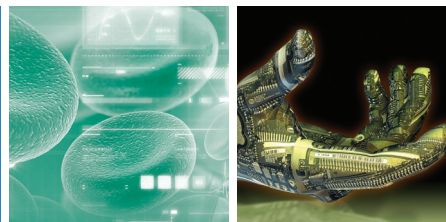


Pulsed RF GDOES for the analysis of films containing metal and metal oxide nanoparticles



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Abstract

This note shows the benefits of using pulsed RF GDOES for the analysis of films containing nanoparticles.

Key words

Pulsed RF GDOES, films, nanoparticles, depth profile distribution

Introduction

Metal and Metal oxides nanoparticles are introduced in films to enhance or add properties – corrosion resistance, photonics effects, antibacterial activity etc.

Metal oxide nanoparticles are widely used for the fabrication of composites due to their availability. Nanoparticles like ZnO, Al₂O₃, ZrO₂, TiO₂, CeO₂, etc., incorporated in various metals and alloys have been used to generate composite layers with superior properties. Similarly, polymer films with embedded metal nanoparticles can be fabricated for instances by melting a powder mixture (Co alloy in polyethylene) or by drying in a powder resin mixture (Ag particles in epoxy resin).

HORIBA Scientific is partner of a French research project that, in the same chamber, couples a spray of nanoparticles onto PVD growing layers.

Pulsed RF GDOES is used in many researches on these new materials to study the depth profile distribution of the particles and the thickness of the composite layer (Ref 1-5).

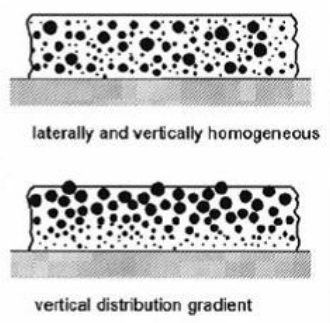


Figure 1. Domain of application of pulsed RF GDOES (from Ref 6)

HORIBA also proposes several complementary techniques in this field such as Particle Size Instruments (ref 7) and Spectroscopic Ellipsometry to monitor the optical response of various media with photonics particles embedded (ref 8).

Instrumentation

The GD Profiler 2 couples an advanced Pulsed RF Glow Discharge Source to a high resolution, wide spectral range Optical Emission Spectrometer. The source permits a precise and fast sputtering of a representative part of the material investigated (typically 4mm in diameter). The depth resolution is material dependant but could be at the nanometer scale. Pulsed RF operation is crucial for applications on composite materials as it avoids unwanted diffusion of the species in the layers during the measurement. For polymeric materials, the UFS operation mode is mandatory (cf GD application notes AN21 & AN28).

Sputtering issues could happen when particles aggregate or if they are of micrometric size rather than nanometric: cf Ref 9. GD sputtering could in such cases be still extremely useful to reveal the particles for SEM observation (Ref 10).



Figure 2. GD Profiler 2

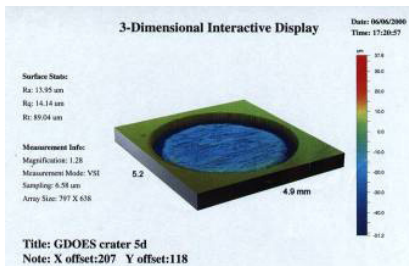


Figure 3. Typical GD crater (4mm diameter)

Typical measurements

The result shown here is taken from Ref. 1. The addition of CeO₂ nanoparticles to Zn material lead to finer grained deposits with better corrosion resistant properties. GD reveals here the uneven distribution of the nanoparticles.

The Ultra Fast Response Time of GD (such measurement is done in about 4 minutes) permits to use the technique to optimize and control deposition parameters.

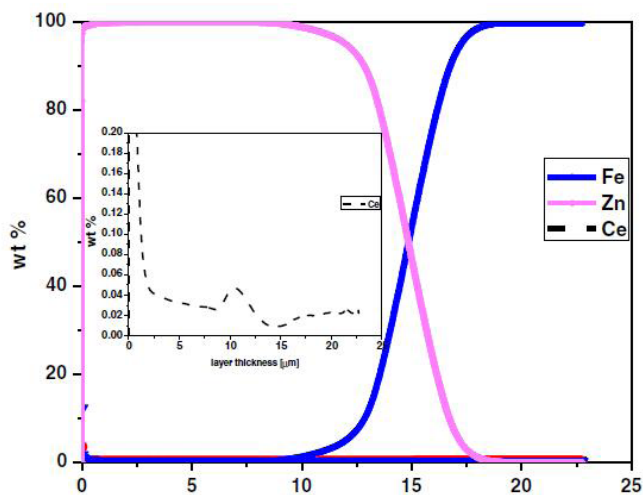


Figure 4. Pulsed RF GDOES depth profile (insert: the Ce distribution in the layer)

Conclusion

Metal and Metal oxide nanoparticles are added to thin and thick films to enhance or add properties. Pulsed RF GDOES is used to study the depth profile distribution of these particles averaged over a representative area and allows optimizing the deposition process to reach the required specifications.

References

- 1) "Influence of the electrodeposition current regime on the corrosion resistance of Zn-CeO₂ nanocomposite coatings". P. Nemes, M. Lekka, L.Fedrizzi, L. M.Muresan. Surface & Coatings Technology 252 (2014) 102–107
- 2) "The effect of SiO₂ nanoparticles dispersion on physico-chemical proprieties of modified Ni-W nanocomposite coatings". W. Sassi L. Dhoubi P. Berc, M. Rezrazi. doi:10.1016/j.apsusc.2014.10.142
- 3) "Synthesis Mechanisms of Organized Gold Nanoparticles: Influence of Annealing Temperature and Atmosphere. M. Bechelany, X. Maeder, J.Riesterer, J. Hankache, D.Lerose, S. Christiansen, J. Michler, L.Philippe. Crystal Growth & Design, 2010, Vol 10, 587-596
- 4) «Erosion–corrosion resistance of Ni composite coatings with embedded SiC nanoparticles». J.A. Calderón, J.E. Henao, M.A. Gómez. Electrochimica Acta 124 (2014) 190–198
- 5) "Antimicrobial Activity of Thin Solid Films of Silver Doped Hydroxyapatite Prepared by Sol-Gel Method". S. L.Iconaru, P. Chapon, P. Le Coustumer, D. Predoi. http://dx.doi.org/10.1155/2014/165351
- 6) "Polymer films with embedded Metal Nanoparticles". A Heilmann. Springer
- 7) "Particle Size Measurements". H. Merkus. Springer
- 8) "Ellipsometry at the nanoscale". M. Losurdo, K. Hingerl. Springer
- 9) "Rf GDOES analysis of composite metal/ceramic electroplated coatings with nano- to microceramic particles'size: issues in plasma sputtering of Ni/micro SiC coatings. A Lanzutti, E. Marin, M. Lekka, P. Chapon, and L. Fedrizzi. DOI 10.1002/sia.3768
- 10) « SEM Observation of Inclusions in Steel Samples Using Fast Cleaning and Modification of the Surface by Glow Discharge». K. Tsuji, K. Shimizu. ISIJ International, Vol. 53 (2013), No. 11, pp. 1936–1938