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APPLICATION NOTE 30267

Oxidized low alloy steel
500 nm

1650 SM
Mixed Layer 30 nm

13 RM 19
Passivation Layer 2 nm

Al oxide on low alloy steel
AlOx 500 nm

Full survey chemical analysis of thin films with pulsed fast flow GD-MS

Authors

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Keywords

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Introduction

High resolution glow discharge mass spectrometry is broadly recognized as one of the most sensitive and robust analytical techniques for direct bulk trace element determinations. However, due to the nature of the magnetic field, scan speed is traditionally considered to be a limiting factor for depth specific measurements and thus unsuitable for thin film or layer analysis of conductive and semi-conductive samples.

The fast flow Thermo Scientific™ Element™ GD Plus GD-MS equipped with a microsecond-pulsed power supply feeding its DC ion source, provides the means to overcome these limitations. In pulsed mode, the source of the Element GD Plus GD-MS is running at much lower nominal power conditions (typically 3–4 W) as compared to standard continuous DC modes, but does not compromise the inherent high instrumental sensitivity. The combination of significantly reduced sputter rates with very high sensitivities, provides the analytical prerequisites for performing full survey analyses even at nanometer scale depth resolution.

In pulsed mode it is possible to achieve a depth resolution of <10 nm whilst still achieving trace to ultra-trace level detection power. For the analysis of major components in layered systems, the GD power can be further decreased to give even greater depth resolution at a lower overall sensitivity.

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Layer analysis on low alloy steel

Quantitative multi-Element measurements with high sensitivity

A well-characterized low-alloy steel sample with a 500 nm thick oxidized layer has been measured using the new pulsed source on the Element GD Plus GD-MS (Figure 2). A comparison of the obtained results with those from the international round-robin studies related to ISO 25138:2010 GD-OES Test Method developments clearly reveals that the µs-FF-GD-MS results are in excellent agreement with the GD-OES test results (Figure 3). In addition, the µs-FF-GD-MS generates several orders more sensitive readings compared to the optical emission techniques. Figure 4 shows that even features at the low ppm range can be distinguished in the depth profile.

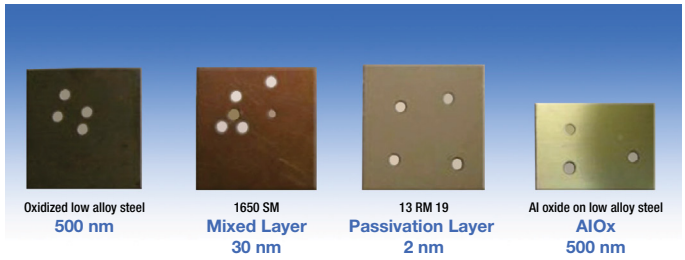


Figure 1. Iron based substrates with a variety of layer chemical compositions and thicknesses.

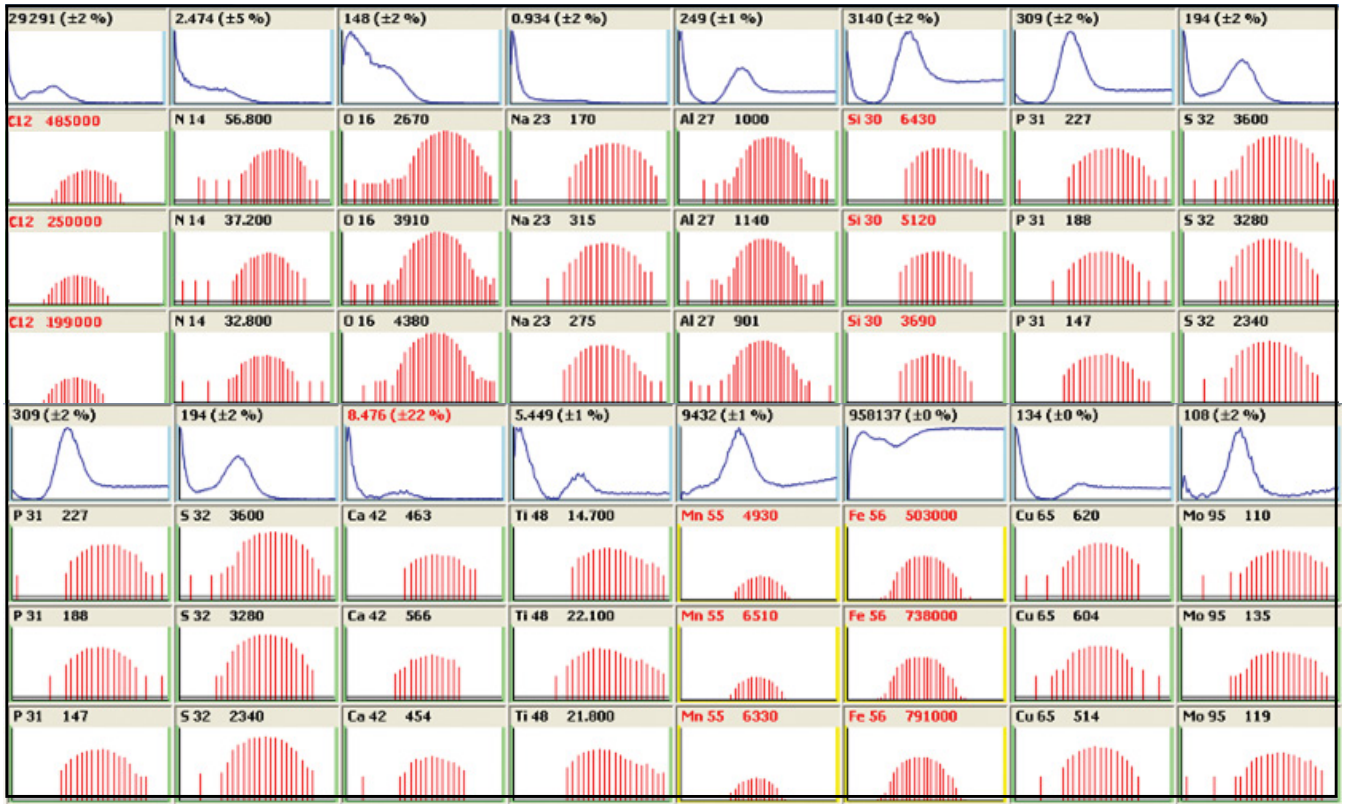


Figure 2. Multi-Element determination on oxidized low-alloy steel sample; 100 points for 14 Elements; data frequency around 10 nm. Profiles in rows 1 and 5 correspond to those shown exemplarily in Fig. 3. At the spectra, numbers indicate ppm concentrations at arbitrarily selected measurement times.

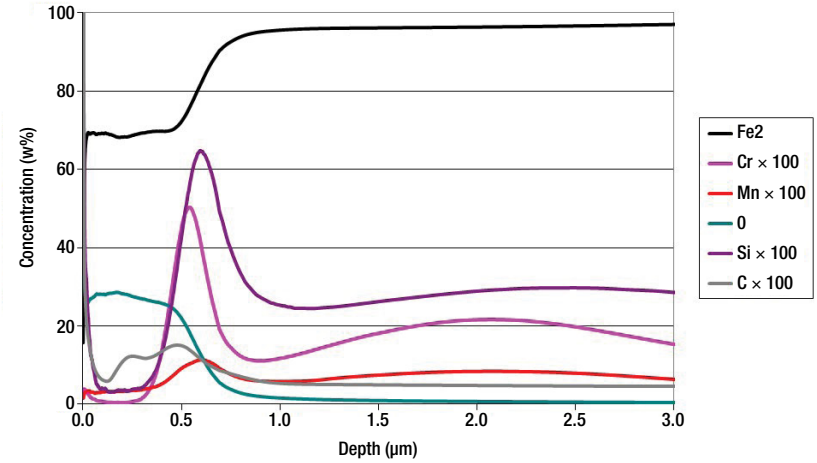
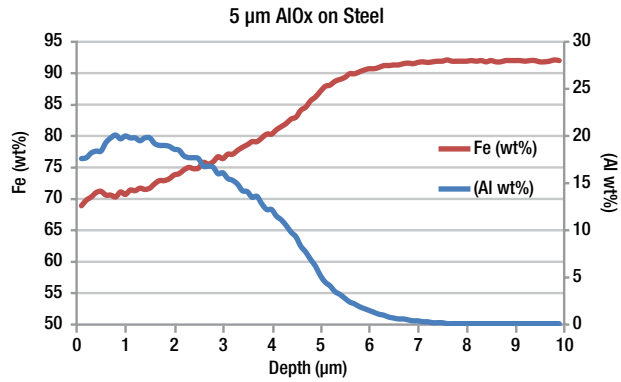


Figure 3. Typical GD-OES depth profiling results of oxidized layer on low-alloy steel (Results from ISO 25138:2010 International Test Method development).



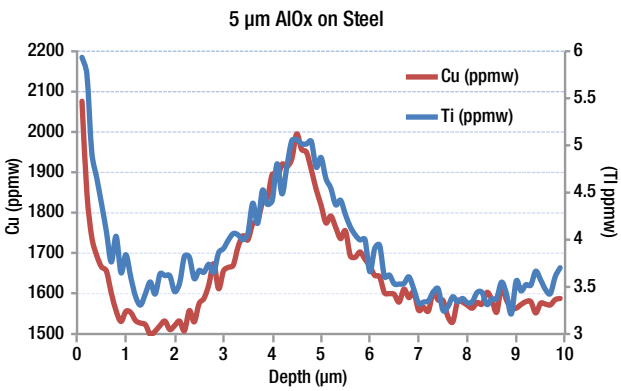


Figure 4. µs-FF-GD-MS results illustrating the distribution of Cu and Ti at the ppm level in the interface regions between the alumina coating on high-alloy steel. Alumina coating on HA steel: 5 µm; 50 points for 16 Elements; data frequency ~ 100 nm.

Robustness

The robustness of the µs-FF-GD-MS has been studied and shows that:

A. The technique is generally applicable to rough surfaces. Figure 5 implies that the atomization occurs layer by layer, since the initial roughness is maintained throughout the GD-MS run.

B. Pulsed and continuous modes can be combined for profiling thick engineering coatings, such as Zn plated steel (Figure 6).

C. The smooth transition of the profiles in Figure 6 also indicates that calibration factors are not affected significantly if the source is running in pulsed or continuous mode.

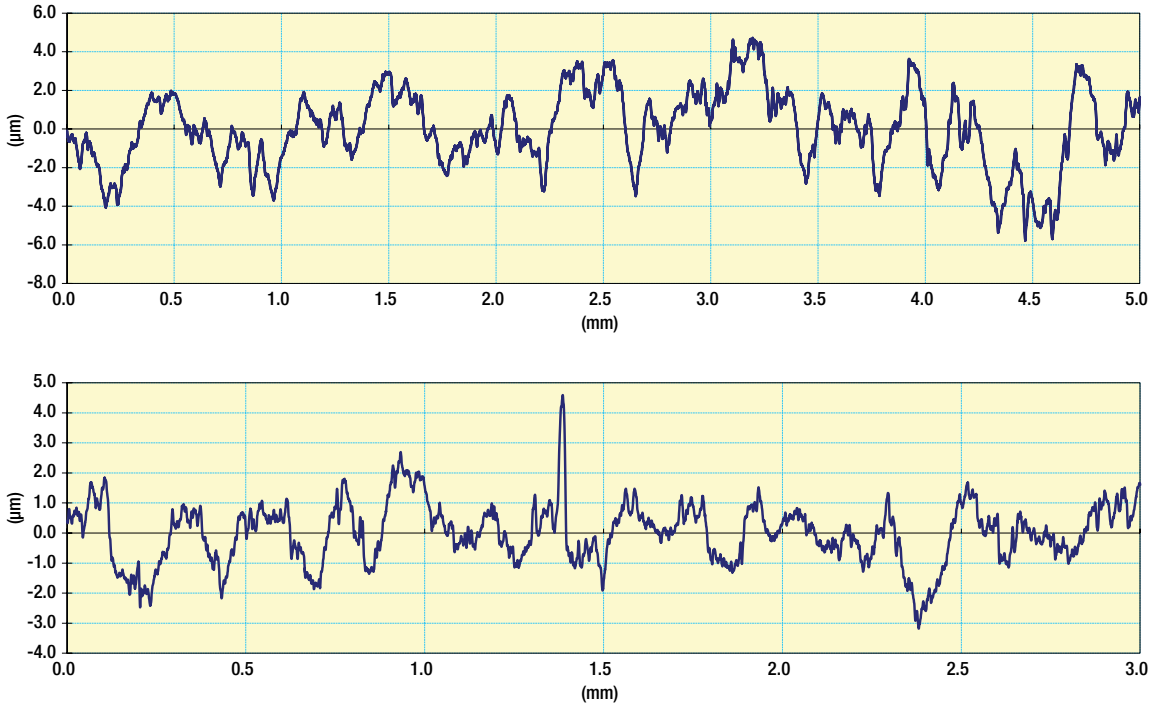


Figure 5. Roughness of sample surface (top figure) and of the bottom of a 4 mm diameter sputter crater (lower figure). Note that crater side walls are not shown.

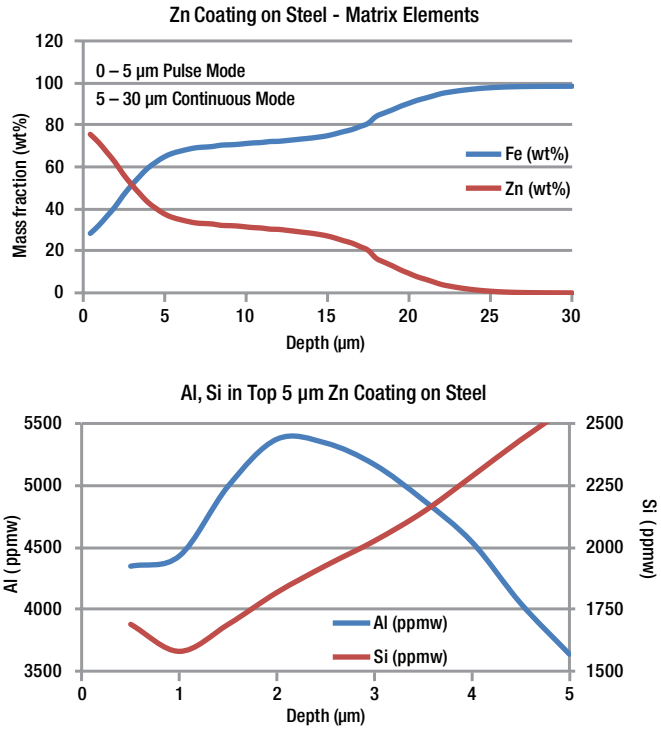


Figure 6. Combination of Pulse mode with continuous mode for extended depth profiling. For matrix elements the distribution from top to 30 µm depth is shown; for minor elements only the pulsed mode profile of the top 5 µm layer is shown.

Conclusion

- µs-FF-GD-MS is the most sensitive analytical technique today for determining the full chemical composition and trace Element distributions in engineering coatings or thin films.
- The simple adjustment of the source components results in easy user handling.
- The µs-FF-GD-MS results are in excellent agreement with GD-OES test results, which is still the most common analytical technique today for chemical analyses of coatings or thin films.
- The application field of this new approach is exceptionally broad and will likely impact many new research areas.

Acknowledgements

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Find out more at **www.thermofisher.com/GD-MS**

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