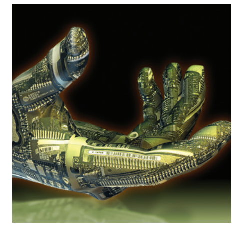
HORIBA Scientific

GDOES

Thin layers of MoS_2/Pb nanocomposite coatings for solid lubricants: depth profile analysis by GDOES



Application Note

Material Science GD41

Yanhong Wu[1], Akira Fujimoto[2], Sofia Gaiaschi[3], Kayvon Savadkouei[4], Patrick Chapon[3]
[1]HORIBA Trading Co., Ltd China, [2]HORIBA Techno Service Co., Ltd Japan., [3]HORIBA Scientific France, [4]HORIBA Scientific USA

Abstract: Pulsed RF Glow Discharge Optical Emission Spectrometry offers ultra-fast elemental depth profiling capability for the investigation of thin and thick films. Thanks to the use of a pulsed RF source, coupled with a high resolution optical spectrometer, the GD Profiler 2 provides an excellent depth resolution, allowing the fast evaluation of the coating quality. In this application note, we focus on a MoS_2/Pb composite multilayered sample, used as a solid lubricant. The analysis of such a sample shows the excellent performance of this instrument for the study of nm-thick complex coatings.

Key words: Multilayer, Depth Profile Analysis, GDOES, Depth Resolution, Pulsed RF source

Introduction

Conventional lubricants might be inadequate for applications such as:

- **Ceramics**, since for these particular surfaces, chemically active lubricant additives have not been found.
- **Fasteners**, which are usually tightened and unscrewed after a high temperature treatment during which a liquid lubricant will not survive.
- **Metal forming applications which involve plastic deformation**, which require lubricants that can support a high bearing-load, combined with a low shear stress.

For these applications, dry lubricants, or solid lubricants, are very useful. These materials, despite being in the solid phase, are able to reduce friction between two surfaces sliding against each other without the need for a liquid oil medium, and they offer lubrication at temperatures higher than liquid and oil-based lubricants.

The low-friction characteristics of most dry lubricants are attributed to a layered structure on the molecular level with weak bonding between layers. Such layers are able to slide relative to each other with minimal applied force, thus giving them their low friction properties.

The predominant materials used as dry lubricants are graphite and molybdenum disulfide (MoS_2). MoS_2 has a hexagonal crystal structure with the intrinsic property of easy shear. Its lubrication performance often exceeds that of graphite, and it can also be used for vacuum applications, whereas graphite cannot.

Despite such good performance, the application of MoS_2 is greatly limited in a terrestrial atmosphere due to its sensitivity to moisture. However, there is an increasing demand for robust solid lubricants that can adapt to different environments. More work needs to be performed in order to optimize MoS_2 not only to gain a coating structure with favorable frictional properties, but also to minimize the sensitivity to the attack of water molecules. The doping of the MoS_2 coating is an economical way to achieve such results. Among all elements, Pb is one of the most promising candidates, as not only is it an excellent solid lubricant itself, but it also can prevent the formation of randomly oriented MoS_2 in the sputtering plasma.

Instrumentation and sample preparation

The GD Profiler 2 (**Figure 1**) couples an advanced Pulsed RF Glow Discharge source to a high resolution, wide spectral range Optical Emission Spectrometer.

Figure 1. GD Profiler 2

This technique relies on the precise and fast (typically µm/min) sputtering of a representative area of the investigated sample by Ar+ ions and accelerated neutrals with very low kinetic energies. The sputtered atoms are then excited by the plasma and their de-excitation leads to the emission of photons with characteristic wavelengths, enabling their elemental identification. All elements of interest are simultaneously measured as a function of the sputtering time, thanks to a spectrometer.

Thanks to our pulsed RF source, a high depth resolution can be achieved, avoiding unwanted diffusion of the elements during the measurements. Moreover, this excitation source makes the GD Profiler 2 an excellent instrument for the analysis of conductive, insulating and hybrid materials.

A MoS_2/Pb composite was synthesized by an unbalanced magnetron sputtering system. The composite coating was deposited on 304 steel. At the interface between the steel and the coating, a Ti interlayer (200nm thick) was deposited in order to improve not only the adhesion, but also the bearing capacity.

The microstructures of the composite coating changes from a loose columnar structure for pure MoS_2 to a compact amorphous phase for Pb-doped MoS_2. The increase of the Pb content leads to an increase of hardness and elastic modules of the MoS_2/Pb composite coating.

Results

Figure 2 shows the result of the GD-OES analysis. A multilayer structure can be clearly identified.

In **Figure 3,** a zoom on the Pb depth profile is presented. Despite the extremely low thickness of a single stack, with each layer being about 2~5nm thick, the GD Profiler 2 is able to provide an excellent depth resolution, as every layer can be clearly resolved.

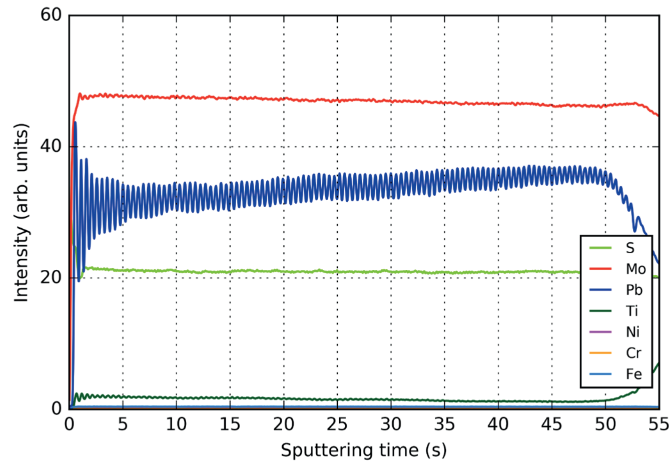


Figure 3. Zoom on the stacks coatings to see the depth resolution

The same sample was also tested on competitors' equipment and the obtained result is presented in **Figure 4.** The multilayer structure cannot be observed.

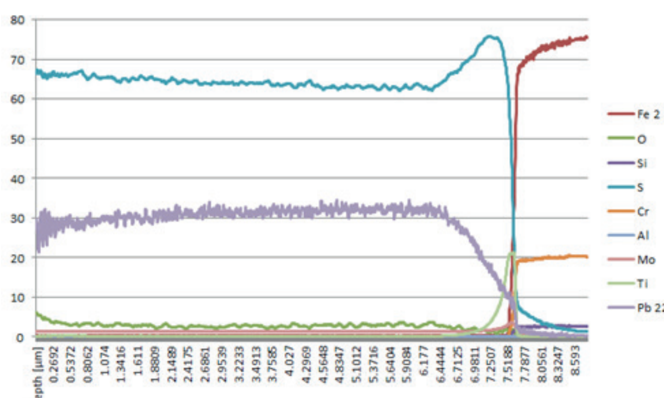


Figure 4. Full depth profile of MoS_2/Pb coating obtained by the competitors.

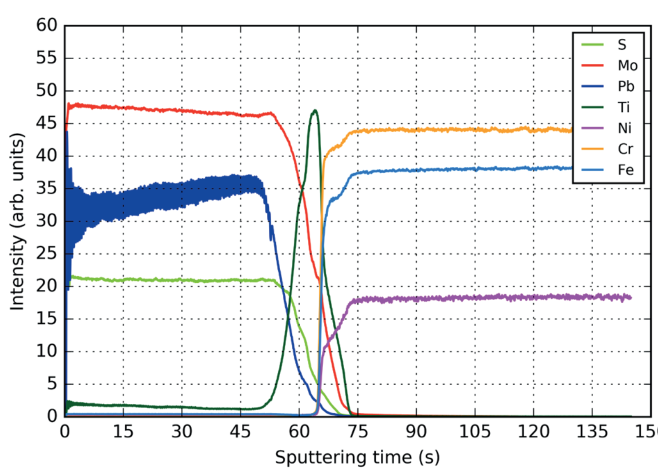


Figure 2. Full depth profile of MoS_2/Pb coating obtained with the GD Profiler 2.

Conclusion

HORIBA Pulsed RF Glow Discharge Optical Emission Spectrometry offers superb depth resolution down to the nanometer scale or below, made possible by the unique characteristics of the advanced pulsed RF GD source and the ultra fast detection capability of the optical system.

Reference

Low humidity sensitivity of MoS_2/Pb nano composite coatings. Hao Li, Guangan Zhang, Liping Wang. Wear 350-351(2016)1-9.