

- Ensure goods received match purchase orders by measuring metal grade and composition
- Reduce production costs by minimizing production errors
  - Coatings that are too thin can result in poor corrosion resistance, high warranty costs and/or product failure
  - Coatings that are too thick give money away by using more product than is necessary
  - Non-destructive analysis means there is no need to cut or damage high-value product
- Improve quality by ensuring consistent coating across a product via multiple measurements and automatic averaging
- Provide faster turnaround with immediate results and no sample preparation (versus statistical sampling followed by lab analysis which is time consuming)
- Generate quality reports and certificates through simple report generation tools
- Create audit trails of product from goods incoming inspection to products out the door
- Achieve peace of mind with compliance to international methods ISO 3497 and ASTM B568

The Niton XL5 Plus Coatings Mode provides a significant step forward from the traditional process of metal identification and composition measurement.

References

1. S. Piorek, *Coatings, Paint and Thin Film Deposits*, Chapter 4 in *Portable X-ray Fluorescence Spectrometry Capabilities for In Situ Analysis*, Edited by Philip Potts and Margaret West, RSC Publishing 2008
2. ASTM B568-98 (2009) *Standard Test Method for Measurement of Coating Thickness by X-Ray Spectrometry*.
3. ISO 3497-2000-*Measurements of coating thickness – X-ray spectrometric methods*.



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