Homogeneity of lubricant layer on rough metallic substrates - A multivariate analysis of spectroscopic ellipsometry data

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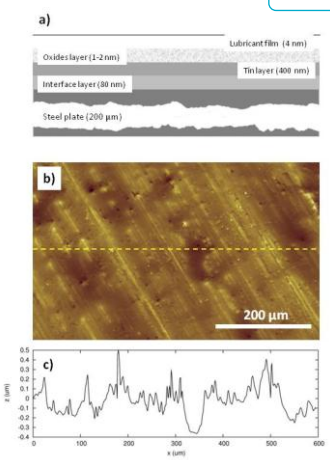
Introduction



- Tinplate steel (TPS) obtained by electroplating a tin layer over a mild steel substrate having low carbon content
- Roughness : Ra = 153 ± 24 nm ; Rq = 193 ± 28 nm
- Protective lubricant film by electro-spraying. Film thickness : 10 nm

Challenge : detecting the lack of uniformity of the lubricant layer on a rough industrial metallic substrate without optical model for the substrate

Constraints : 'standard' optical techniques not applicable (roughness >> film thickness)

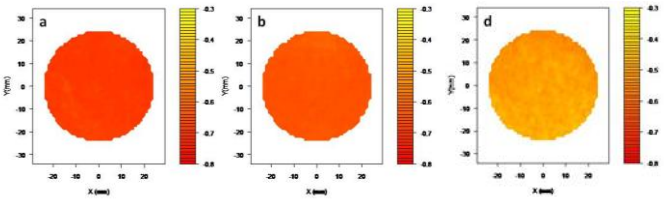
Suggested solution : spectroscopic ellipsometry combined with statistical analysis for multivariate outlier detection

Fig. 1 : Complex structure and roughness of the TPS substrate (a) scheme (b) Optical profilometry (c) Scan

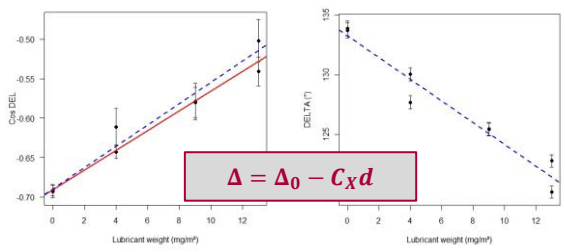
Spectroscopic ellipsometry mappings

Spectroscopic ellipsometry : Non-destructive optical analysis technique based on the relative change of polarization of the p and s components of light at the interface between two media characterized by different optical properties

$$\rho = \frac{r_p}{r_s} = \tan\Psi\, e^{i\Delta}$$

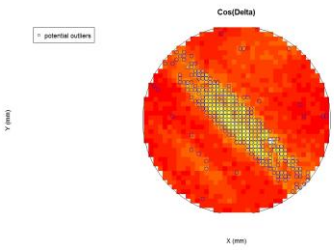


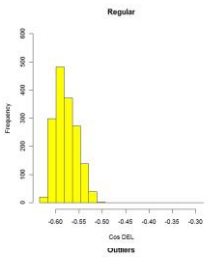
- Imaging of the surface at 1.49 eV
- Increase of the cos Δ value with the lubricant film thickness (From letft to right : 0 mg/m², 4 mg/m² and 13 mg/m²)



Validity of the Drude approximation for dielectric films after correction for potential outliers

Outlier detection – multivariate data



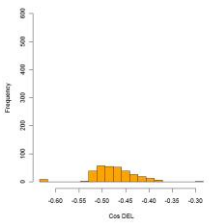


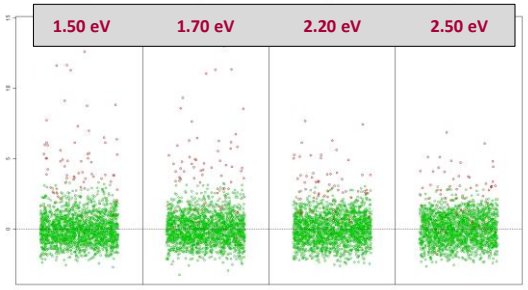
Outlier detection in a multivariate problem: Values of the Mahalanobis distance

$$D_M = [(x-\mu)^T C^{-1} (x-\mu)]^{\frac{1}{2}}$$

distributed according to a χ^2 law.

Breaks in the tails of the distribution = potential outliers





Conclusion

- Solution does not require an optical model as usually requested in spectroscopic ellipsometry
- Identification of potential outliers in a multivariate problem : importance of the spectral range (IR better than UV-vis due to roughness)
- Validity of the Drude approximation (linearity of Δ) for (very) thin dielectric films on rough metallic substrates
- Applicable to other imaging techniques (e.g. Imaging ellipsometry) and other complex surfaces