

PHYSICAL AND CHEMICAL CHARACTERIZATION OF A DEPOSIT OF CHROMIUM OXIDE

Manon DURAND

Chemistry Laboratory (TE-VSC-SCC)

Student in Chemistry Engineer School (ESCOM)



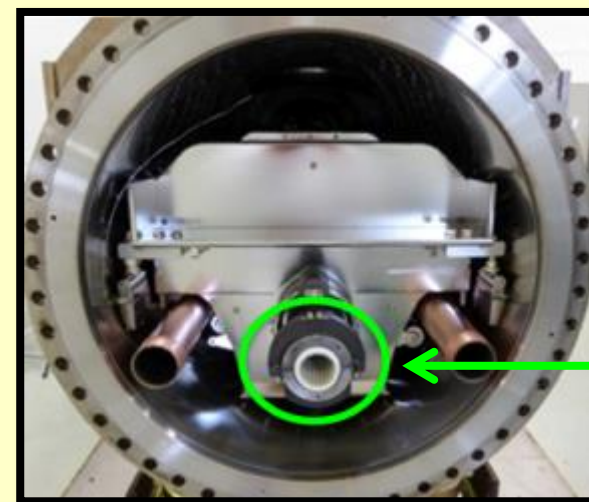
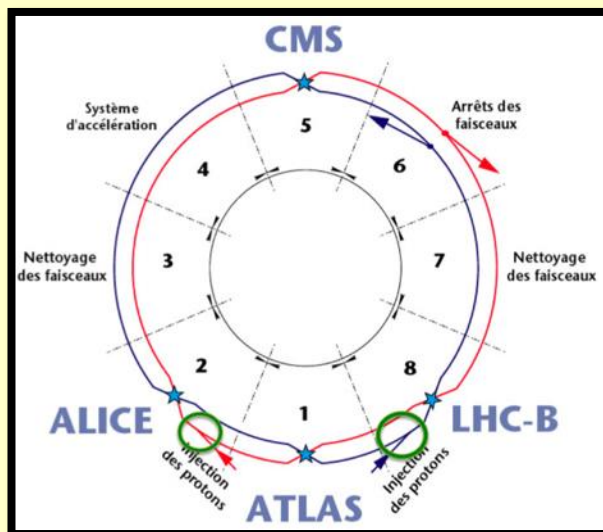


- **Introduction**
- **Establishment of the analytical methods and parameters**
- **Heat treatments**
- **Conclusion**



Injection Kicker Magnets

- electromagnetic devices composed of 4 magnets and 4 pulse generators
- guide, maintain and focus the particle beam on its near-circular orbit
- installed on the injection points of LHC
- 0,12Tesla in less than 900ns, for about 8ms.

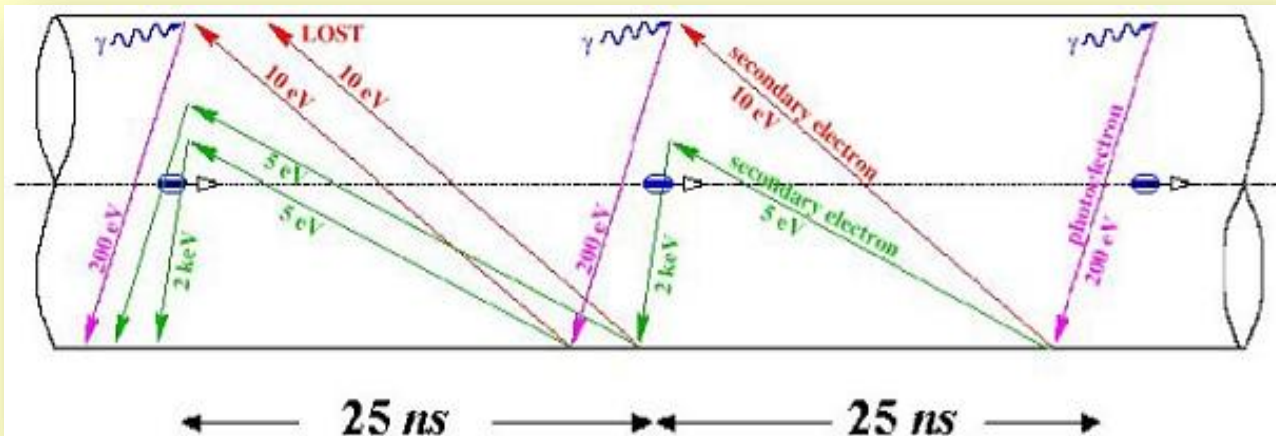


Vacuum chamber



- Problem with electron clouds

- vacuum chambers in alumina
- high SEY ~ 9-10



(by F. Ruggiero)

- adverse effects:
 - increase in pressure
 - rise of temperature of the cold magnets
 - instabilities, loss of beam.

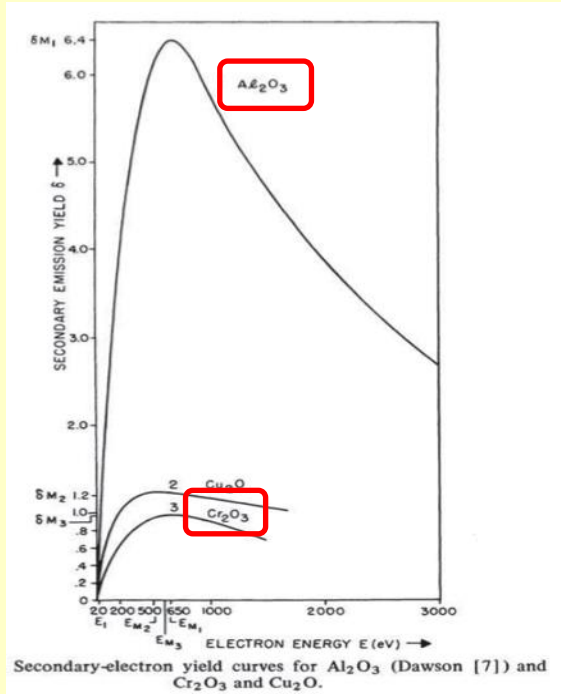


• Topic of the internship

- Mike Barnes (TE-ABT-FPS)

FINAL AIM: SEY reduction **1,4**

Cr₂O₃
Cr(III)



CrO₃
Cr(VI)



>500°C
Intermediary reductions
Oxygen release



Dr. Sudarshan, The Effect of Chromium Oxide Coatings on Surface Flashover of Alumina Spacers in Vacuum (1976).

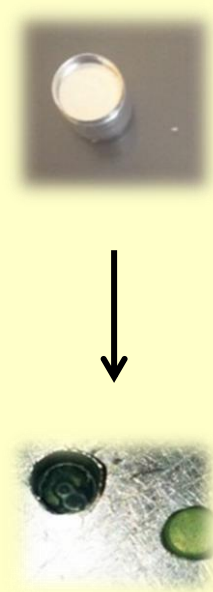
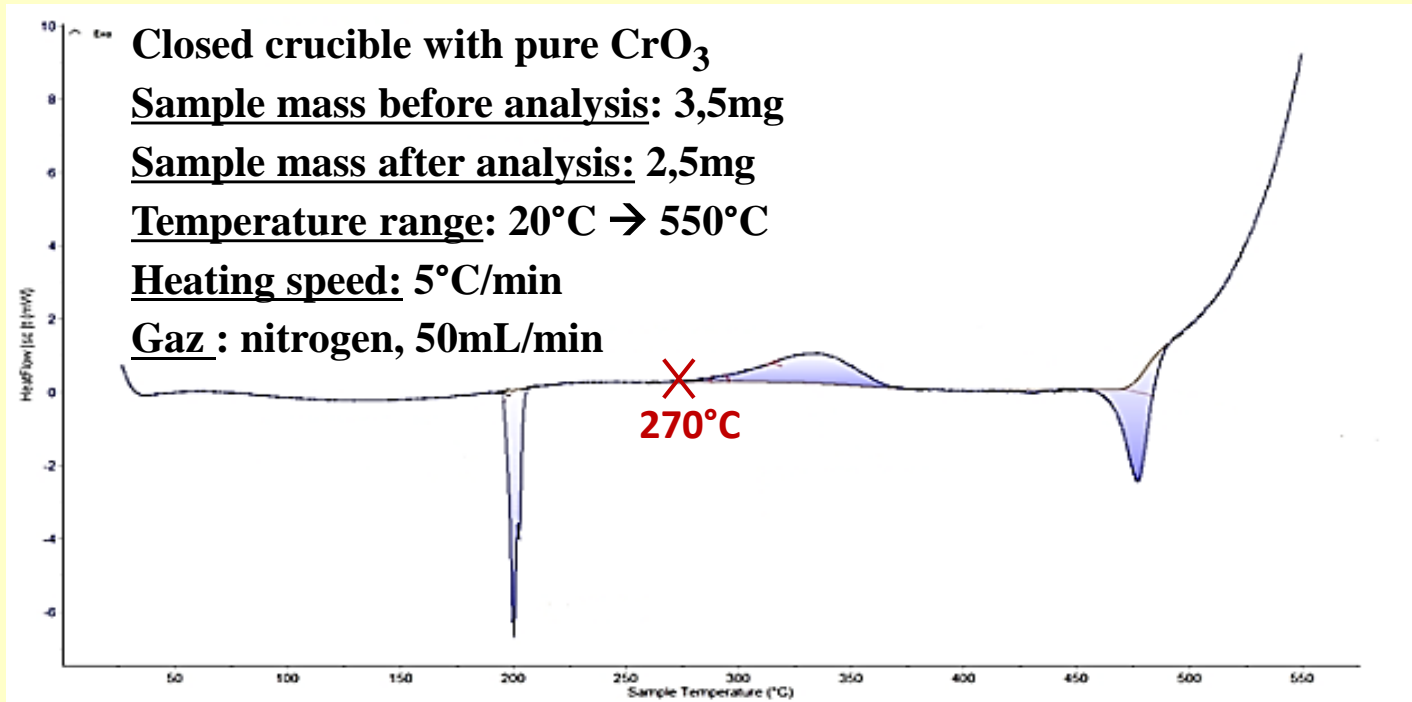
- Objectives:

1. Establishment of analysis methods
 - FT-IR spectroscopy
 - Thermal analysis
 - X-Ray diffraction (EN-MME-MM)
 - SEY measurements
2. Heat treatments at different temperatures.

Establishment of the analytical methods and parameters



- Differential Scanning Calorimetry

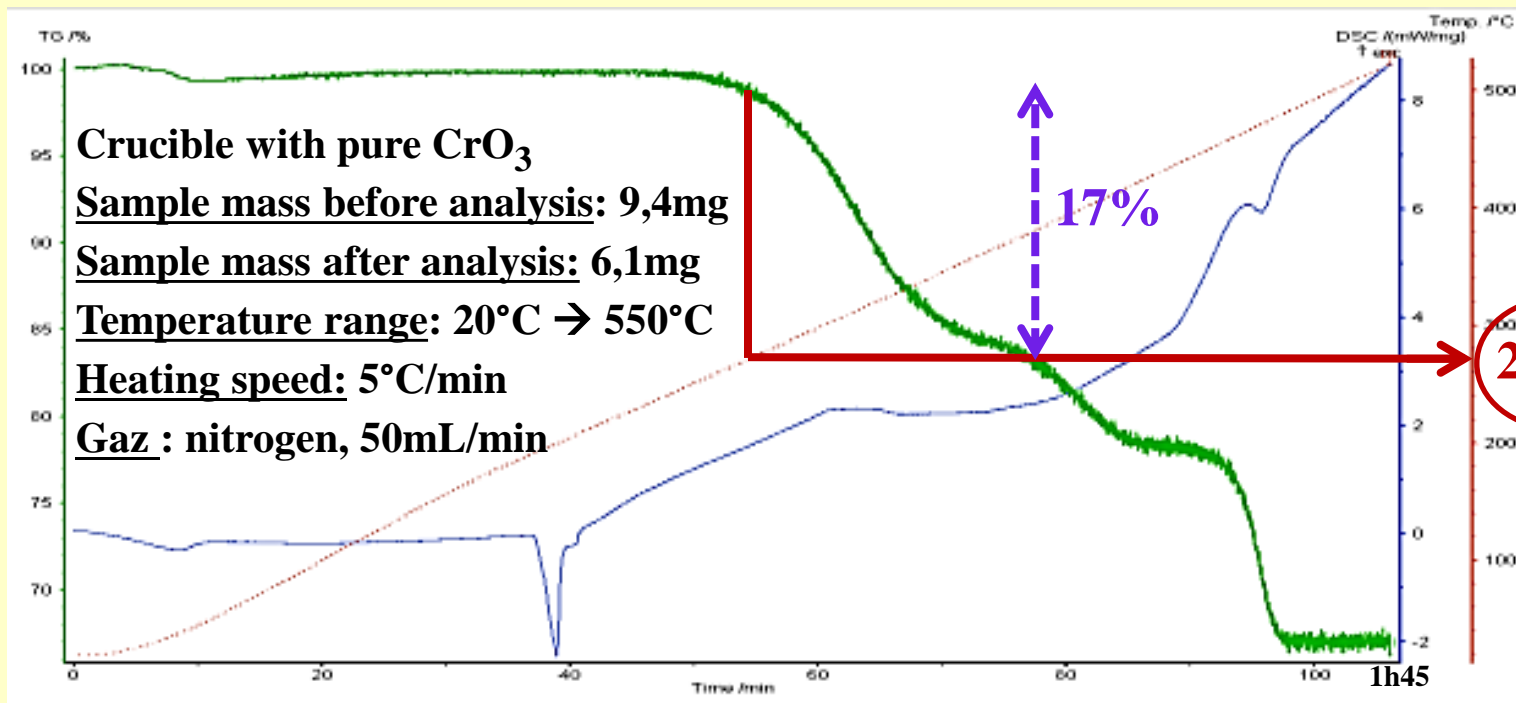
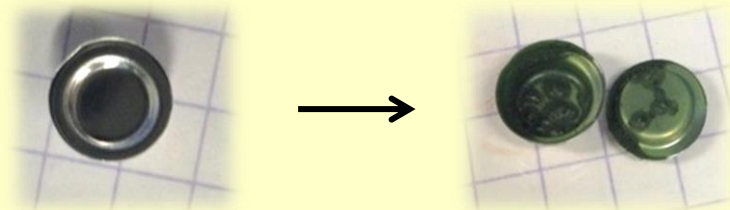


Total mass loss: 30% > 23% (theory: $4\text{CrO}_3 \rightarrow 2\text{Cr}_2\text{O}_3 + 3\text{O}_2$)

Evaporation?



- Thermogravimetry

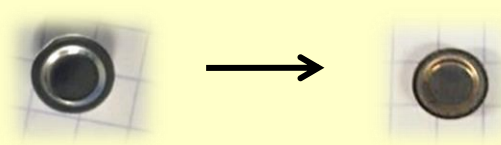
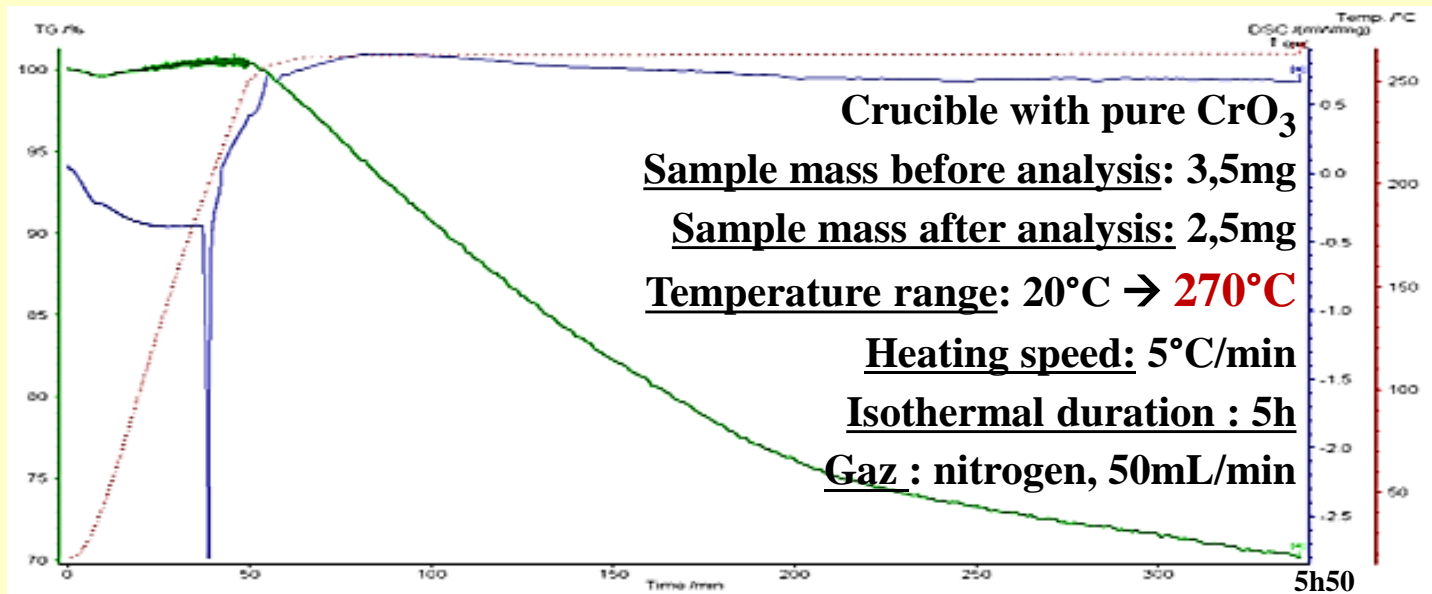


Total mass loss: 35% > 23%

Evaporation?

Mass loss (1st reaction): ≈17%

Thermal Analysis



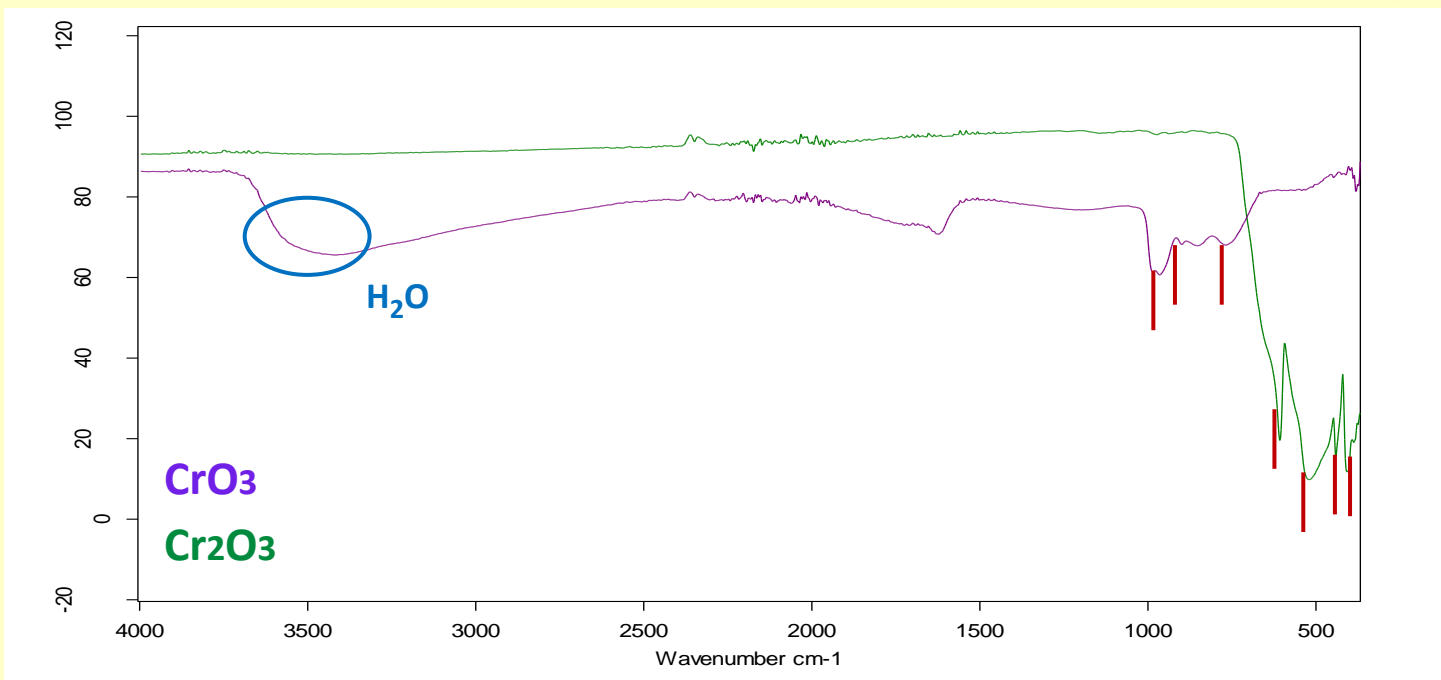
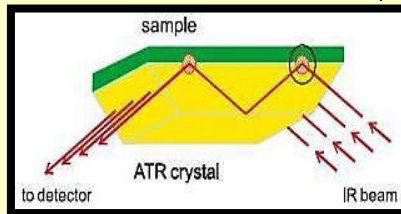
Total mass loss: 30% > 17% **Reaction + Evaporation?**

→ **Characterisation of the Cr(VI) thermal reduction.**

→ **Restart of the thermobalance.**



- Attenuated Total Reflectance (ATR)

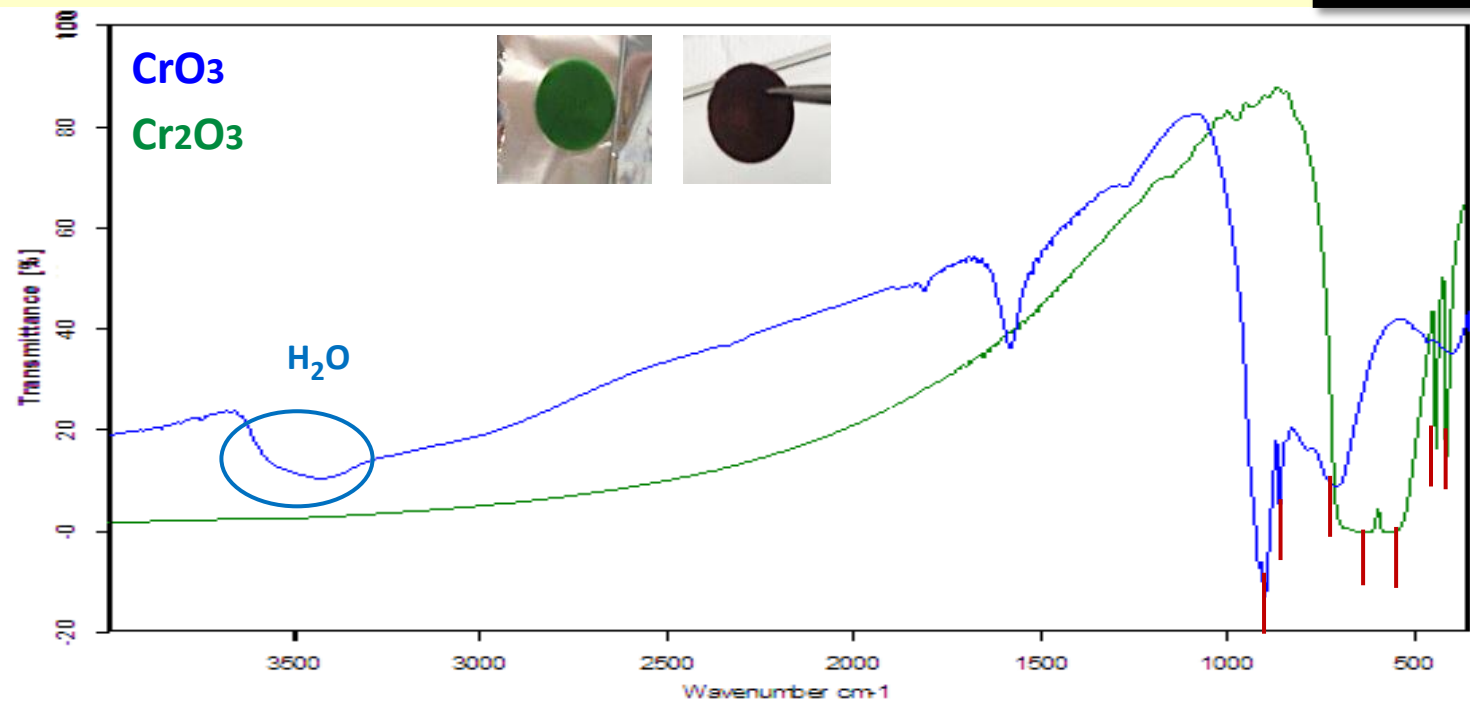
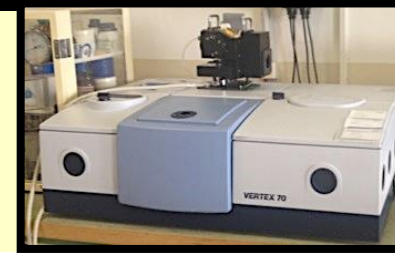


+ Fast, non-destructive.

- Unsuitable for in-situ analysis in vacuum chambers.



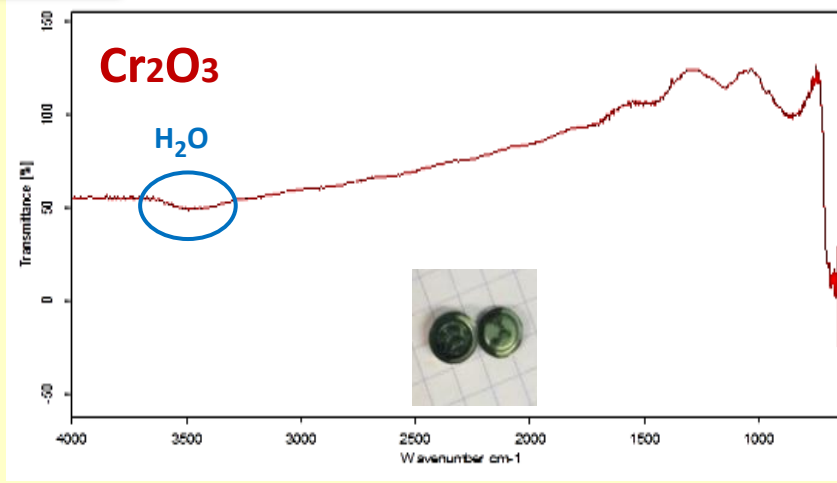
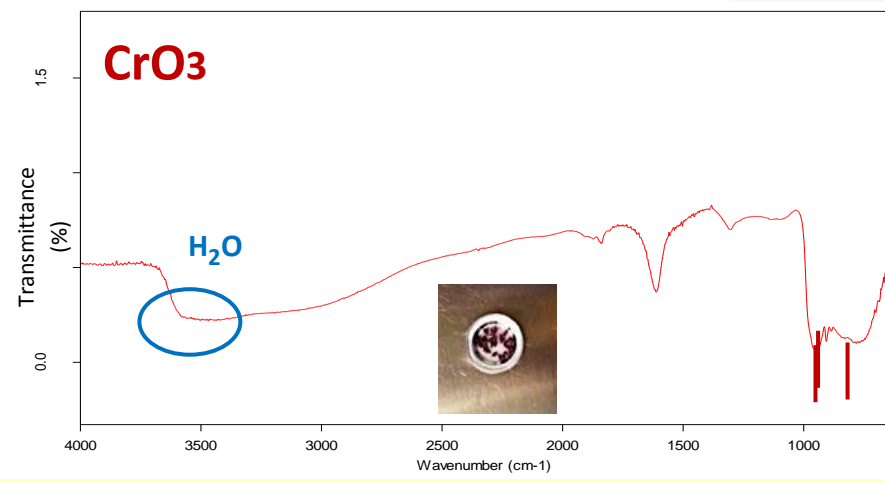
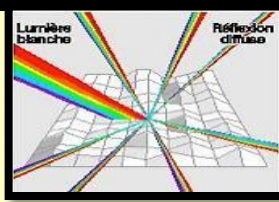
- Transmission



- + Suitable for in-situ analysis.
- Characterisation of the oxides.
- Samples storage (CrVI and KBr hygroscopic).

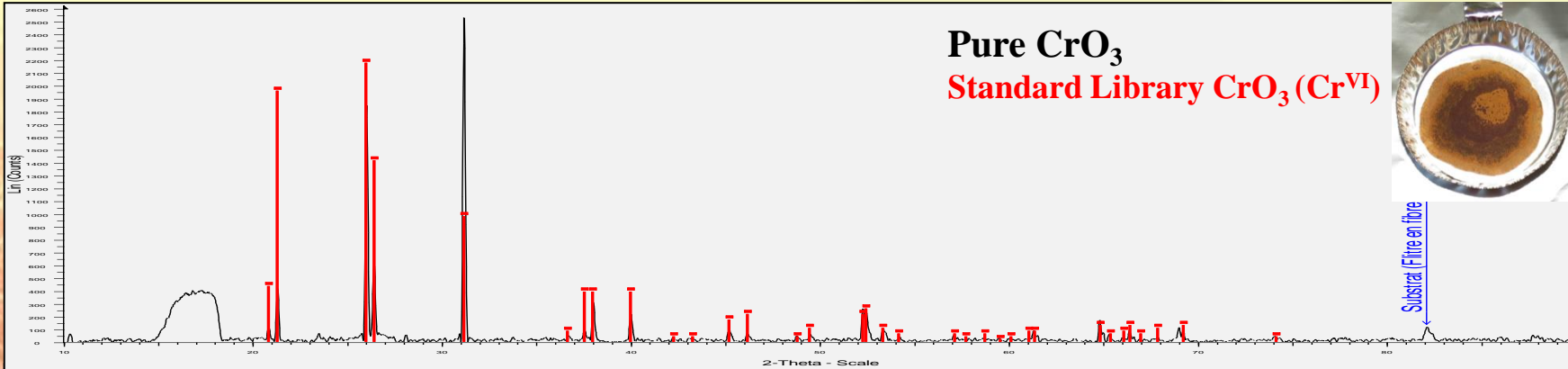
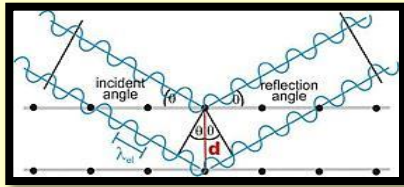


- Diffuse Reflectance

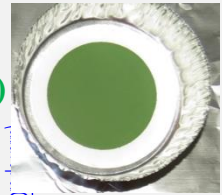
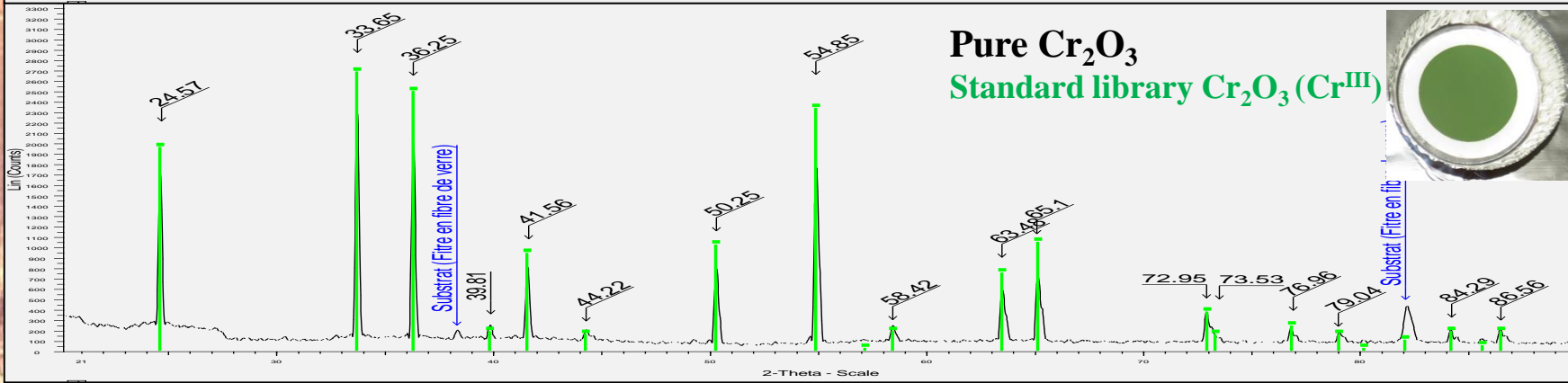


- + Suitable for analysis of residues in crucibles.
 - Characterisation of the oxides.
 - Apparatus limitation (minimum wavelength: 600cm⁻¹).
- Characterisation of the chromium oxides.**





Substrat (Fibre en fibre)



Substrat (Fibre en fibre)

(Floriane Leaux, Gonzalo Arnau Izquierdo (EN-MME-MM))

→ **Characterisation of the chromium oxides.**

SEY Measurements



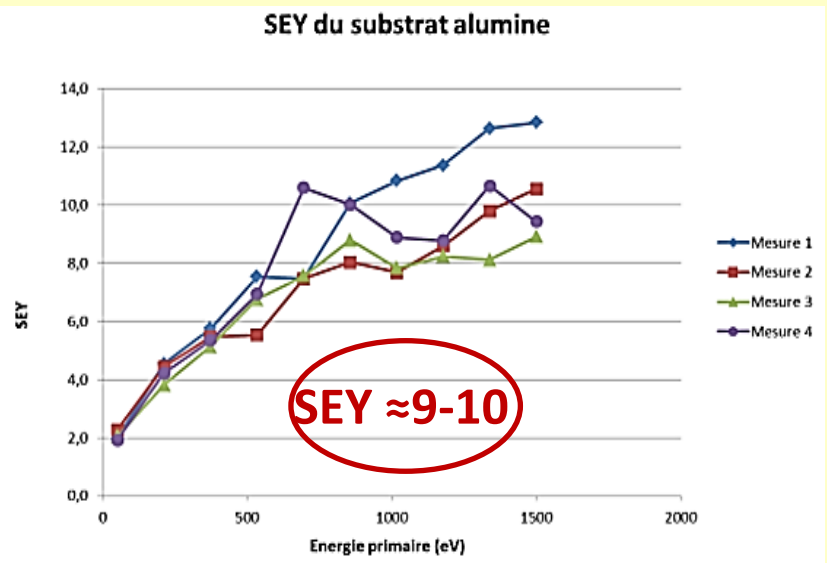
- Alumina



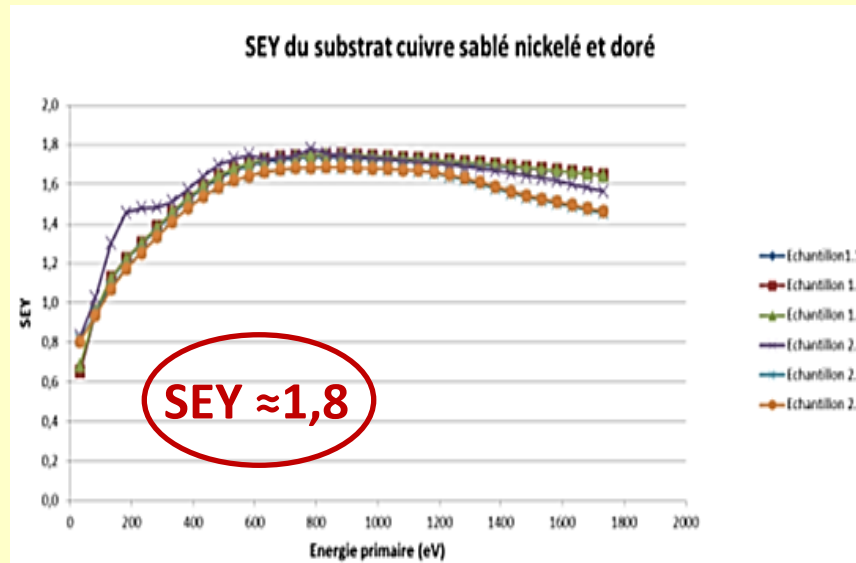
- Sand-blasted, nickel-coated and glided copper



SEY du substrat alumine



SEY du substrat cuivre sablé nickelé et doré

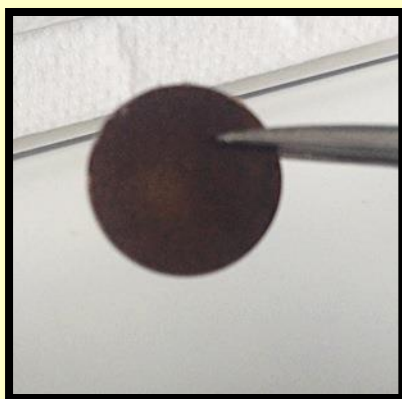


(Holger Neupert, Valentin Nistor (TE-VSC-SCC))

Heat treatments



$\text{CrO}_3(\text{aq})$ 10% (w/w) coating (Cr(VI))
 (1h dipping + 1h drying (120°C))



FT-IR spectroscopy



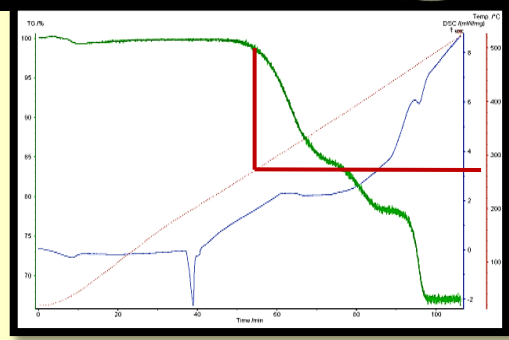
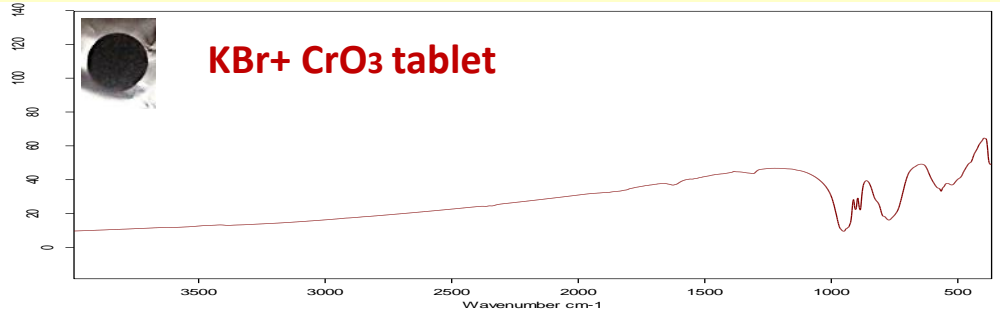
X-Ray diffraction



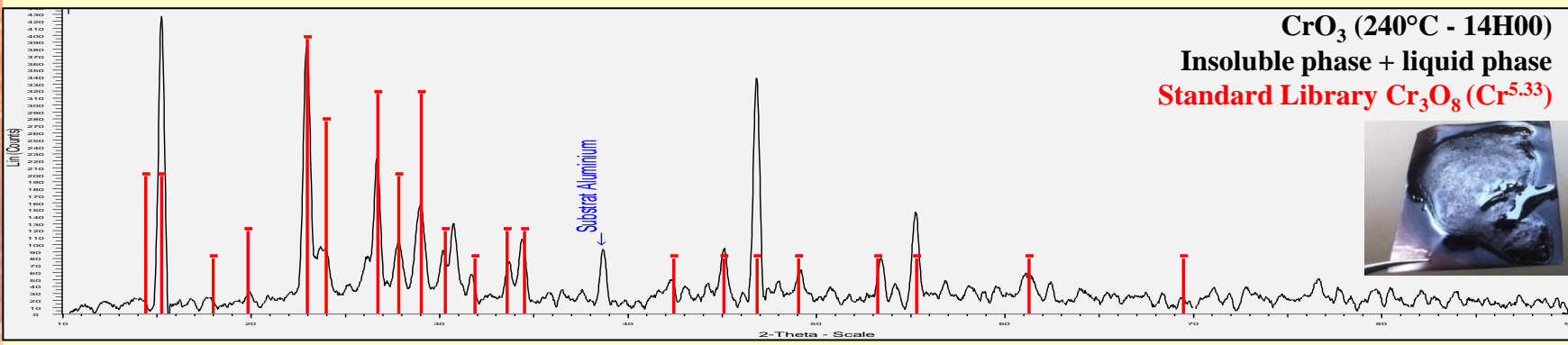
SEY measurements



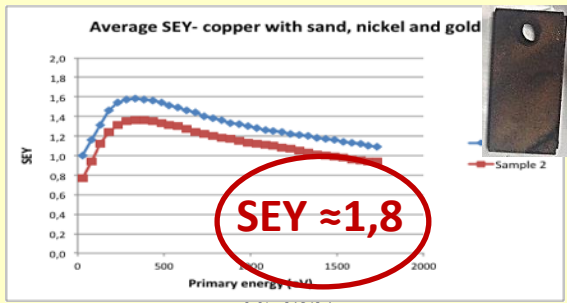
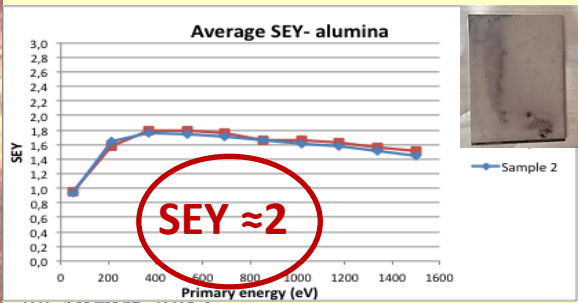
➤ FT-IR spectroscopy



➤ X-Ray diffraction (EN-MME-MM)



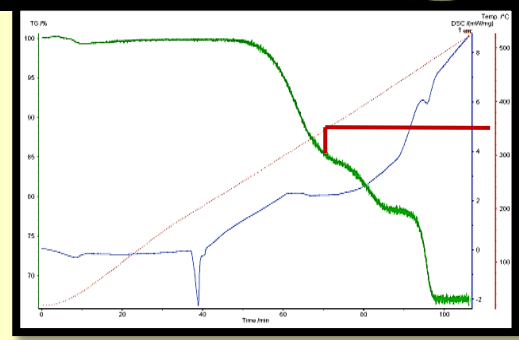
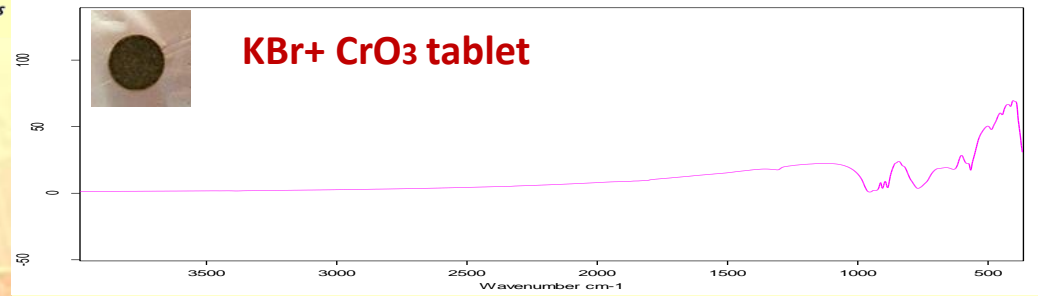
➤ SEY measurements



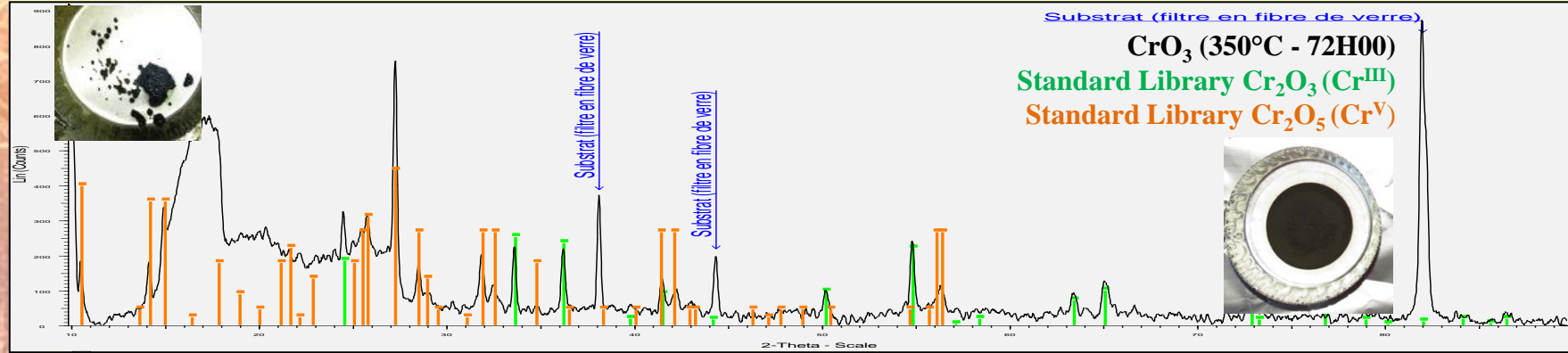
➔ **Decrease in SEY.**
 ➔ **Inapplicable solution (hygroscopic).**



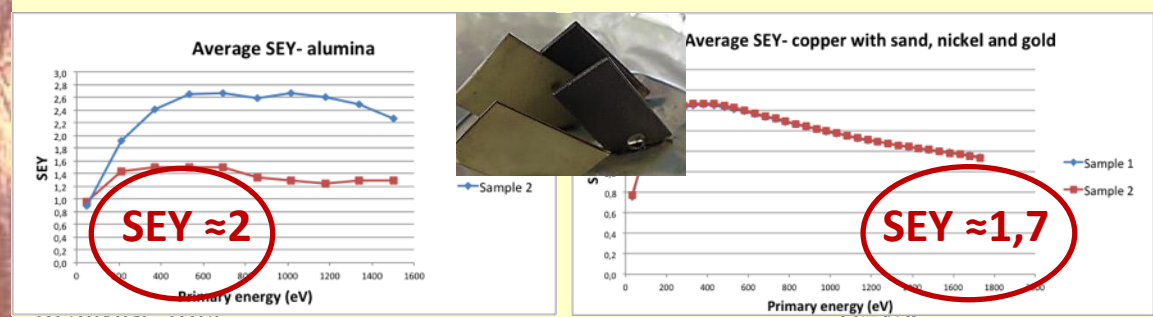
➤ FT-IR spectroscopy



➤ X-Ray diffraction (EN-MME-MM)



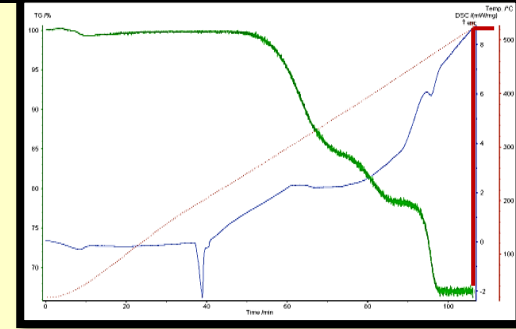
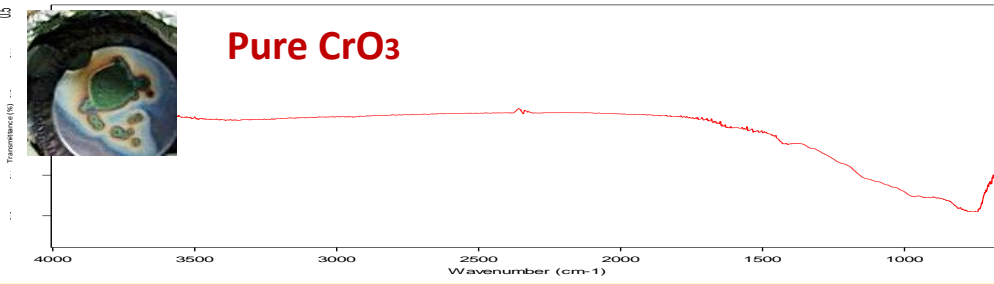
➤ SEY measurements



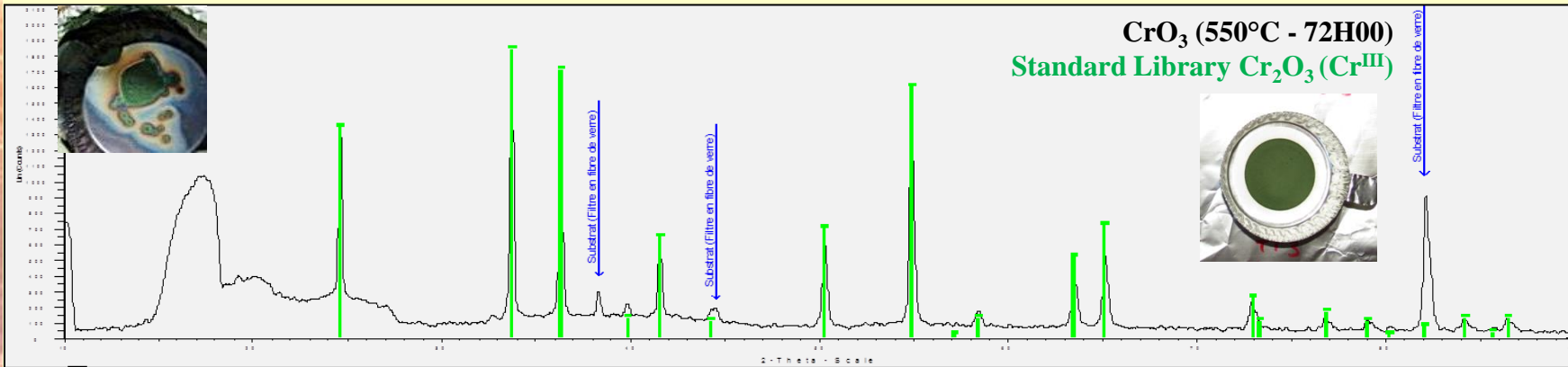
➔ Creation of Cr(III).
➔ Unstable solution.



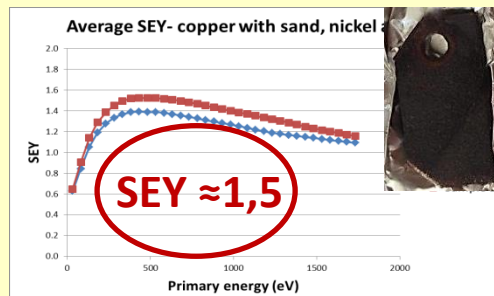
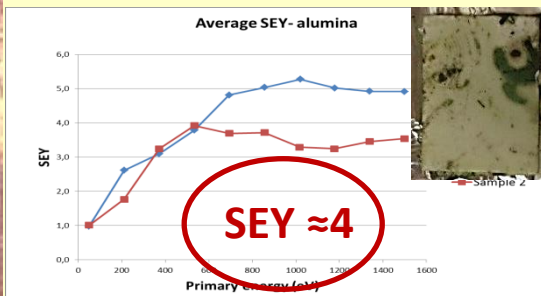
➤ FT-IR spectroscopy



➤ X-Ray diffraction (EN-MME-MM)



➤ SEY measurements



- ➔ Inhomogeneous coating.
- ➔ Presence of Cr(III).



- Analytical methods and parameters established.
- Several solutions to control coatings in the vacuum chambers.
- Cr(III) seems to be a stable solution.

Improvements:

- Study homogeneity of the coatings.
- Identify intermediary oxides, create a FT-IR data base.
- Study the influence of number of backouts for the Cr_2O_3 concentration.



- Paolo Chiggiato (TE-VSC)
- Mauro Taborelli (TE-VSC)
- Benoit Teissandier (TE-VSC)
- [EN-MME](#): F. Leaux, G. Arnau.
- Polymers Laboratory
- [TE-ABT](#): L. Ducimetiere, M. Barnes.
- [TE-VSC](#): C.Charvet, D. Letant-Delrieux, F. Fesquet, H. Neupert, L. Bardo, P. Maurin, R. Setnescu, R. Leber, V. Nistor.

THANK YOU FOR YOUR ATTENTION