NEW APPLICATIONS OF CONTINUOUS AND PULSED RF-GD-OES FOR THE ANALYSIS OF ADVANCED MATERIALS

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Radiofrequency glow discharge (rf-GD) coupled to optical spectrometry (OES) is a powerful tool for the direct solid analysis of bulk materials, either conducting or insulating, and to carry out the in-depth profile analysis of thin layers. Generally, the rf-GD is operated in continuous mode; however, lately the interest in pulsed rf-GD (PGD) is steadily increasing because of its special features such as higher instantaneous powers leading to enhanced emission intensities and reduced thermal stress, important for heat-sensitive samples. Nevertheless, little investigation has been made regarding the analytical performance of rf-PGD-OES, compared with the continuous counterpart, and deeper studies for different kind of samples and coatings, especially for non-conducting and thermally unstable samples, is still lacking.

In this light, the capability of rf-GD-OES has been investigated for the compositional in-depth profiling for semi-conductors such as highly ordered and self-assembled magnetic nanostructures, to compensate for the existing demand of quality control during their development processes. For the case of insulators, limits still exist related to the low power deposited in the plasma. To improve their analysis, thin conductive layers were deposited on the surface and their influence together with the addition of a magnetic field, were measured in function of the sample dimensions and evaluated in order to improve instrument performances.

To investigate properly the effective use of rf-PGD-OES a more complete understanding of the temporal separation of various excitation and ionisation processes is required. Therefore, time-resolved measurements have been carried out, concentrating on the 'pre-peak' behaviour. A large number of copper emission lines have been studied and the effect of key discharge parameters (pressure and power) and pulse parameters (frequency and duty cycle) on the pre-peak behaviour has been investigated in an attempt to gain a better insight on these phenomena.

As last part, a critical qualitative comparison of the analytical performance of rf-GD-OES, applied either in continuous or pulsed mode, was carried out for a broad range of conducting and insulating materials. The comparison will be in terms of crater shapes, sample sputtering rates and emission yields calculated for the materials under both discharge modes and through qualitative in-depth resolution measurements of a tinplate coating and a multi-coated commercial glass sample. These optimised conditions will be applied during the calibration in pulsed mode to obtain quantitative in-depth profiles for a new type of composite electrodes with semi-conductive nanoparticles, employed in the extraction and degradation of organic pollutants in aquatic systems.