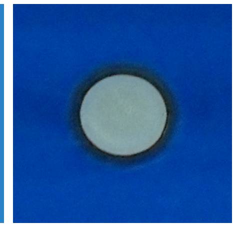
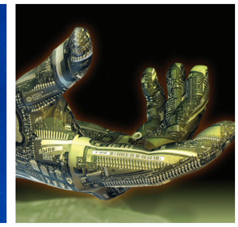
HORIBA Scientific

JOBIN YVON Technology

GDOES

Depth Profile analysis of organic and organic/inorganic multilayered materials by pulsed RF GDOES





Application Note

Material Science GD28

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Abstract
Some applications of pulsed RF GDOES for the analysis of organic and organic/inorganic films are described highlighting the potentialities of the technique in this domain.

Key words
GDOES, pulsed RF, polymers, depth profile, “UFS”.

Introduction

GDOES relies on the fast sputtering of a representative area of the material of interest by a dense plasma and the real time optical analysis of the emission light produced by the sputtered species excited by the same plasma.
With the development of pulsed RF source and the invention of the “UFS” (Ref 1 and Application Note AN GD21), the technique now contributes to the development and characterization of polymeric materials (as shown in references 2 to 6). Additives can be seen and localized, the structure of the material can be revealed quickly and therefore the elaboration process can be controlled and optimized.
Figure 1 illustrates a GDOES depth profile on a multilayered polymer. The layers are distinguished by representative elements. No molecular information (as given for instances by Raman Spectroscopy) is obtained with GDOES but the observation of representative elements and/or changes in the intensity levels for the same elements (that relate to changes in sputter rate from material to material) anyhow clearly reveals in many cases the structure of the multilayered compound.

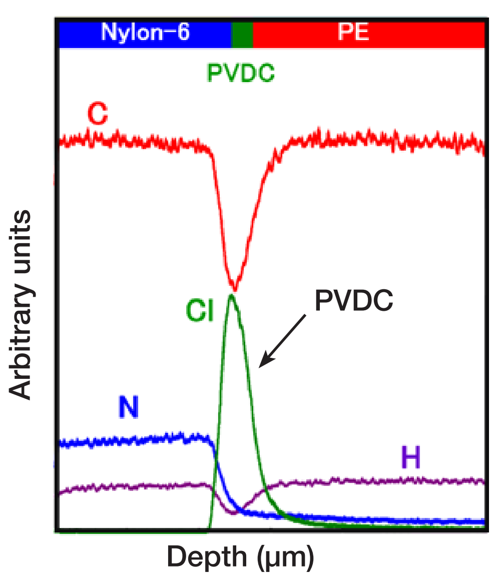

Figure 1. TRC presentation at the 2014 japanese GD day

Multilayered organic/inorganic materials

Rather than opposing RF GDOES to other techniques (SIMS, XPS, Raman) one should look at their complementarities.

The GD sputtering is extremely fast, crater is large and erosion results from bombardment by low energy particles (Ref 7, chap 16 & 17) allowing further investigations to be conducted on embedded layers easily exposed by GD. For instances, with the advent of Gas Cluster Ion Beams (GCIB), SIMS and XPS can now sputter polymeric films and provide some information on compounds but the GCIB are not efficient on metallic layers. Coupling techniques could therefore be of great interest in multiple applications.

The example below shows the GD result of an organic tandem solar cell. The structure of the sample (from the surface) is Au/Top cell/ZnO/Bottom cell/ZnO/ITO. Operating conditions for this analysis require the use of Pulsed RF and patented “UFS” – which consists in using a mixed gas ArO as plasma gas.

The total analysis is 4 minutes only, making the tool ideal for comparing samples and optimizing processes. One could note that GD could measure H and other light elements. The real time display of the elements of interest during the measurement also allows stopping the discharge at any depth.

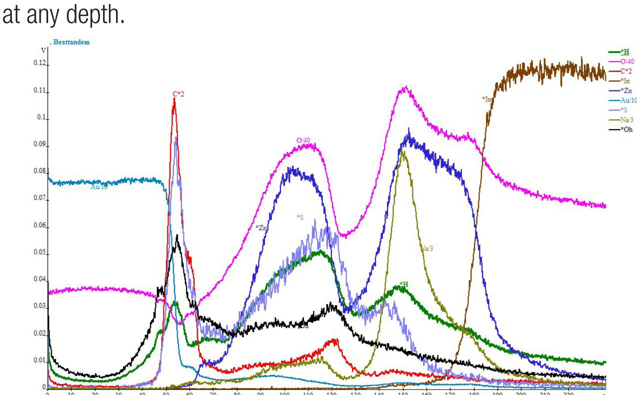

Figure 2. GD measurement of a Tandem organic solar cell (courtesy of Jinwoo Choi, PICM)

Sample handling

Polymeric samples are often thin and flexible. In order to be able to analyze them properly with GDOES they should be glued on rigid substrate. The photo below shows a PET film with a multilayer coating on top (multi InOx/Ag). The PET film has been glued on an Al plate using a proprietary methodology and obtained GD craters are nicely visible. The depth profile is given for reference. Here the analysis was done without UFS but in pulsed RF mode.

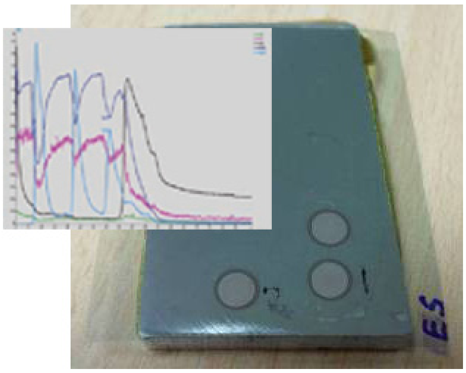

Figure 3. Coated PET film glued on a rigid substrate and analyzed by GD (first presented GD day 2010)

"UFS" for access to embedded layers

UFS means Ultra Fast sputtering. This is a patented system from HORIBA. Through a gas change (addition of O2 to the plasma gas), ultra fast sputtering of some polymers is obtained as a chemical effect combines with the sputtering.

This notably allows to easily access to embedded layers. An illustration is given in the following example. The sample is a chromated Al plate with 2 different varnish layers on top that react well to UFS. The top layers are sputtered in a minute with UFS.

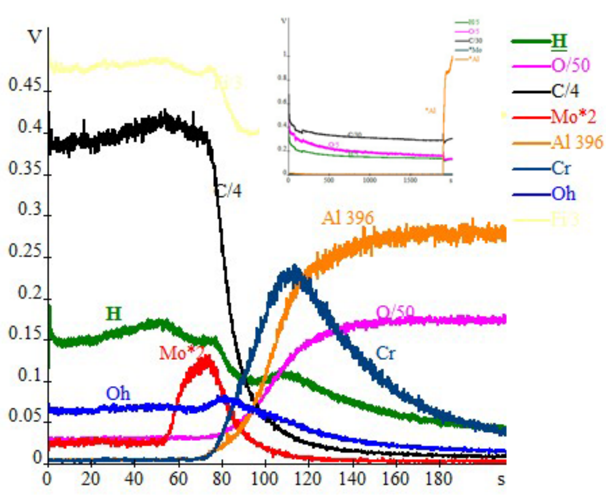

Figure 4. Analysis with UFS (and without UFS – insert)

Without UFS (insert), the analysis is quasi impossible: soft conditions must be used in order not to burn the layer affecting the sensitivity (as few atoms are sputtered and excited per unit of time), analysis time becomes very long (over 2 hours) and crater shape is poor.

Conclusion

Examples of application of pulsed RF GDOES to the analysis of polymer layers and organic/inorganic films have been show illustrating the possibilities of the technique. Thanks to the use of pulsed RF source and UFS, GDOES takes its role in the multidimensional characterization of advanced multilayered organic materials where its specific strong points (speed, ease of use, large observation zone, measurement of all elements) are of great interest.

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