HORIBA Scientific

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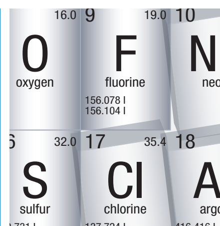
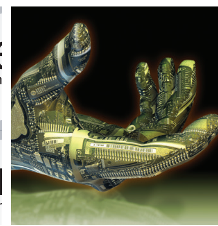
GDOES

Measurement of halogen elements by RF GDOES





Application Note

Material Science GD23

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Abstract

RF GDOES offers Ultra Fast Elemental Depth Profile Analysis of thin and thick films. All elements can be measured, notably the halogens Cl, Br, I and F of interest in multiple applications.

Key words

Cl, Br, F, I. GD plasma. Depth Profiles.

Introduction

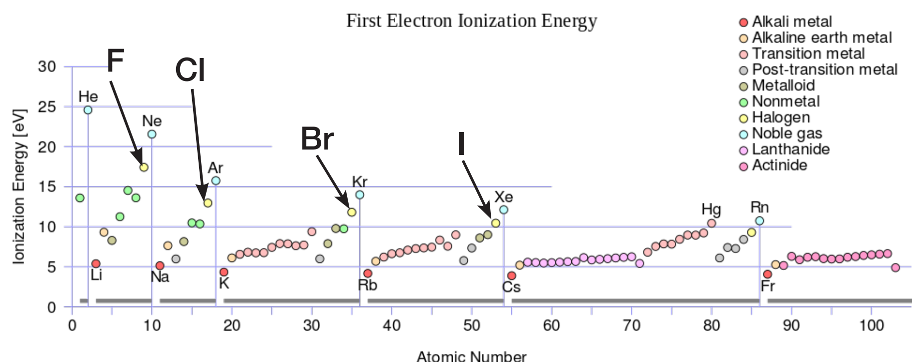
Halogen elements are present in multiple polymers and are of interest for many surface studies (corrosion etc). They were not classically measured by GD but plasma studies on gas changes, the introduction of pulsed RF sources and the invention of the UFS (HORIBA Jobin Yvon patent for fast sputtering of polymers) are opening new domains of applications for RF GDOES.

Instrumentation

The most sensitive lines for the halogens are in the UV/VUV range for Cl, Br and I (Cl 134 nm, Br 148 nm, I 145 nm or I 183 nm) and in the IR region for F (F 685 nm). The instrument should therefore offer high sensitivity from the VUV to the IR. This is achieved in the HORIBA Scientific GD instruments by the use of a dedicated optics with 2 gratings covering the entire spectral range.

Plasma gas

If Argon can be readily used as plasma gas for the analysis of Cl, Br and I, the measurement of F requires using Neon instead of Argon as plasma gas. Ne plasma is less efficient for sputtering but the excitation energy is higher which is needed to excite F.



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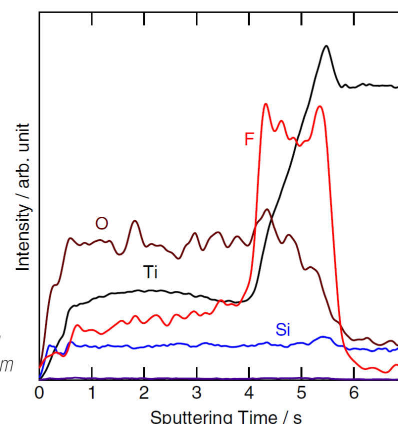
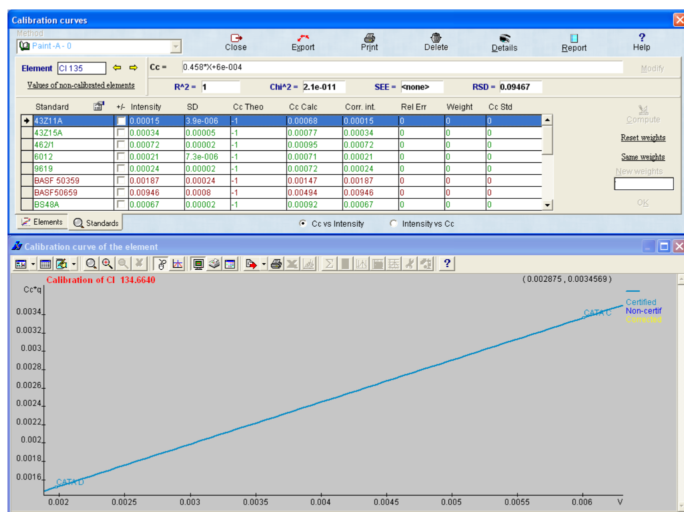


GDOES depth profiles of the anaode film formed to 50 V at a constant current density of 50 A m^{-2} in 0.1 mol.dm^{-1} ammonium fluoride electrolyte at 293 K (from reference 1)

Calibration of Cl in cataphoresis layers for automotive

Calibration can be done with our layer mode if at least one sample is known. It can also be done in a standard approach as in the example below. The 2 samples selected were polymers with various Cl contents. The sputtering rate mode was selected.



Halogens, pulsed RF source and UFS (Ultra Fast Sputtering)

Pulsed RF is often mandatory for fragile materials such as polymeric layers or Li batteries (See our application note GD 18).

However pulsing the source might reduce the erosion rate and consequently the sensitivity of the measurements (if less material enters the plasma per unit of time, less light is obtained). The application of our patented UFS device (patent N° EP2434275A1) enhances drastically the erosion rate of such layers, it is therefore possible to combine fast erosion and low induced damages for high sensitivity. The UFS can be used with Ar and Ne plasmas.

References

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3) "Amount of iodine responsible for I-SCC of Zircaloy-4 in PCI-conditions: recoil-implanted and thermally released iodine .M Fregonese & al. Nuclear Engineering and Design 186 (1998) 307–322

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