Application Note # 133

Characterization of Atomic Layer Deposited Metal Films and Nanolaminates

Thin metal layers and nanolaminates ($Pt-Al_2O_3-Pt-Al_2O_3$) can be effectively characterized in terms of thickness and optical properties with Multi-Parametric Surface Plasmon Resonance (MP-SPR)

Introduction

Miniaturization of optics, electronics and photovoltaics is an on-going process in todays industries. For effective application and manufacturing development is highly important that the materials can be characterized effectively. As the miniaturization is progressing more and more to the nanometer scale, the characterization techniques are pushed to the limit. There are also new phenomena available in the nanoscale, for example in hybrid nanolaminate materials, which do not have any effective characterization techniques.

Characterization of nanoscale metal layers has been a challenging task [1] with the current prevailing methods, and nanolaminates with alternating layers have been even more challenging systems to characterize. Surface Plasmon Resonance (SPR), an optical surface phenomenon more commonly utilized in biochemical interaction characterization, is extremely sensitive to thin layers having plasmonic properties, including most transition and noble metals. Multi-parametric SPR (MP-SPR) measures the intensity of the reflected light for a wide range of angles at multiple wavelengths, and can be used as an extremely sensitive thickness characterization tool for metals and metal hybrid laminates.

Atomic Layer Deposition (ALD) provides precise control of film thickness down to atomic scale. With the possibility to deposit various metals and nanolaminates of different materials, ALD enables a high order of control in fabrication of high quality coatings.

Materials and methods

Pure glass substrates were coated with target thickness of 11 nm Platinum (Pt), or with a nanolaminate of alternating Al_2O_3 and Pt 5 nm each. In the nanolaminate preparation, one sensor was removed for analysis after each consecutive deposition step. The sensors were coated in a PICOSUN™ R-150 ALD reactor, using (trimethyl) methylcyclopentadienylplatinum and air as the Pt precursors and trimethylaluminum and air as the Al_2O_3 precursors.

The coated substrates were analyzed using BioNavis SPR Navi™ 200 instrument by recording intensity of the reflected light for angles 38 to 78 degrees at 785 and 670 nm wavelengths. The SPR curves (angular spectra) were analyzed using Fresnel-formalism [2].

Results and discussion

The Pt metal layer was on average 11,0 nm thick, with deviation of less than 1,5 nm (N=3) (Fig. 1). As the Pt deposition is a fairly difficult ALD process, this amount of deviation is normal.

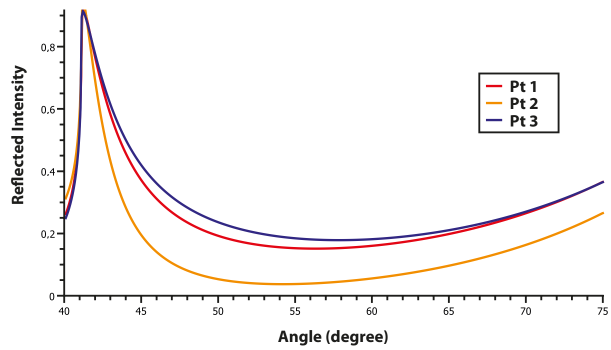
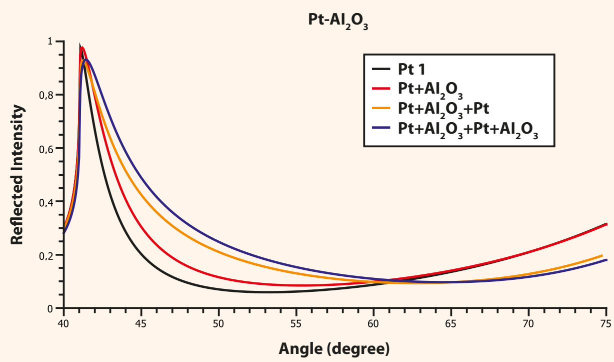


Figure 1. ALD deposited platinum layers (target thickness 11 nm) measured with MP-SPR. Thickness of each layer was determined: Pt1=10.8, Pt2=9.9 and Pt3=12.3. Even small differences in the thickness can be seen from full SPR curves.



Layer#	Material	Target thickness [nm]	Measured thickness [nm]
1	Pt	5	5,4
2	Al_2O_3	5	4,7
3	Pt	5	6,0
4	Al_2O_3	5	7,3

Figure 2. Full SPR curves of Al_2O_3-Pt nanolaminates measured with MP-SPR. Each of the following four layers (Pt- Al_2O_3-Pt- Al_2O_3) added formed a logical series and thickness of the layers was successfully calculated.

Excellence in Surface Plasmon Resonance

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The nanolaminate deposition steps were measured individually from different sensors (Fig. 2), and they formed a logical series with new layers added. It was also possible to determine the thickness of all the individual nanolaminate layers separately from the 4-layer deposition (Fig. 2, Table).

The deviation of the ALD process for platinum layers was less than 1,5 nm in all different sensors in the laminate deposition.

MP-SPR is able to distinguish between Pt-Al_2O_3 (10 nm each) and a nanolaminate of Pt-Al_2O_3-Pt-Al_2O_3 (5 nm each) (Fig. 3). While both surfaces have same overall thickness of 20 nm with same ratio of the components, each of them produces a distinct shape in the SPR curve. Using theoretical modelling, it was clear that the curve shape for the Pt-Al_2O_3-Pt-Al_2O_3 cannot be obtained by the two layer model Pt-Al_2O_3.

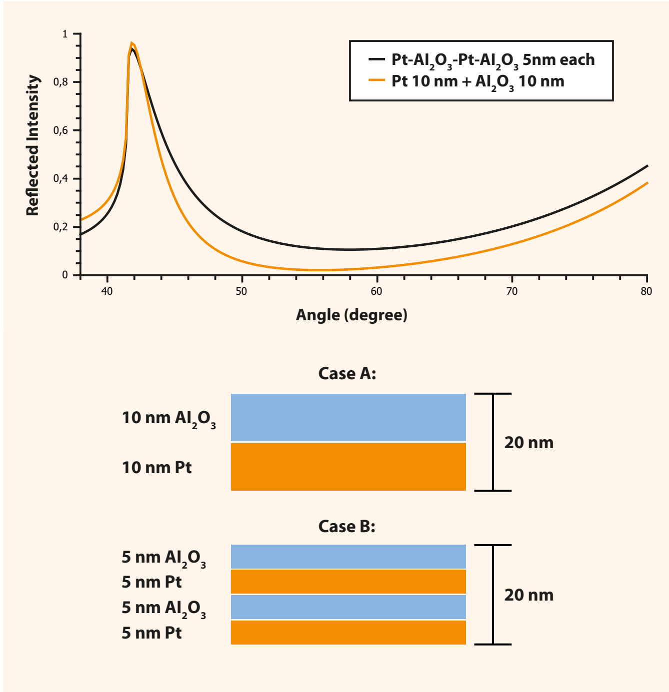

Figure 3. SPR curves of Pt-Al_2O_3 (10 nm each) and a nanolaminate of Pt-Al_2O_3-Pt-Al_2O_3 (5 nm each). Shapes of the curves are different even if coating has same overall thickness of 20 nm with same ratio of the components.

Conclusions

The SPR proved to be an effective tool for the nanoscale metal layer characterization. Even small deviations in the metal layer thickness are easily characterized due to SPR sensitivity to the plasmonic material thickness. It was similarly possible to characterize the nanolaminate thicknesses at each step and after whole deposition which, to our best knowledge, is not possible with other methods. The new characterization method, together with advanced ALD deposition method, allows new applications and innovations in optics, electronics and photovoltaics.

References

[1] Hilfiker, J.N., et al., Thin Solid Films, 2008. 516(22): p. 7979-7989.

[2] Sadowski, J., Korhonen, I., and Peltonen, J., Optical Engineering, 1995. 34(9): p. 2581-2586.

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