Investigation of the impact of surface cleaning procedures on near surface depth profiling using Glow Discharge Optical Emission Spectroscopy

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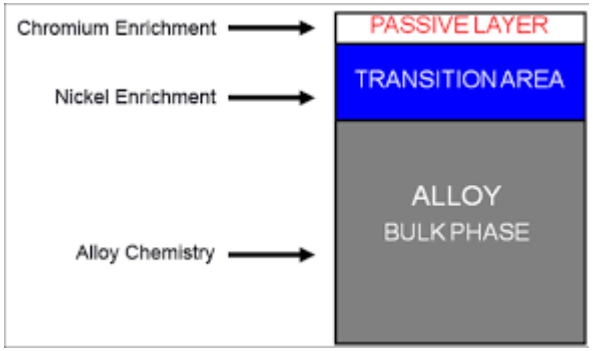
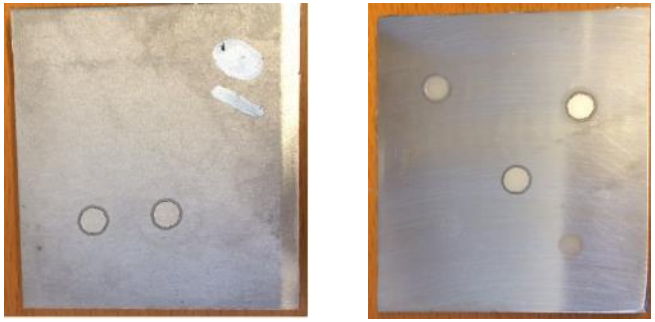
Introduction

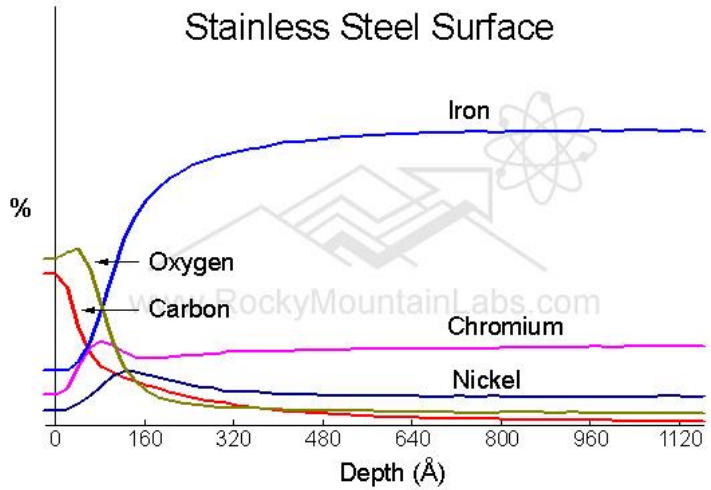
- Glow Discharge Optical Emission is an extremely fast technique for near-surface analysis in the few nm range, with considerably lower detection limits than electron the spectroscopic techniques XPS and AES.
- One technically – industrially important application to near – surface analysis is passivation layers on stainless steels.
- A challenge for all near-surface analytical techniques is sample preparation i.e. surface cleaning.

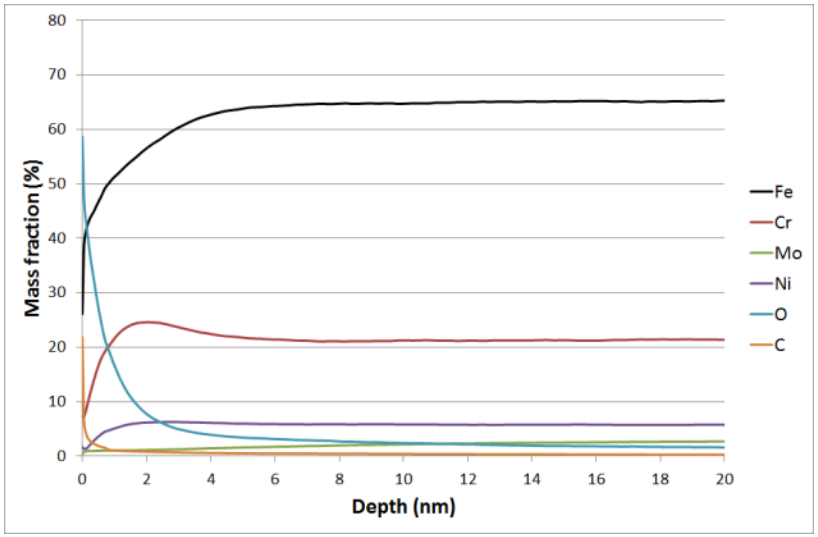




Pickled shot peened left, milled dry linished right

Examples of elemental depth profiling with AES and GD-OES



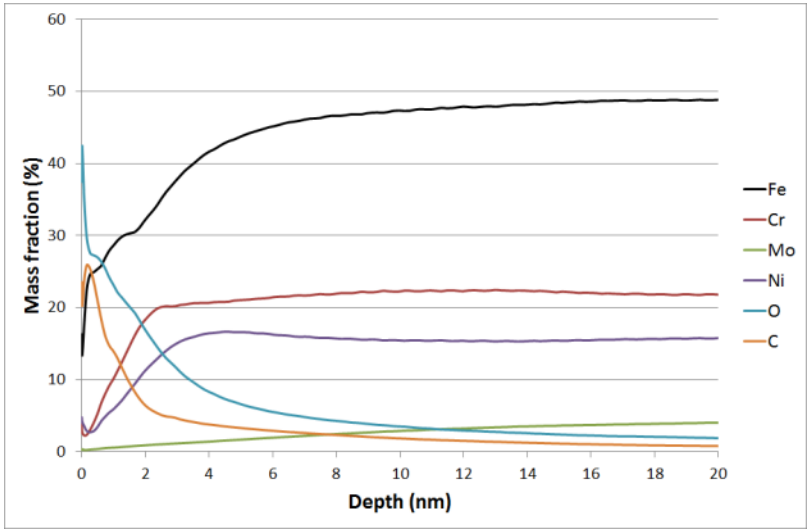


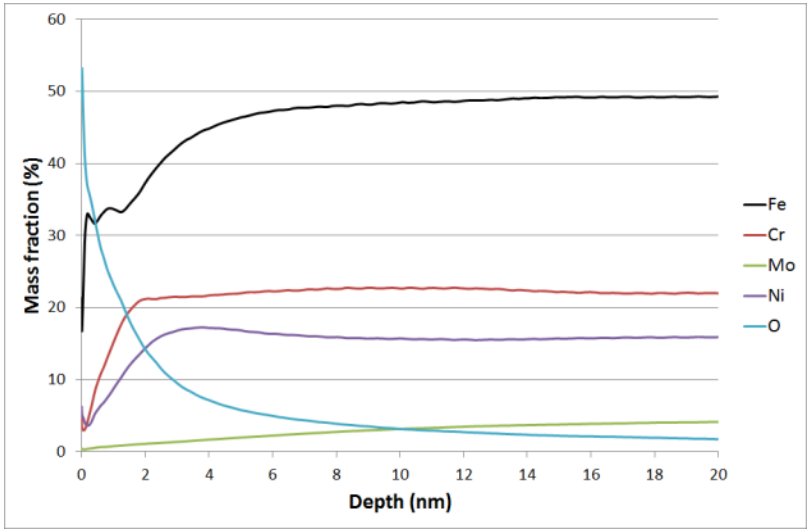
AES

GD-OES dry linished

The two techniques give very similar information (except the depth, the AES depth scale is clearly wrong!). The extremely thin chromium oxide layer is not distinctly resolved, but is seen as an enrichment of Cr in the first few nm.

GD-OES depth profile of a slightly contaminated stainless steel plate (pickled and shot peened)





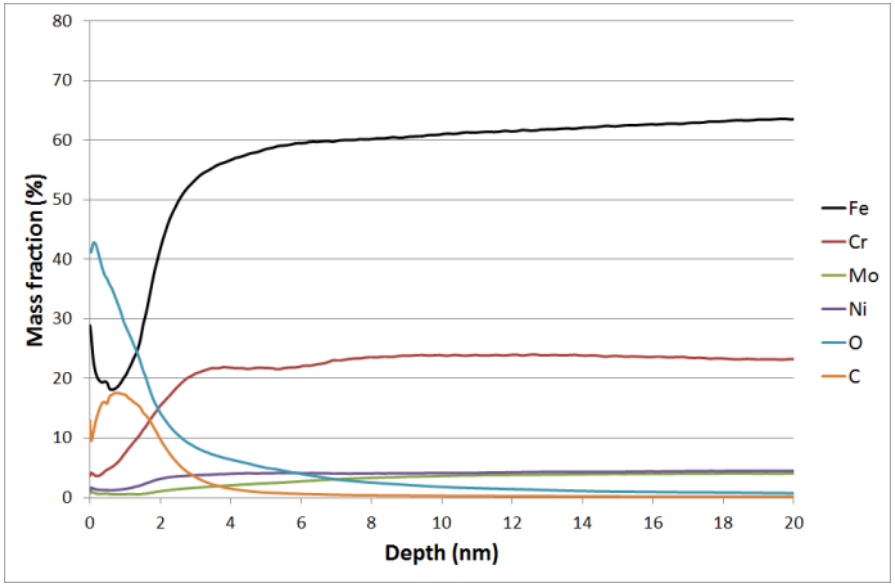
This sample was “vigorously” cleaned by first acetone, followed by ethanol and then dried by hot air. In spite of this, the residual surface carbon “masks” the Cr surface enrichment. Removing C from the quantification (and thereby the sum normalisation to 100%) does not help much.

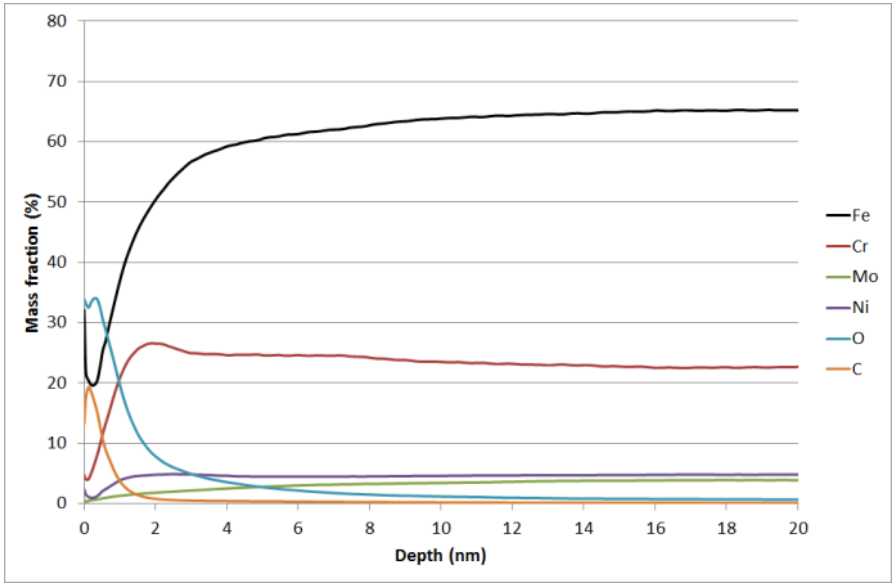
How to clean a surface more effectively – carbon dioxide snow spray

- The different carbon dioxide cleaning methods can remove gross contamination, paint, overlayers, grease, fingerprints, particles down to nanometers in size, hydrocarbon and organic residues.
- The basic methods include solid dry ice pellets, liquid CO2, CO2 snow (a hybrid method), and supercritical CO2.
- In CO2 snow cleaning, compressed liquid or gaseous carbon dioxide is expelled from a nozzle, condensing into a mixture of solid particles and gas, which impact the surface to be cleaned.

CO2 “snowgun”

Pickled shot peened stainless steel surface, cleaned in two steps

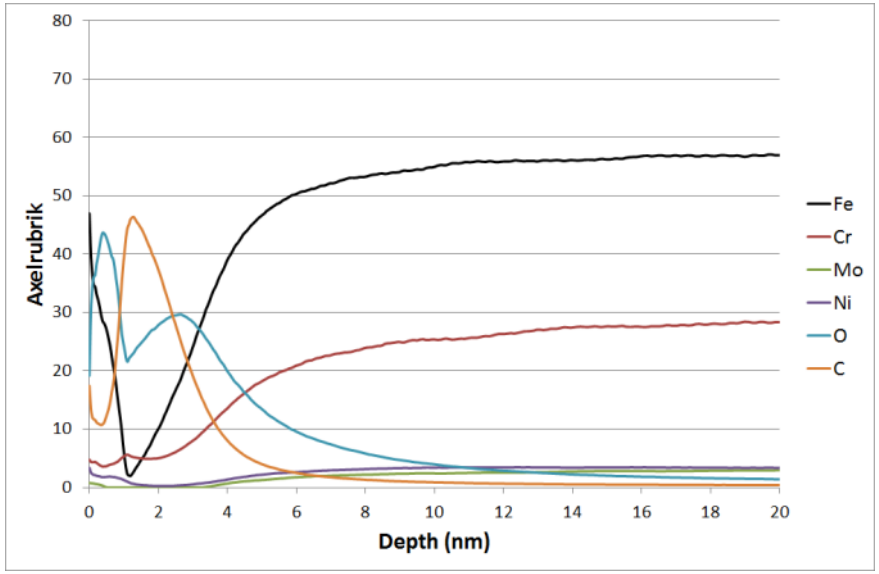


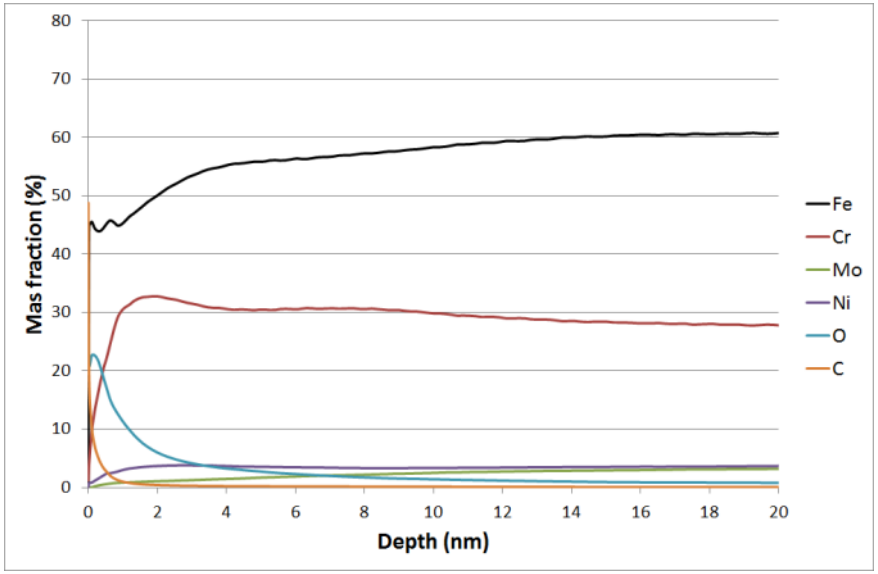


Cleaned with isopropanol and hot air – Not good enough!

CO2 snow as the final touch – Much better!

Dry linished stainless steel surface slightly contaminated by body grease (fingerprint)





Surface contaminated by fingerprint.

Contaminated surface cleaned by isopropanol and CO2

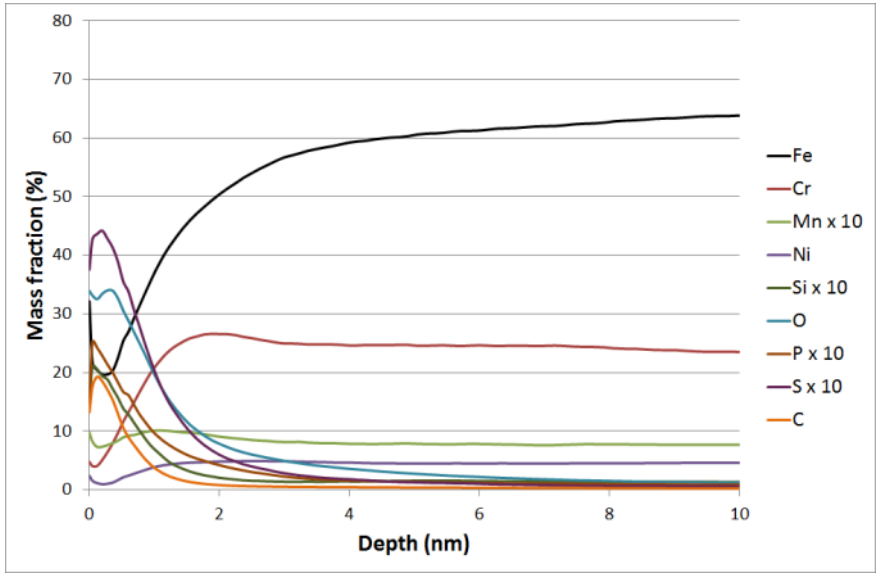
Surface carbon measured for different surface conditions

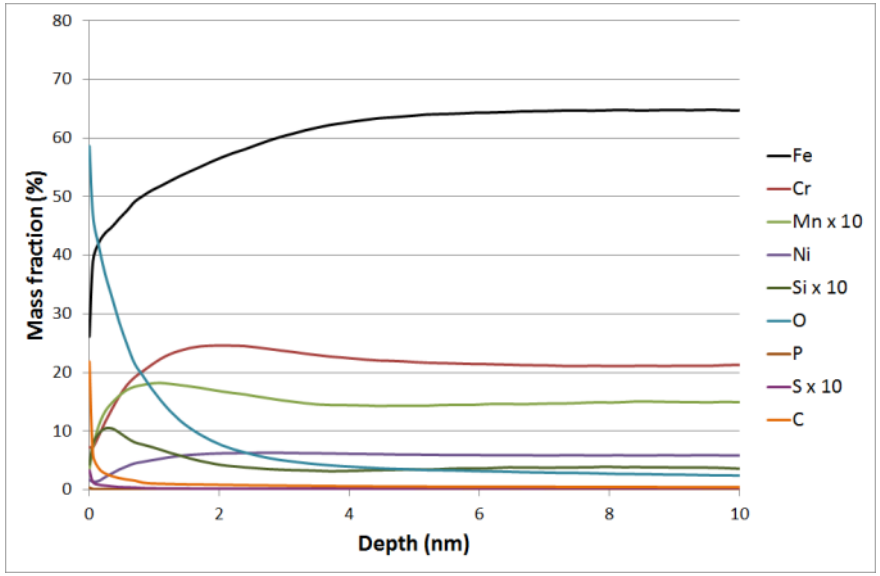
Sample	C (mg/m^2)	Corresponding layer thickness (nm)
Dry linished Al oxide 220	1,7 ± 0,6	3,4
Dry linished + CO_2	2,1	4,2
Dry linished + isopropanol +CO_2	4,0	8
Dry linished + body fat	28	56
Dry linished + body fat cleaned with isopropanol +CO_2	5,8	12

Blowing a clean surface with CO2 dry ice does not add any carbon, but rinsing with isopropanol adds a little. Cleaning a contaminated surface with isopropanol Followed by CO2 is very effective in removing the fat.

Also note that the GD-OES quantification underestimates the thickness of the contamination layers. This due to an error in the density calculation.

GD-OES provides much additional analytical information of minor elements





Cleaned pickled and shot peened steel surface. The presence of S (pickling) and Si (peening) is clearly visible.

Milled and dry linished (bulk) steel surface.

Discussion and conclusions

- Effective surface cleaning prior to analysis of nm passivation layers is critical in order to obtain correct analytical information (surprised?)
- Degreasing steel surfaces with solvents like isopropanol leaves too much residual surface carbon for a good characterisation of the passivation layer.
- Spraying the surface with CO2 dry snow as a final cleaning step is effective, yet a “reasonably” simple procedure compared with many other more extreme surface cleaning methods.
- Thank you for your attention!