Global Research and Development

Breakthrough on Glow Discharge Optical Emission Spectroscopy analysis applied to rough and oxidized metal surfaces

S. CREMEL(1), E. GAGATEK(1), P. CHAPON(2)

(1) ArcelorMittal Global R&D, Maizières Automotive Products, Voie Romaine, 57280 Maizières-les-Metz

(2) HORIBA Jobin Yvon S.A.S. 16-18, rue du Canal - 91165 Longjumeau

Industrials samples can be difficult to analyze by GDOES, especially Press Hardening Steels (PHS) after Hot forming treatment exhibit a strong surface oxidation and roughness (fig1) leading to difficulty in analysis (strong leaking and Ar plasma strongly contaminated by Oxygen and Nitrogen from the atmosphere).

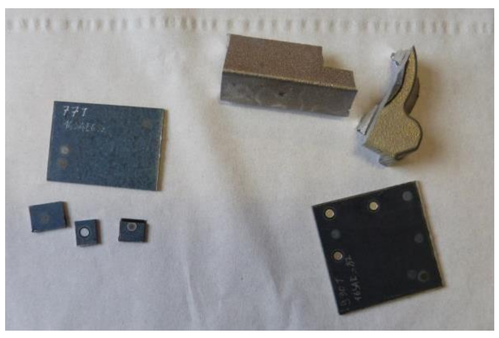


Figure 1 :Typical oxidized and rough surfaces of a thermal treated PHS steel or molden parts

GDOES analysis

GDOES is a destructive sputtering based analytical method. An Argon based cold plasma sputters the sample surface resulting in optical emissions whose specific wavelengths depends on constitutive chemical elements. The rather fast technique allows assessing chemical distribution profiles depending on analysed depth. This technique is widely used on research and quality issues in Arcelormittal R&D laboratories dealing with subjects such as metallic coating, surface selective oxidation, surface treatments, surface coloration, protective oil paints, pre-phosphatation and phosphatation, etc.

Developed solution

An interesting solution –currently at the prototype stage- has been found by HJY and is now used by Arcelormittal surface Analysis Laboratory. It consists in using a hermetic protective bell that surrounds the sample.

Application 1 : oxidized & porous surfaces

Figure 3 shows an Alusi® coating depth profile. The product has already underwent a press hardening thermal treatment: the different coatings phases and iron diffusion in coating are clearly visible. This kind of depth profiles were typical quasi-impossible to obtained before using the Argon over-pressure protective bell.

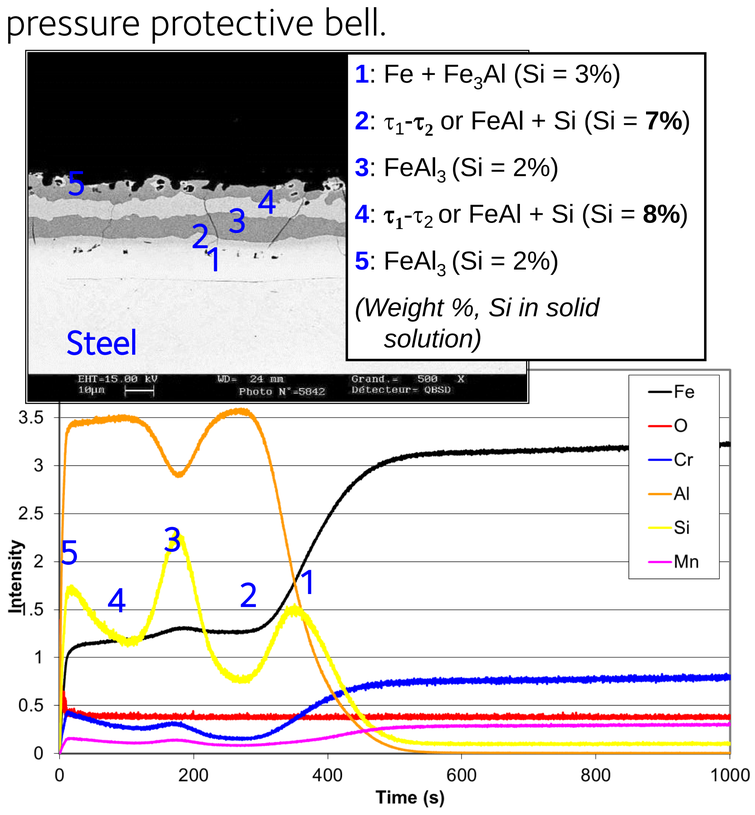


Figure 3 : GDOES depth profile of a thermal treated Alusi coating evidencing perfectly the different coating phases

Application 2: rough surfaces

Figure 4 shows how improvement of depth profiles quality is clear on a high surface roughness Automotive aluminium molten piece. Using the bell allows acquiring a relevant depth profile no more contaminated by strong O and N signals, optical emission intensities are in agreement with a smooth sample and surface layer and interface are well resolved. On this specific product GDOES can now be used to characterize nanometric MgO passivation layer on aluminium alloys, before that X-Ray Photo Electron Spectroscopy were required.

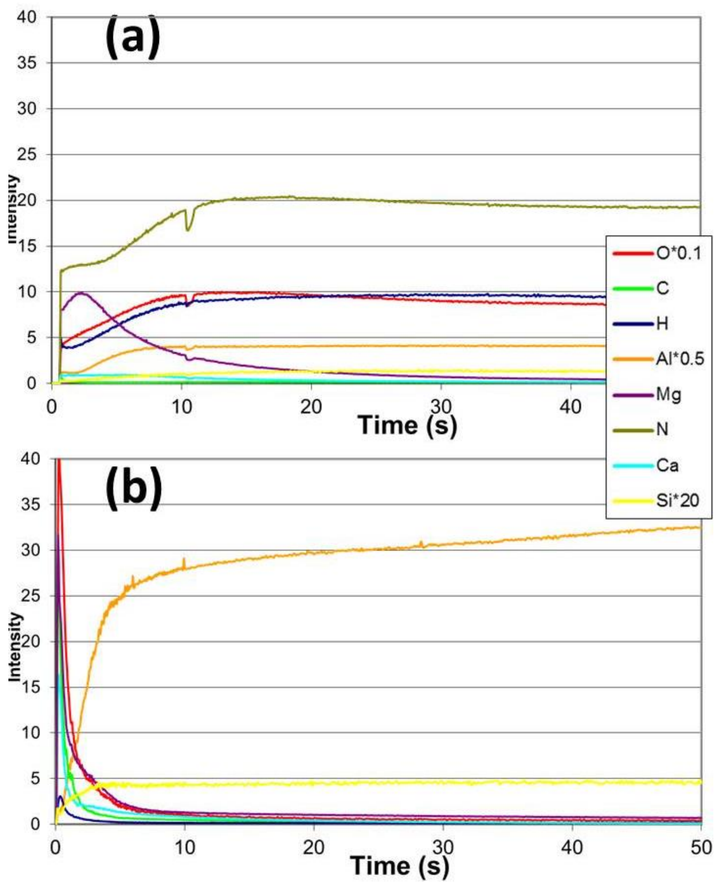
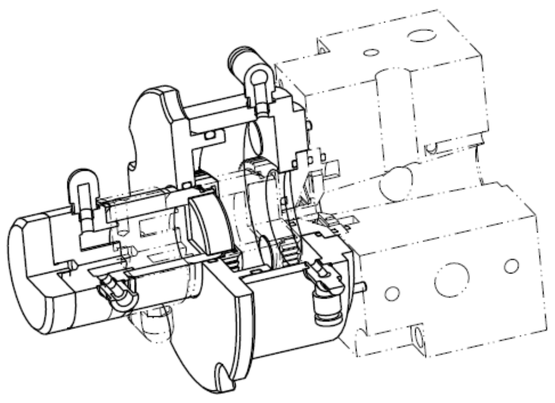


Figure 4 : GDOES depth profile without (a) or with (b) the Argon saturation protective bell



The Argon over-pressure bell

This bell developed by Horiba JY is in fact an additional hermetic cover that surrounds the sample and the anode. Within this cover a constant 10L/min Ar flow is applied. Thus, if a leaking occurs due to sample shape or bad surface state or planarity, Argon will leak into the analyzing plasma instead of the highly detrimental Nitrogen and Oxygen

Conclusion

This new tool is now implemented at ArcelorMittal surface analysis lab, helping us analyzing complex industrial samples. The mythological breakthrough is high as the range of applications is wide. The next steps are to test repeatability of profiles, study dependency of Optical Emission with Argon flow value in the Argon saturation protective bell and thus study how semi-quantification procedure can be transfer efficiently from non treated to thermal treated samples.

Document updated on March 2012