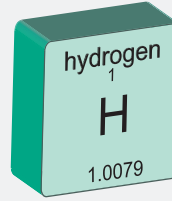


H & D

GDOES

Measurement of Hydrogen (and Deuterium) by RF GDOES



Application Note
Material Science
GD22

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Abstract

RF GDOES is able to measure and follow Hydrogen profile in depth which is of great interest for multiple applications. Deuterium (H isotope) can also be measured.

Key words

RF GD OES, Hydrogen, Deuterium, VUV, plasma cleaning.

Introduction

RF GD OES is well known for ultra fast elemental depth profile of thin and thick films. All elements can be measured including Hydrogen (H) which is important in many application fields - for corrosion studies (ref 1), for PV (ref 2), in metallurgy (ref 3), for the development of hydrogen storage materials (ref 4) and for all polymeric coatings studies (ref 5) to name a few.

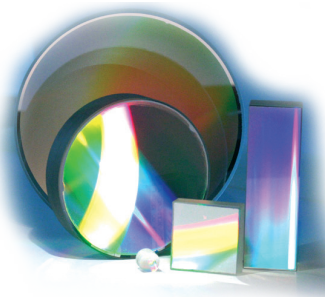
The most sensitive emission line for H is in the VUV range at 121,567 nm.

Isotopic separation usually requires MS spectrometry. However for Hydrogen, it is possible to separate it from its isotope Deuterium (D) in an optical spectrometer with adequate resolution: the 2 emission lines being at 121,534 nm (for D) and 121,567 nm (for H) – so separated by 30 picometers!

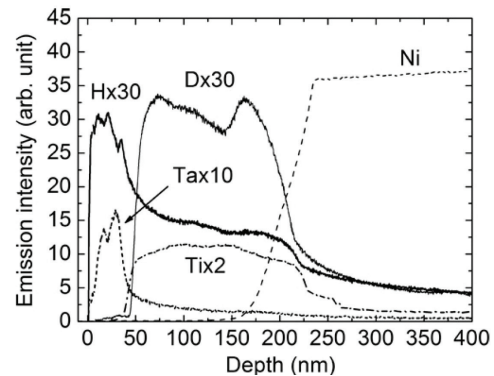
Deuterium profile is of great interest in fusion studies for plasma facing components (ref 6), it is also of interest in corrosion studies (D being substituted to H in the corrosion medium (ref 7).

Instrumentation

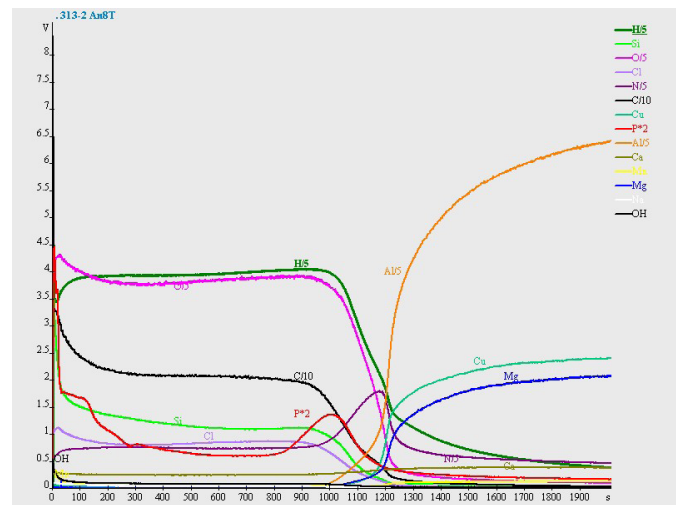
Measuring H (and D) at its most sensitive wavelength (121nm) requires an optical spectrometer very efficient in the VUV range. This is made possible by the use of a dedicated HORIBA Jobin Yvon grating.



The grating is also efficient in 2 orders of diffraction. It is therefore possible to measure simultaneously H & D with 2 HDD detectors.



Depth profiles of H, D, Ta, Ti and Ni in the Ta(H)Ti(D)/Ni layered structure (from ref 6)



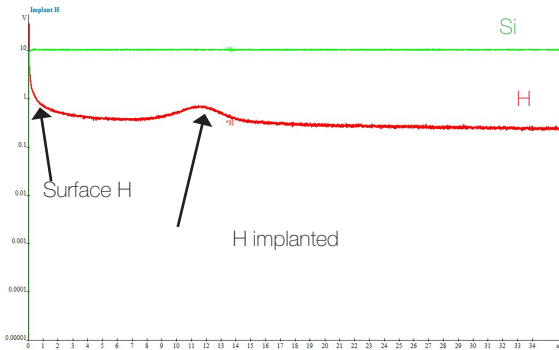
Sol gel polymeric coating on Al

Plasma Cleaning

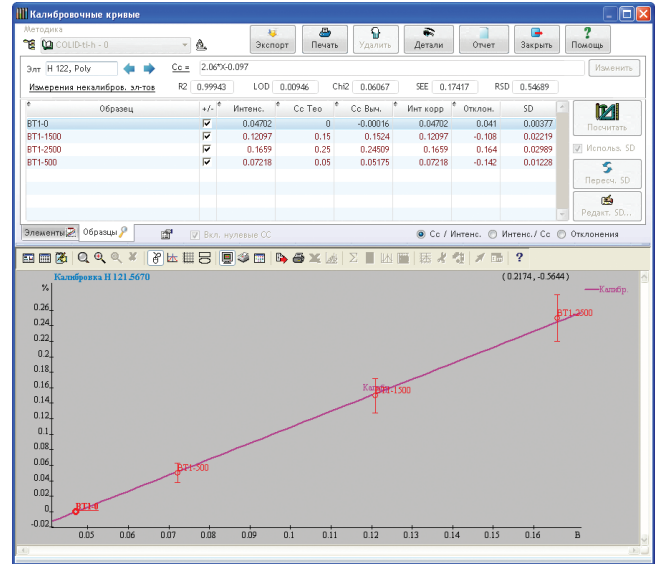
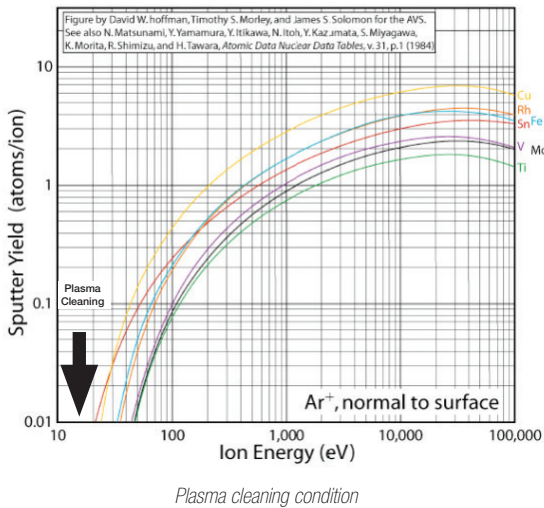
In GD operation the sample is not inserted in a vacuum chamber. Surface contamination is therefore always present.

This is a reason why a D profile is of interest in corrosion studies. If D replaces H in the corrosion medium, the profile of D has no ambiguity where H could result from corrosion or surface contamination.

The following example shows the depth profile of a Si sample with H implanted (log scale).



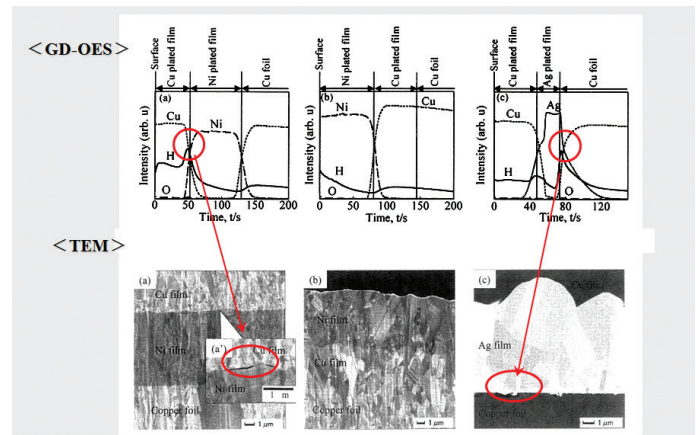
Surface H can however be minimized using the plasma cleaning operation (ref 8). This is a standard feature in our instrument – first introduced in 2009. The idea is, prior to analysis, to expose the sample to a plasma with very low energy (inducing no sputtering – typically less than 3W in pulsed operation mode) but active enough to induce the desorption of the surface of the sample.



N. Pushilina at the 7th GD day did show a calibration of H in Ti samples obtained after adequate optimisation of the plasma cleaning.

H at interfaces

If H at surface could come from surface contamination, a peak of H at an interface could always be related to the presence of a residual layer or to a void in the sample. The following examples correlate GD profiles with H peaks to TEM cross sections showing voids



H detection. Presence of Voids.

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