Comparative depth-profiling analysis of nanometer-metal multi-layers by ion-probing techniques

R. Escobar Galindo, R. Gago, A. Lousa, J.M. Albella

We examine here the depth resolution (interface width) in elemental analysis and depth profiling of complex layer systems of three ion-probing techniques, each of which has pros and cons:

- **Rutherford backscattering spectrometry (RBS);**
- **secondary ion mass spectroscopy (SIMS); and,**
- **glow-discharge optical emission spectroscopy (GDOES).**

RBS is a non-destructive technique that requires no standards for quantification, although access to medium-scale ion-source facilities is needed.

SIMS maintains nanometer (nm) resolution at greater depths but at the expense of longer data-acquisition times.

Finally, GDOES allows depth profiling quickly and accurately, although depth resolution degrades linearly with depth due to sputtering effects (e.g., crater shape and chemical modifications), among other factors.

We discuss these ion-probing techniques in the light of new results obtained with chromium/titanium multilayer structures with individual layer thicknesses between hundreds of nm and a few nm. We resolved ultra-thin chromium layers of 2.5 nm and 5 nm, buried at different depths in titanium matrixes with thicknesses up to 3 μm, and used the results to evaluate the depth resolution of the ion-probing techniques.

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1. Introduction

In recent decades, the use of metal and/or metal-compound multilayer coatings has been extended to an enormous range of applications. By combining synergistically the properties of different materials, it is possible to tune and to optimize desired functional properties. Typical examples involve hard protective coatings for mechanical parts and tools, optical coatings for lenses, filters and architectural glass panels, barrier contacts for microelectronics, quantum superlattices, thermal barriers for gas turbines, thin-film waveguides, and biomedical coatings. In general, for these and other applications, complex coating structures have to be designed in order to accomplish very strict requirements (e.g., thickness homogeneity in the nanometer (nm) range, low surface roughness and abrupt or graded interfaces).

Fig. 1 shows a typical advanced multilayer coating. The first property that any coating should fulfill is good adhesion to the substrate, so it is common in many devices to add adhesive or barrier interlayers (e.g., 50–100 nm of Cr or Ti for metallurgical protective coatings). This buffer layer may also serve as a barrier layer avoiding element inter-diffusion at the film/substrate interface. The structure of the coating itself may comprise a multilayer structure, a graded in-depth composition or even 2D and 3D nanocomposite systems. Of special relevance are multilayer coatings with bilayers of the order of some nm (superlattices), which, among other applications, are designed to reach film hardness in the range of superhard materials (>40 GPa) [1,2], to improve magnetic or transport properties [3,4] or, finally, to tune the optical performance of the films [5,6]. In all these applications, sharp interfaces and a low

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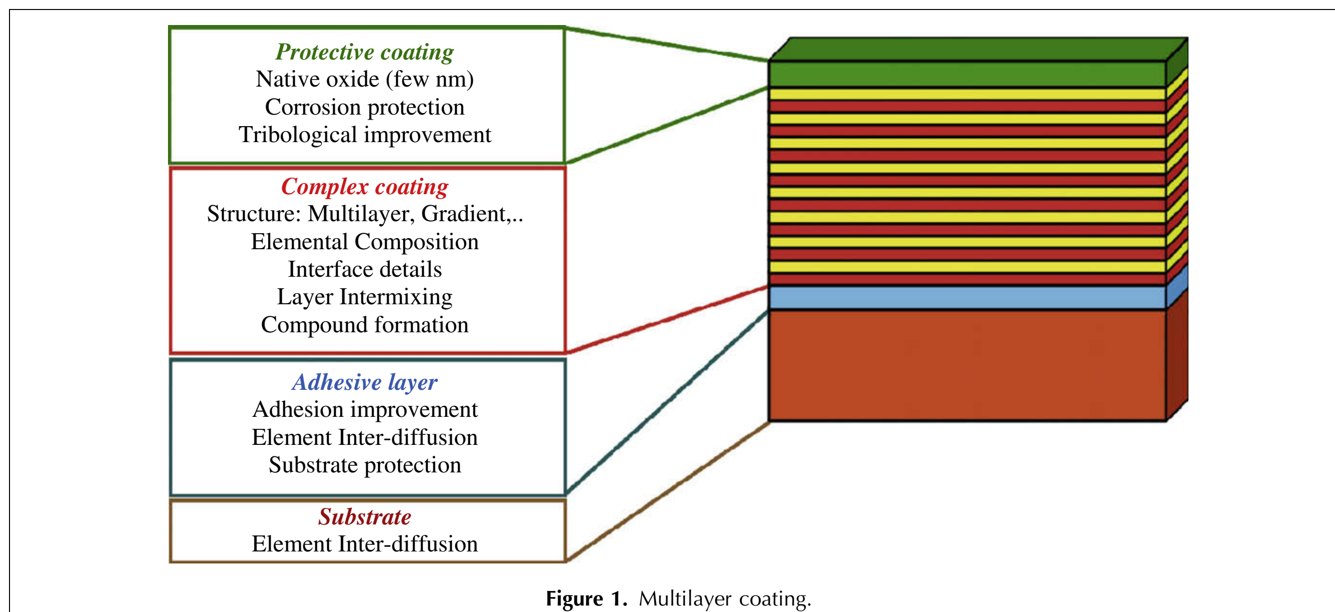

Figure 1. Multilayer coating.

degree of mixing between the component materials are strictly required. Finally, most coatings have a top functional layer to tailor the desired surface properties (e.g., low friction, optical reflectivity, or corrosion resistance).

The analytical characterization of these layered structures requires high-resolution analytical techniques able to provide information about surface and depth composition at the nm level (for references, see the excellent review by Werner and Garten [7]). Well-known techniques [e.g., Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), secondary ion mass spectroscopy (SIMS), Rutherford backscattering spectrometry (RBS) and, more recently, glow-discharge optical emission spectroscopy (GDOES)] are generally used for this purpose. In Table 1, the main characteristics of these five techniques are compared. XPS, AES and SIMS suffer from preferential sputtering artifacts. However, in XPS and AES, this effect derives from the analysis of the residual surface composition and, in SIMS, from the flux composition of sputtered ions. Moreover, the maximum depths of analysis of AES and, in particular, XPS are restricted to the sub-μm range, thus limiting the study of thicker coatings. On the other hand, RBS, SIMS and GDOES are all ion-probing techniques capable for analyzing to depths of several μm with nm resolution although without chemical information (as when using XPS and AES). From the above techniques, we have therefore chosen RBS, SIMS and GDOES for the analysis of multilayer systems with individual thickness ranging from a few to some hundreds of nm and a total thickness greater than 1 μm. In the following paragraphs, we briefly describe the principles of the profiling techniques under consideration.

Table 1. Comparative summary of the main characteristics of different surface-analysis techniques

	AES	XPS	RBS	SIMS	GDOES
Excitation probe	Electrons	Photons (XR)	Ions	Ions	Ions
Emission (=detection)	Electrons	Electrons	Electrons	Ions (m/e)	Photons (λ)
Sputtering	Ion beam <10 keV	Ion beam <10 keV	No	Ion beam <10 keV	DC/RF <50 eV
Atom mixing	Yes	Yes	No	Yes	No
Crater effect	No	No	No	Yes (–)	Yes (++)
Max. Depth analysis (μm)	1	0,1	5	5	>100
Depth Resolution (nm)	0.5–2.5	0.5–2.5	5	0.5–2.5	1
Lateral Resolution (nm)	10	10^2	10^5	10	10^6
Chemistry Information	Yes (+)	Yes (++)	No	Yes (+)	No
Detection Limit (ppm)	10^3	10^3	10^3	10^{-1}–10	1
Elemental range	Z > 2	Z > 2	Z > 2	All	All
Calibration Method	Easy	Easy	Standard free	Complex	Complex
Crater diameter (mm)	<1	<1	No crater	<1	4
Vacuum (mbar)	10^{-10}	10^{-10}	10^{-6}	10^{-10}	10^{-2}
Acquisition time	Moderate	Slow	Moderate (minutes)	Slow (hours)	Very fast (seconds)

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