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XRF

Non-destructive Thickness and Composition Analysis of Coatings on Steel Using MESA-50

Application Note

Metallurgy XRF22

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Abstract: Steel coating plays an important role in preventing corrosion of steel samples. Therefore, an understanding of coatings can help in improving the steel properties. XRF is a powerful technique for measuring thickness and composition of coatings. We carried out non-destructive thickness and composition analysis of Zinc-Nickel coating on steel using the MESA-50 X-ray fluorescence analyzer.

Keywords: Steel, coatings, thickness, composition, macro-XRF, EDXRF

Introduction

The steel industry has contributed significantly to the economy, due to its extensive use in automotive and construction work. Despite being very stable, corrosion contributes to premature aging and deterioration of steel. Therefore, steel surfaces are generally coated, for example, by Zn-Ni plating to protect them from corrosion[1]. The composition and thickness of coatings is crucial for anti-corrosion and the adhesion properties[2]. Therefore, it is very important to measure thickness and composition of the coating precisely. SEM cross-section observation and GD-OES are used to measure thickness of the coatings[3,4]. However, they are based on destructive approaches. On the other hand, XRF spectrometer provides a non-destructive way for calculation of coating thickness as well as its composition, with minimal sample preparation thanks to the higher penetration of X-rays. In this application note, we carried out thickness and composition analysis using the HORIBA MESA-50 X-ray fluorescence analyzer.

MESA-50 X-ray fluorescence analyzer

The MESA-50 fluorescence analyzer (Figure 1a) is an energy-dispersive X-ray fluorescence spectrometer with a variable collimator size (1.2 mm, 3 mm, or 7 mm) for a suitable surface analysis. It irradiates primary X-rays from the bottom and detects fluorescent X-rays from a sample on the diagonal position (Figure 1b). Various peaks in the XRF spectrum correspond to the spectroscopic emission of the elements present in the sample, while the intensity is correlated to the elemental composition in the volume of the interaction of the incident beam. For this reason, there is no direct information on the thicknesses, however, the intensity of the peaks in the spectra will be a function of thickness.

The MESA-50 software is equipped with an optional function called Multilayer FPM. It is a powerful, robust and fast function specialized for coating analysis based on a fundamental parameter method (FPM). It can calculate coating thickness and composition for up to four layers (including bulk) without any need of standard samples.

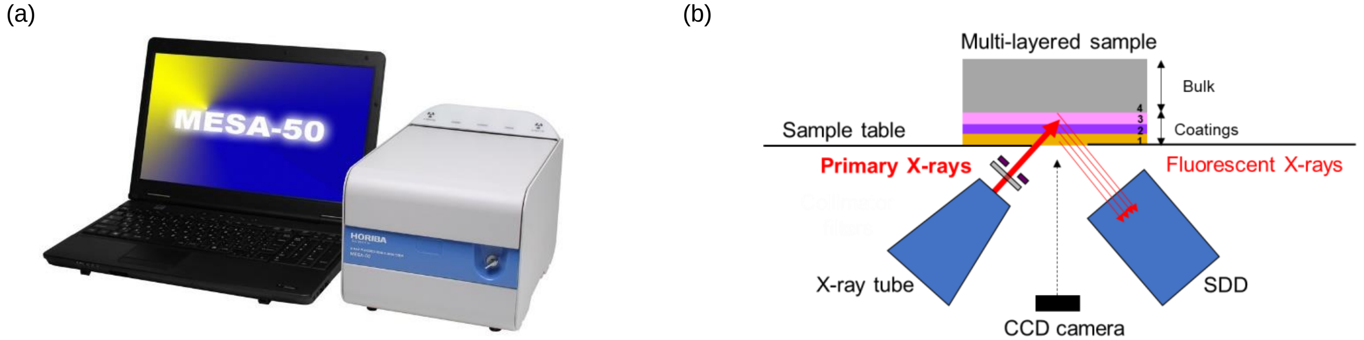

Figure 1. (a) HORIBA MESA-50 X-ray Fluorescence Analyzer (b) Schematic presentation (side view) of coating analysis by MESA-50.

Coatings ranging in thickness from tens of nanometers to tens of micrometers can be measured, though it depends on the sample matrix. In addition, the MESA-50 is light-weight and has a small footprint, and an internal battery backup. Therefore, it can easily be carried to various locations for sample analysis.

Sample information and result

In this application note, we prepared a single coating of zinc and nickel on a steel plate (Figure 2a). The slide was mounted directly on the MESA-50, as there was no need for specific sample preparation (Figure 2b). After setting the measurement position on the optical image, we carried out point analysis on the steel plate to get the X-ray fluorescence spectrum using a 1.2 micron collimator size (Figure 2c). The voltage of the X-ray tube was adjusted to 50 kV. The software auto-calculated the appropriate current value as 13 µA. The measurement time was set 60 sec. The experiments were repeated three times to check the repeatability.

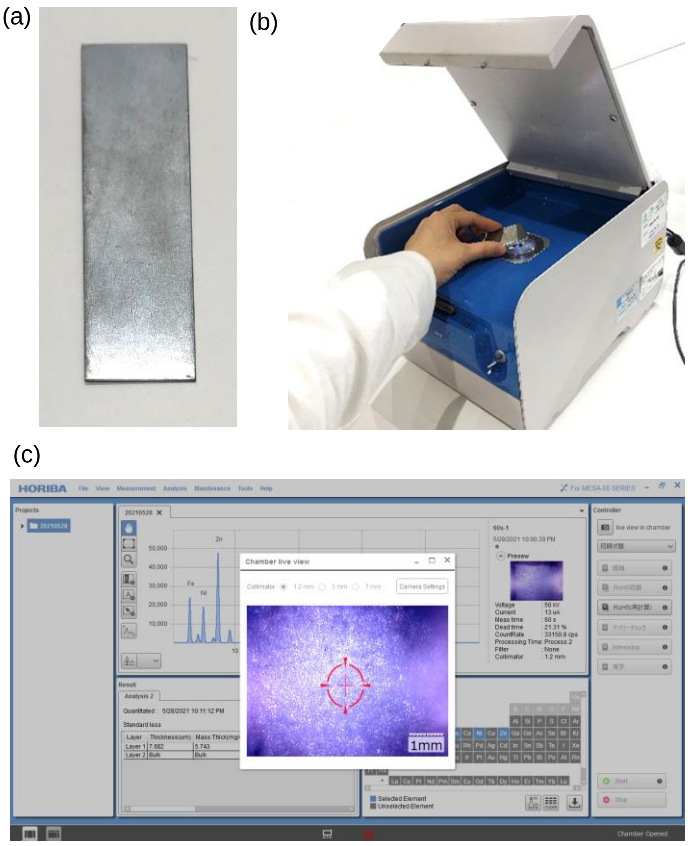

Figure 2. (a) A steel plate with coating of Zn-Ni analyzed in this application note, (b) Sample is mounted on the MESA-50, (c) Chamber live view for setting measurement position.

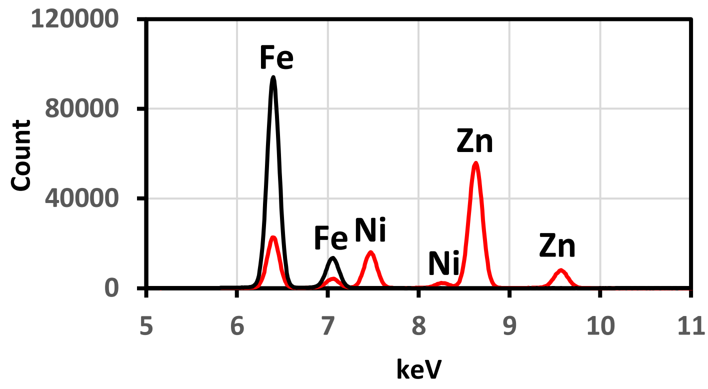

Figure 3. XRF spectrum of a steel plate without coating (black) and a steel plate with Zi-Ni coating (red).

First, we carried out spectrum analysis as qualitative analysis. To compare with bare steel plate, Figure 3 (a) shows the layered XRF spectrum of the steel sample with Zn-Ni coating, and the bare steel plate without the coating. Compared with the bare plate, the spectrum of the Zn-Ni coated sample presents clear peaks of Zn and Ni, which are the components of the coating, in addition to Fe, which is the main component of the steel. Next, we calculated thickness and composition of the Zn-Ni coating of the sample using the Multilayer FPM function of the MESA-50 software. Coating thickness and composition can be calculated using the obtained spectrum and user-defined layer model. The layer model can be defined using layer structure and its elemental information. In this measurement, we defined the layer model as can be seen in Figure 4:

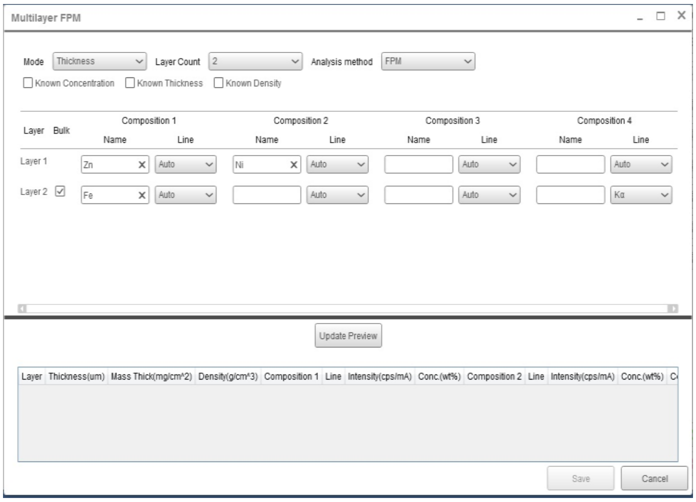

Figure 4. Layer model creation for the Zn-Ni coated steel analyzed in this application note using Multilayer FPM function.

After calculation, we obtained the results of coating thickness and composition of the Zn-Ni layer. Table 1 shows the results (*n*=3): the Zn-Ni coated sample had 9.00 µm of Zn-Ni coating whose composition was found to be 85.59% of Zn and 14.41% of Ni.

Table 1: Thickness and composition of the Zn-Ni coating calculated using Multilayer FPM.

Ex. No.	Thickness (µm)	Composition (mass%)	
		Zn	Ni
1	9.08	85.60	14.40
2	8.99	85.55	14.55
3	8.92	85.61	14.39
Average	**9.00**	**85.59**	**14.41**
SD	**0.08**	**0.03**	**0.03**

Conclusion

The MESA-50 provides a fast and non-destructive analysis of thickness and composition of Zn-Ni coating on steel samples. We could detect a thickness of 9.00 µm non-destructively with good repeatability. Composition of the coating was calculated using FPM and was found to be 85.59% of Zn and 14.41% of Ni. The MESA-50 is a powerful system for analyzing these types of coatings.

References

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