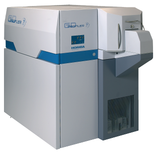
Synergy of GD-OES & DiP with EDXRF techniques for layers thickness and elemental composition determination



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Motivation

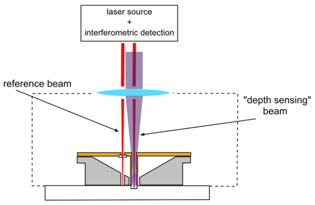
The modern manufacturing world is increasingly focusing its effort on a coating-based cost reduction.

In order to achieve a fruitful optimization, it is of great importance to determine the thickness/composition of the different coatings. Moreover, it is crucial to have access to reliable techniques providing such information within just a single measurement.

Within the HORIBA Scientific catalogue, there are two different techniques satisfying this requirement: Glow Discharge Optical Emission Spectrometry, coupled with the Differential Interferometry Profiling and the X-Ray Fluorescence using both the Fundamental Parameter Method or calibration curves method.

GDOES & DiP

The GDOES analysis relies on the **sputtering of a representative area of the material of interest and the excitation of the sputtered species**, by a dense plasma, operated in pulsed RF mode. Now, with to the addition of the **Differential Interferometry Profiler** (DiP), based on the measurement of the phase shift ($\Delta\varphi$) between two laser beams reflected inside the GD crater and at the surface, it is possible to directly obtain the crater depth (d) as a function of time

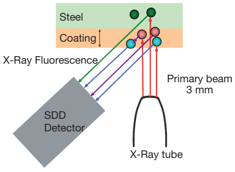


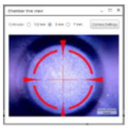
$$d = \frac{\lambda}{4\pi} \cdot \Delta\varphi$$

Conversion factor: 50.5 nm/rad

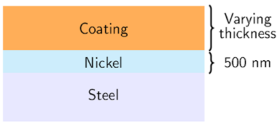
Energy Dispersive X-Ray Fluorescence

EDXRF relies on a X-ray source to excite all the elements in the sample, and on an energy dispersive detector for the simultaneous collection of the emitted fluorescence radiation. Once the XRF spectrum is acquired, the composition of a bulk sample could be easily obtained. Furthermore, a suitable simulation software allows to determine the composition and the thickness of multilayers samples . The advantage of this technique is the fact that it's contactless and non destructive.





The analysed samples



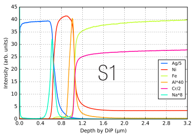
Reference samples for thickness measurements were bought from **KOCOUR.**

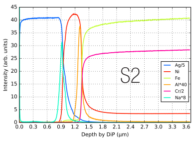
Samples are made of a **Ag coating**, with a varying thickness, deposited on a **Ni coating on steel.**

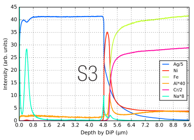
Ag	S1	0.66 µm
	S2	0.96 µm
	S2	4.94 µm

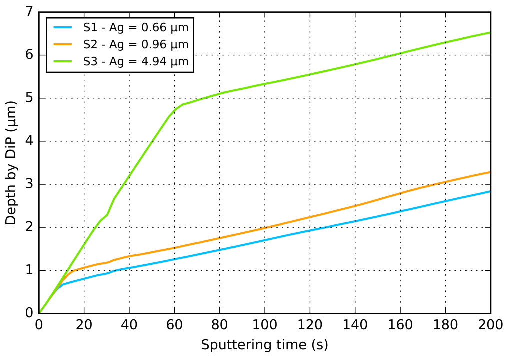
Analysis by GDOES & DiP

	Sample	Ag	Ni	Crater depth by DiP	Crater depth by Profilometer
Ag	S1	0.6 µm	0.4 µm	3.2 µm	3.5 µm
	S2	0.9 µm		3.7 µm	3.7 µm
	S3	4.8 µm		9.7 µm	10 µm







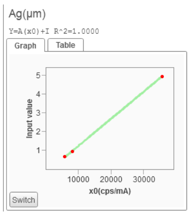


Analysis by EDXRF

	Sample	Coating weight	Density	Linear thickness
Ag	S1	0.71 mg/cm^2	10.5 g/cm^3	0.68 µm
	S2	0.99 mg/cm^2		0.94 µm
	S3	4.82 mg/cm^2		4.59 µm

The **FPM simulation software** calculates the **coating weight** (mass thickness). Then, by using the layer density it is possible to obtain to the linear thickness.

Moreover, **calibration curves** can also be used if known thicknesses are available on a sub-set of selected samples. For this, the GDOES+DiP is an ideal technique to obtain these values



Conclusion

The GDOES+DiP and the MESA-50 are two performant instruments that allow the determination of layer thicknesses. While GDOES+DiP is a destructive technique that requires no standards for the thickness determination, the EDX is a non contact and non destructive method.

However EDX has to rely on some known information about the sample structure, such as the layers distribution and their density. The evident synergy of these two techniques appears clearly for thicknesses determination.