SURFACE TREATMENTS AT CERN

FLORENT FESQUET
PIERRE MAURIN

Outline

1. Infrastructure dedicated to surface treatment

2. Cleaning and degreasing of parts for UHV applications

3. The electrolytic deposition at CERN

   - Why doing a deposition?
   - Preparation of stainless steel prior to electroplating
   - Silver plating on aluminum alloys

4. Improvement and development

Infrastructure dedicated to surface treatment

Building 867 (Site Prevessin)

Infrastructure dedicated to surface treatment

Surface cleaning of large parts intended for UHV applications

Preparation copper OFE

Tube Max IV

Preparation Stainless steel

Tank DTL Linac 4

Preparation aluminum alloy

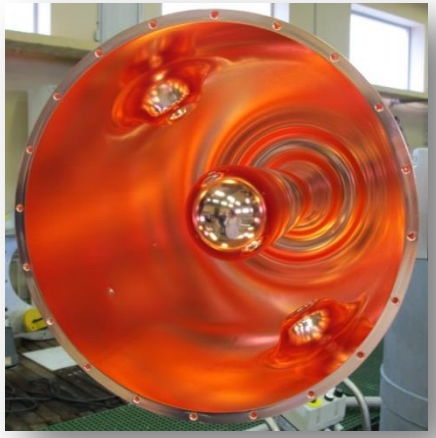
Chambre VJ ATLAS

Building 118 (Site Meyrin)

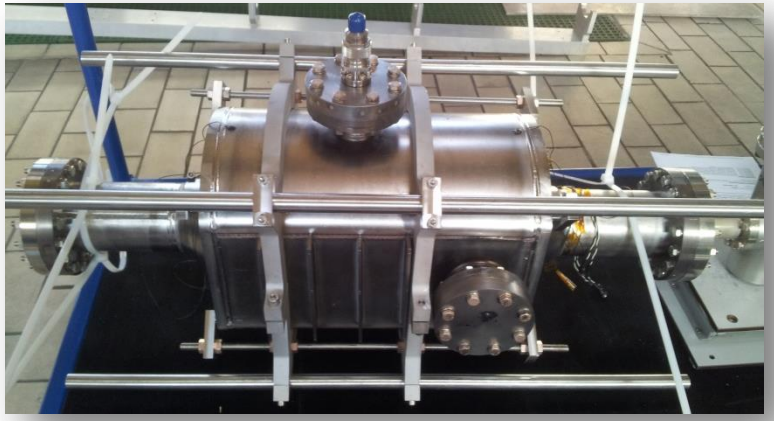
Infrastructure dedicated to surface treatment

Surface preparation: chemical polishing and electropolishing of copper and Nobium

Chemical polishing of copper and niombium



Cavity HIE- ISOLDE



Superconducting cavity CRAB

Electropolishing of Niobium and copper

Superconducting cavity 5 cells Niobium for SPL (700 MHz)

Copper test cavity (1300 MHz)

Building 102 (Site Meyrin)

Infrastructure dedicated to surface treatment

Surface preparation of large parts intended for UHV applications

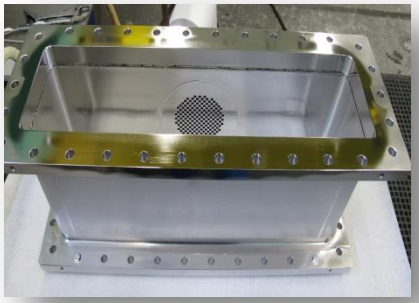
Ceramic

BGV Aluminum chamber

Disque of copper cavity PIMS

Connector stainless steel

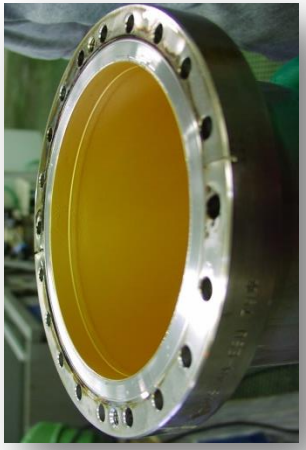
Electropolishing stainless steel



Guide-Onde LINAC4 stainless steel

Electrolytic deposition on stainless steel, copper and aluminum

Gold electroplating

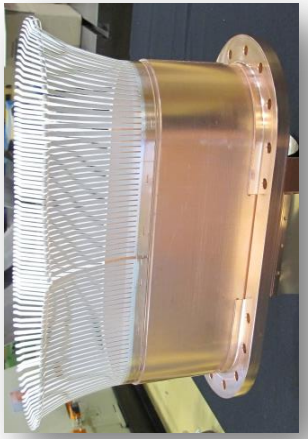


Chambre LINAC

Copper electroplating

Couvercle Tank LINAC 4

Silver electroplating



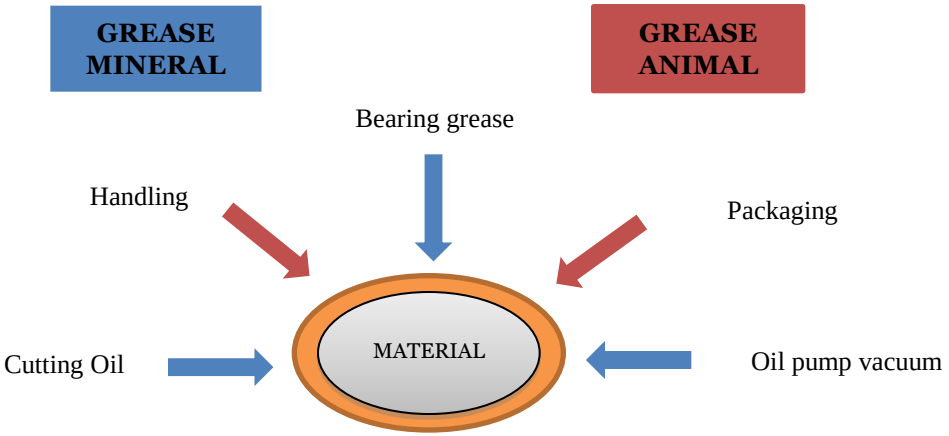
Doigts de contact écran TDI

Rhodium electroplating

Tube de transition LHCVSR

Cleaning and degreasing of parts for UHV applications

Why cleaning a part ?



Eliminate grease which pollute surface.

Eliminate the solid particles present in bold (i.e. particles machining, graphite residuals…

Reduction of vacuum outgasing.

Prepare the surface before vacuum deposition or electrolytic plating.

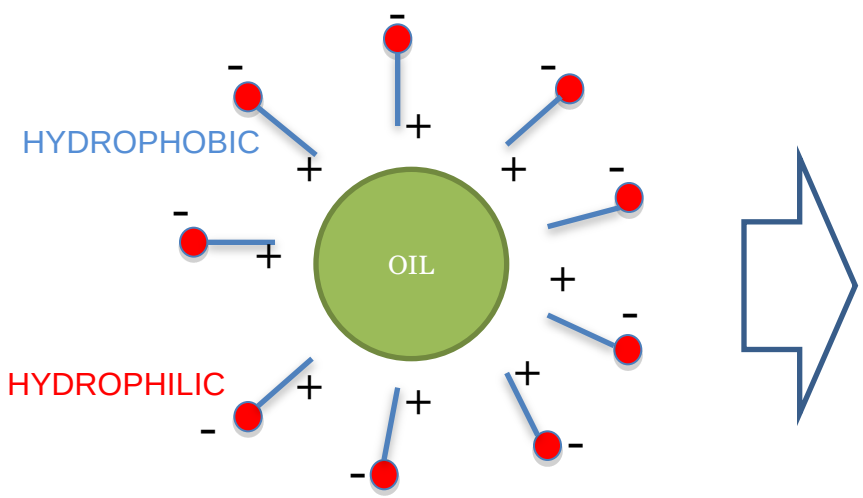
THE ALCALINE ELEMENTS

Take off the grease stains and « trap » with surfactants

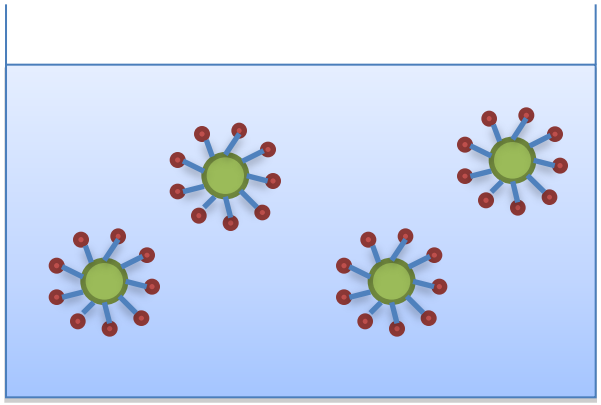
Sodium silicate : Increase the wettability and etching inhibition.
Phosphates : Sequestrant power and enhancement of the action of surfactants.

THE SURFACTANTS (wetting)

The surfactants lower the surface tension and capture of oil drops.



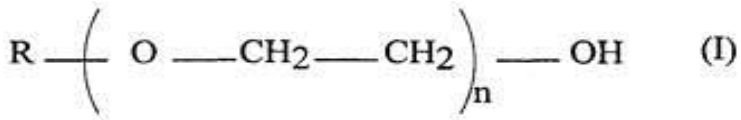
Action of an anionic surfactant



Stable emulsion

Solvent Machine

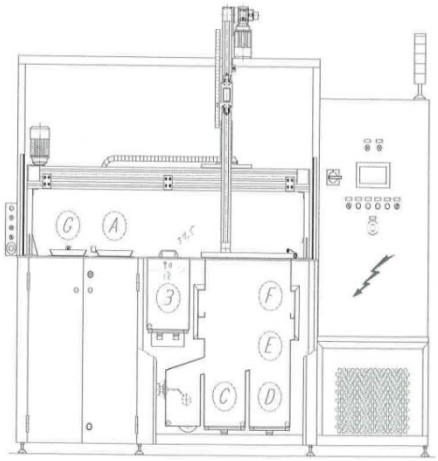
Halogenated / hydrocarbon mixture

example of carbon chain : 

Porous materials

Ferrite | **Tungsten** | **Silicone valve** | **Delicate parts** | **Ceramic**

Evaporation of the solvent



Operating range:

A. Loading
B. Precleaning cosolvent activated U.S
C. Cleaning solvent activated U.S
D. Rinse solvent activated U.S
E. Rinse solvent vapors
F. Drying
G. Unloading

Machine solvent

The 3 steps of the mechanism of action of ultrasound :

1- The cavitation effect

Appearance of bubbles between the substrates and greases

2- The effect of pressure

Compression of the air bubble

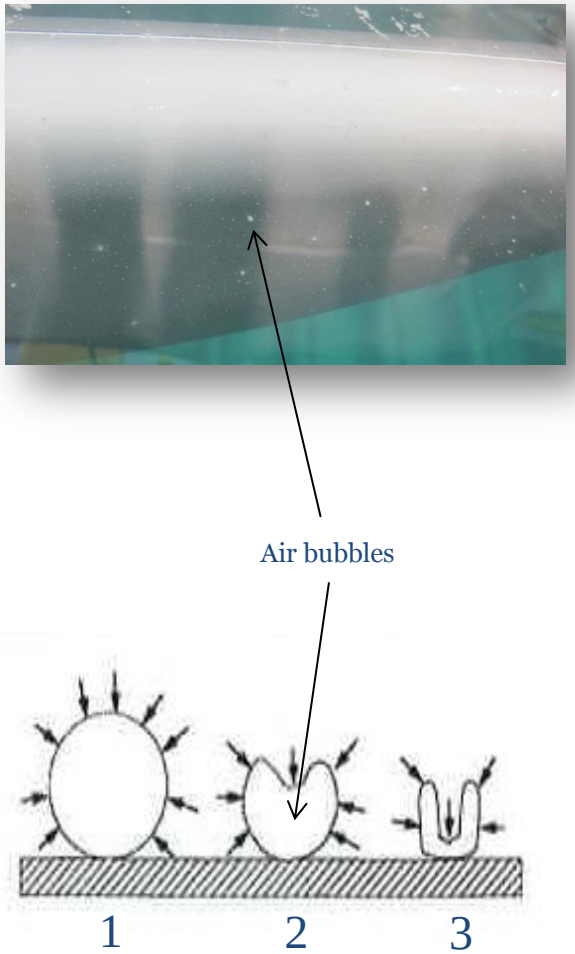
3- Implosion of air bubbles

Drop the grease from the surface

Frequencies used in chemical degreasing :

20 kHz for the copper and 40 kHz for the aluminum

IMPROVES considerably degreasing power

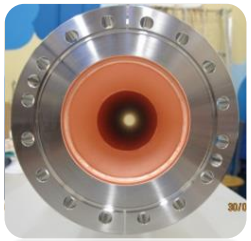


Asymmetric implosion of a cavitation bubble near a solid surface

Electrolytic deposition at CERN

Why doing a deposition?

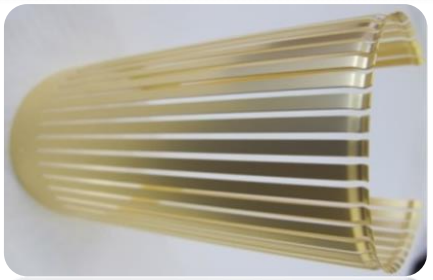
COPPER PLATING :
Thermal conductivity and electrical conductivity (reduction of impedance), RF


Chamber BGV

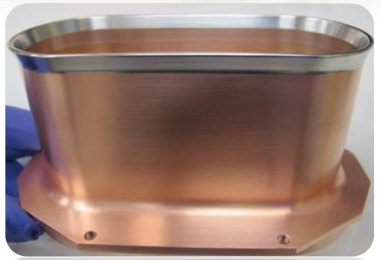
SILVER PLATING:
Electrical conductivity (electrical contact)
Low friction, seizure


Contact fingers

GOLD PLATING :
Chemically stable in the atmosphere and electrical conductivity


Contact fingers

RHODIUM PLATING:
Resistant to high temperature and very high hardness (prevents galling vacuum with the couple Rh-Ag) and seizure


Tube transition

Preparation of stainless steel before deposition

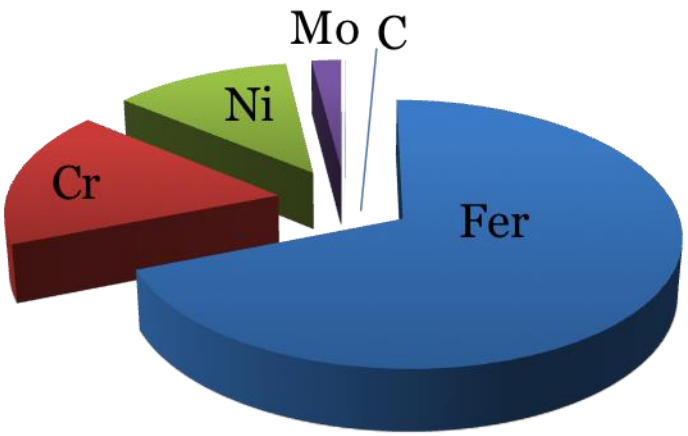
Characteristic of stainless steel

Standardization of a stainless steel

Ex : Stainless Steel 316 LN

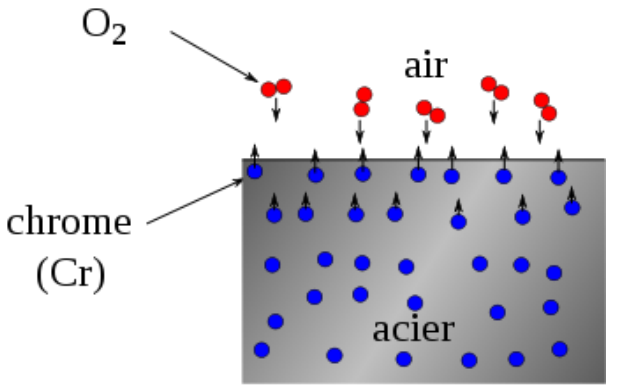
(X2CrNiMo 17-12)

X2 : Alloy steel containing 0,02 % carbon
Cr : 16 – 18 % Chrome
Ni : 11 – 14 % Nickel
Mo : 2 – 3 % Molybdène



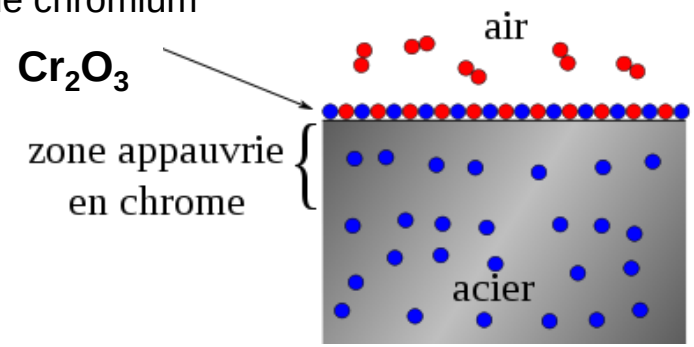
Mechanism passivating a stainless steel

$$4\,Cr + 3\,O_2 \longrightarrow 2\,Cr_2O_3$$



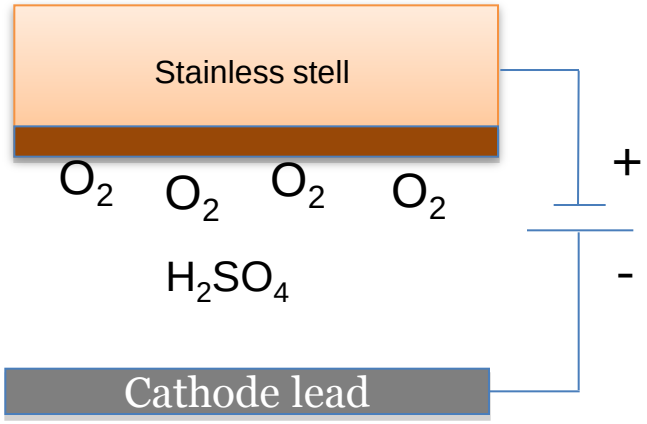
Oxide chromium

Cr_2O_3



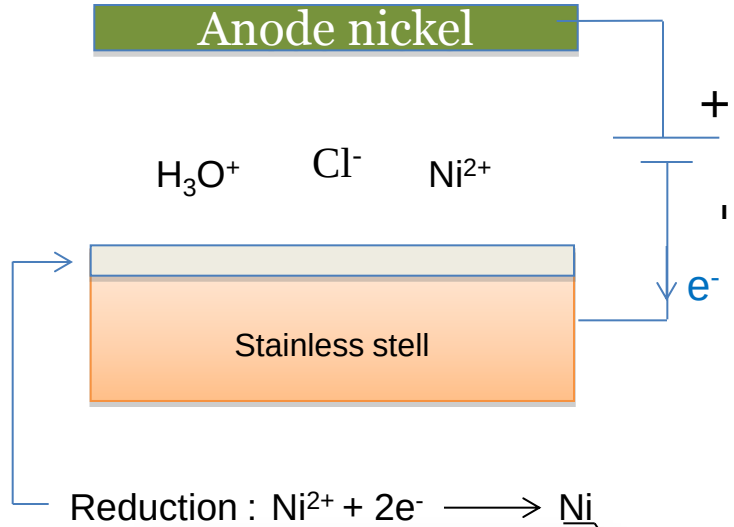
SULFURIC INVERSION

Elimination of the passive layer



WOOD NICKEL

As bonding layer



Reduction : $Ni^{2+} + 2e^- \longrightarrow Ni$

<u>Applications</u> : Undercoat prior to electroplating

Nickel plating chamber BELLOW

Preparation of stainless steel prior to electroplating

1. **Substrate type and geometry**.

2. **Study of assembly**: Nature of deposit, anode assembly, masking and electrical contacts.

3. **Mounting**: positioning of the anode, contact verification and assembly.

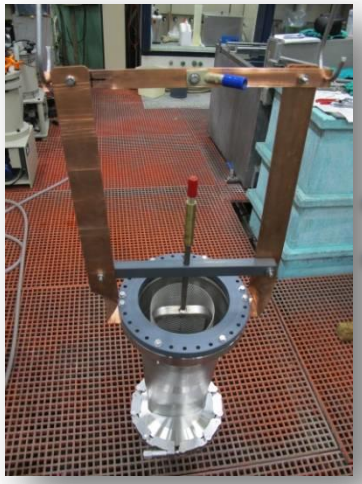
4. **Treatments according to operative range**: monitoring the operative range.

5. **Control of deposit**: Aspect, deposit thickness.

1

2

3



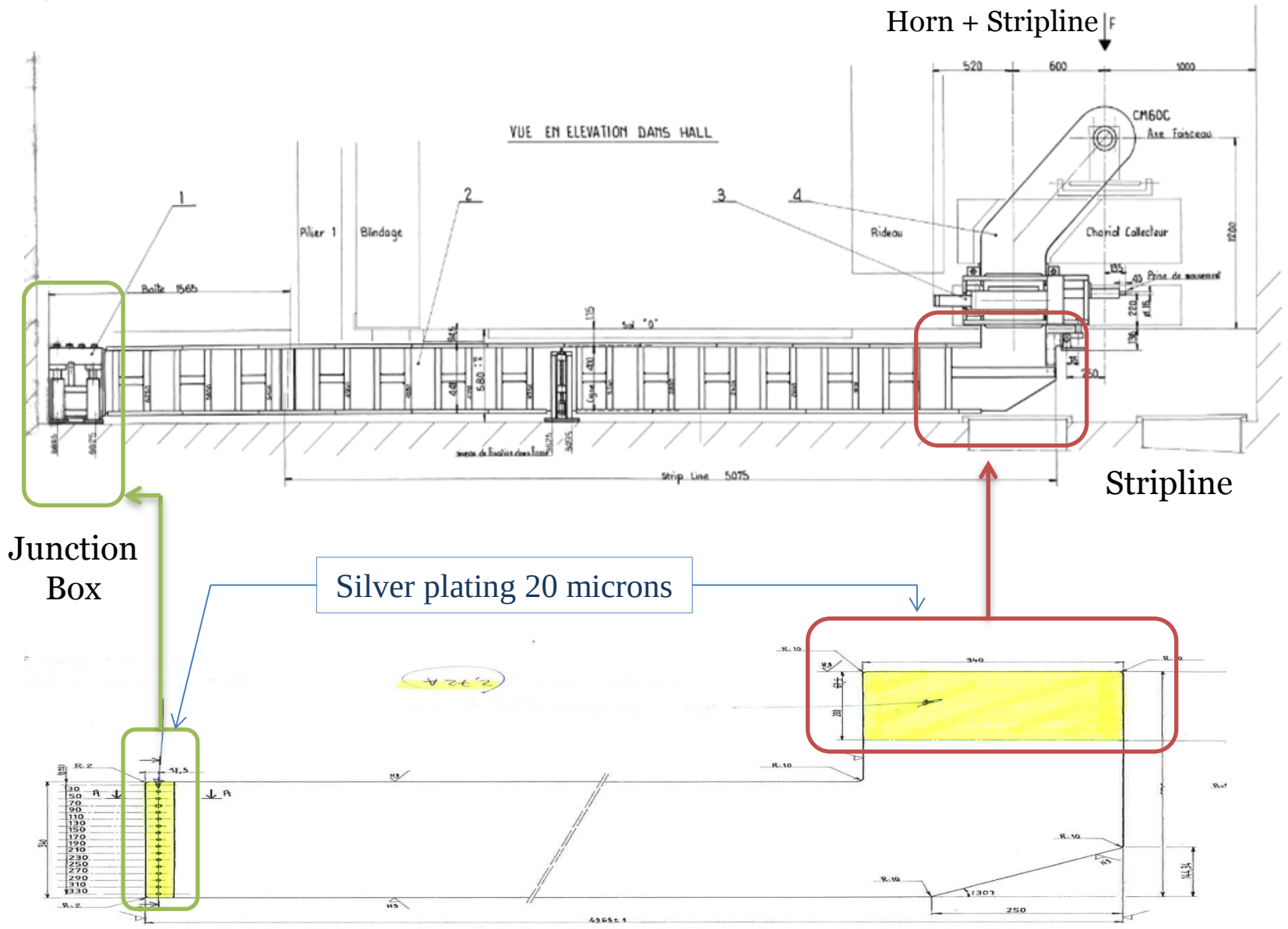
4

5

Electrolytic silver plating on aluminum alloys

Contact Strip and Strip Line (Experiment AD)

Electrolytic silver coating on aluminum alloys



Electrolytic silver coating on aluminum alloys

Old procedure

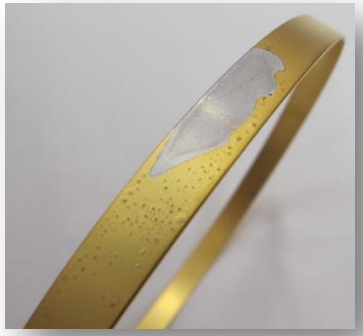
Surface preparation
Degreasing / Etching

Step 5 Nickel-Zinc plating

Step 6 Nickel Sulfamate

Step 7 Pre Silver plating

Step 8 Silver plating



Pell-off gold coating

New procedure

Surface preparation
Degreasing / Etching

Step 5 Double-Zincate

Step 6 Nickel Sulfamate

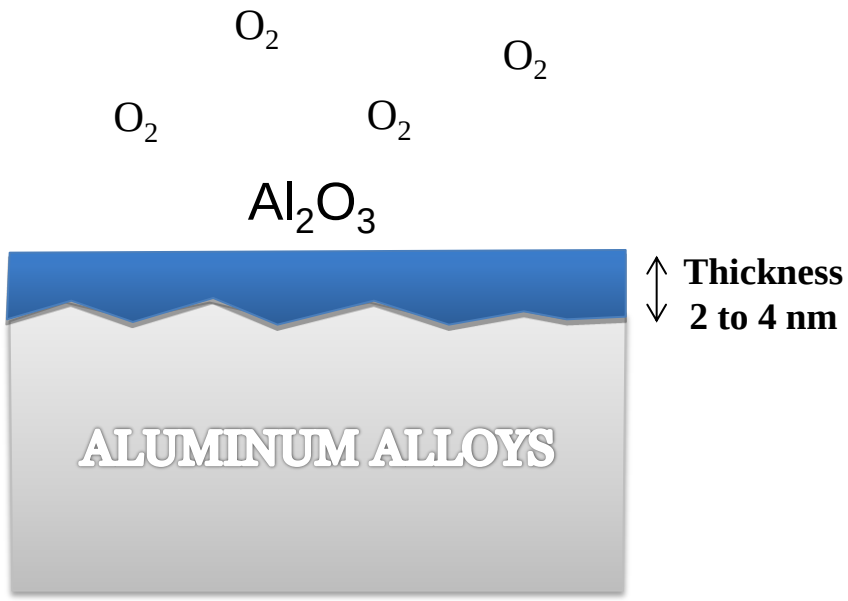
Step 7 Pre Silver plating

Step 8 Silver plating

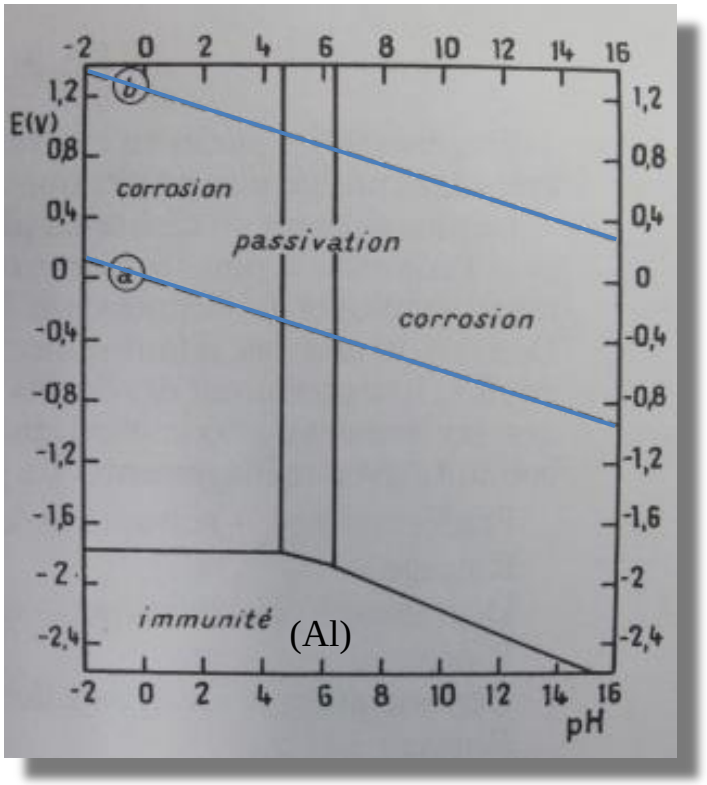
OPTIBOND CONCENTRATE

COVENTYA

$$4Al + 3O_2 \longrightarrow 2Al_2O_3$$



Forming a thin oxide layer of alumina



Equilibrium diagrams voltage-pH
Aluminum-water system at 25° C

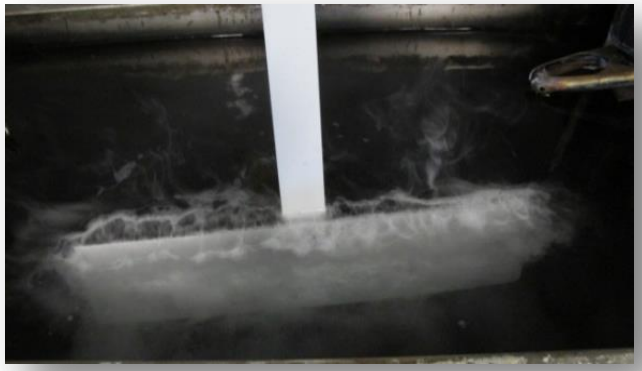
Area of water stability thermodynymique

ALUMINUM IS AMPHOTERIC METAL

Step 3 : Sodium Stripping

Sodium hydroxide at 40°C

$$Al + 2NaOH + 2H_2O \longrightarrow 2NaAlO_2 + 3H_2\uparrow$$



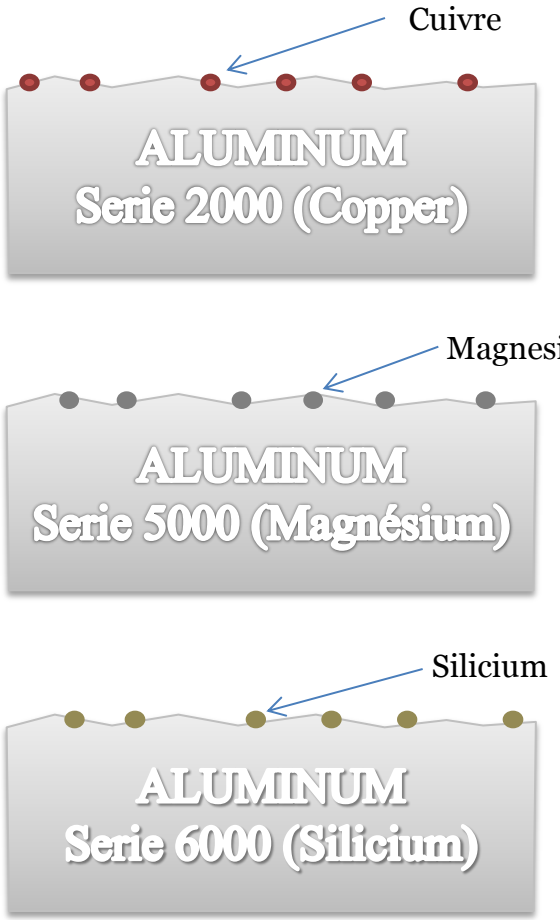
Dissolution of alumina

Nitric acid

Nitric acid
+
Sulfuric acid

Nitric acid
+
Fluorhydric acid

Step 4 : whitening



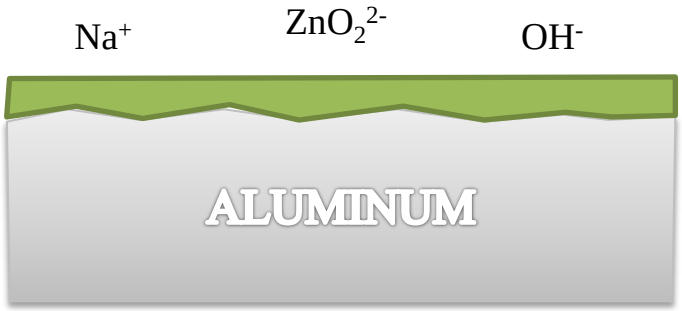
Dissolution of alloying elements

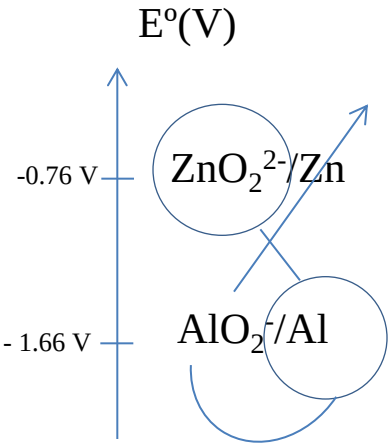
Step 5 : Double Zinc plating

After stripping reformation of native alumina layer

ZINCATE

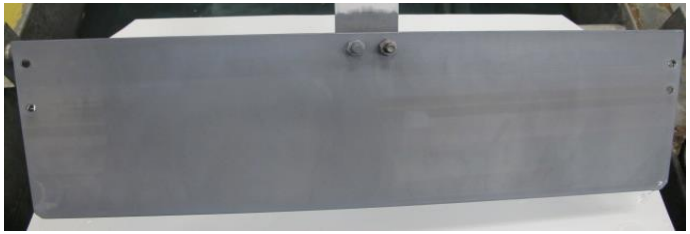
ZnO
+
NaOH





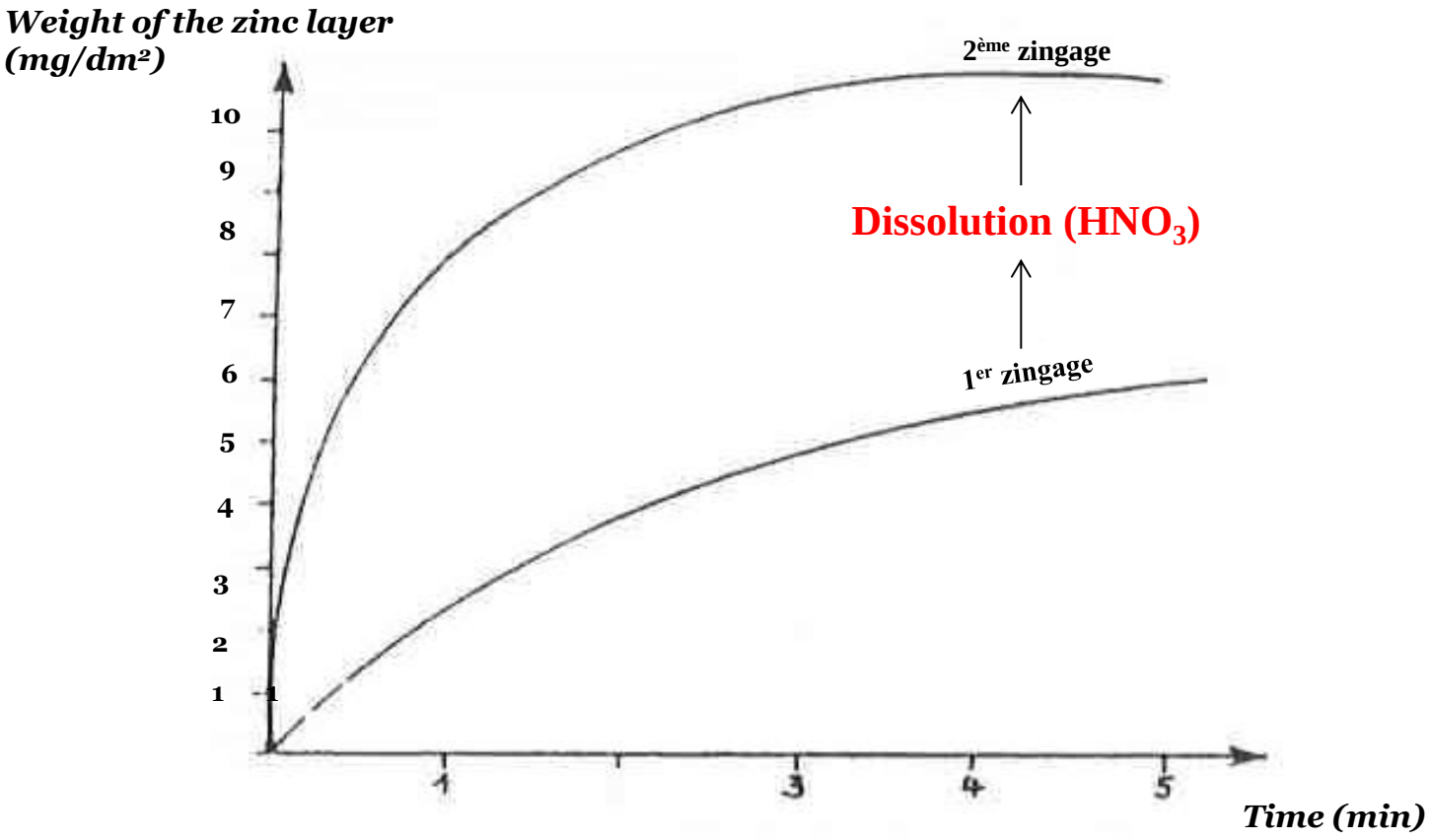
$$2Al + 3ZnO_2^{2-} + 2H_2O \rightleftharpoons 2AlO_2^- + 3Zn + 4OH^-$$

Formation of a chemical deposition by zinc displacement



Few nanometer thickness

Evolution of coating weight between 1st and the 2nd Zincate



Provides a thinner deposit with a more compact structure

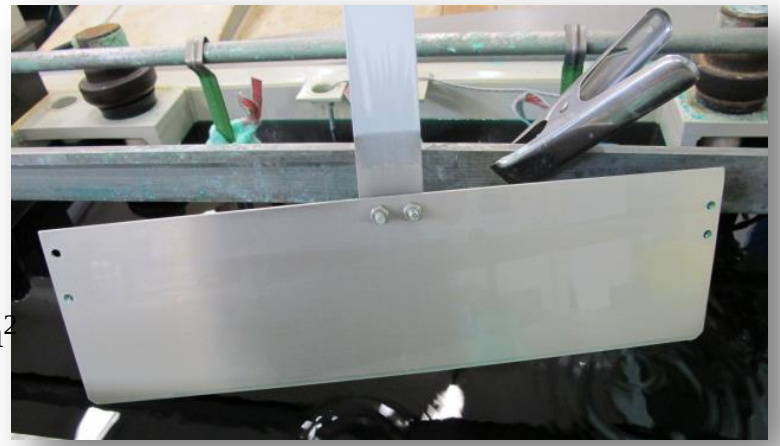
Step 6 : Nickel Sulfamate

Sublayer Nickel sulfamate electrolyte (dip the piece under current)

Good covering power and deposition constraints

Parameters :

pH	**5.5**
Temperature :	50°C
Currents density :	2 - 16 A/dm^2
Deposition rate :	0.5 µm/min to 10 A/dm^2

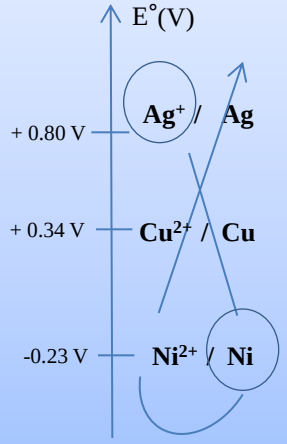


Deposit Nickel sulfamate

Electrolytic silver coating on aluminum alloys

Step 7 :Silver plating

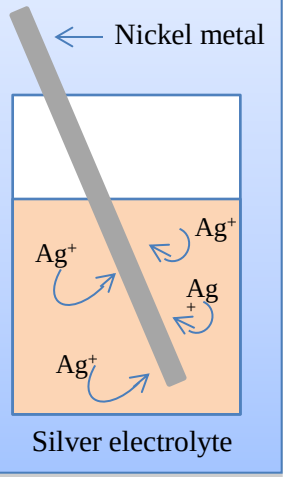
Moving powdery silver plating on a nickel coating



Reduction $Ag^+ + 1e \rightleftharpoons Ag$

Oxydation $Ni \rightleftharpoons Ni^{2+} + 2e$

Reaction sheet $2Ag^+ + Ni \rightleftharpoons 2Ag + Ni^{2+}$

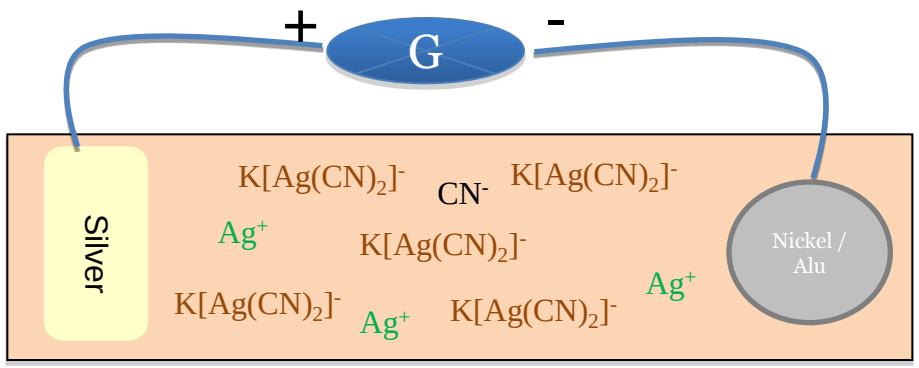


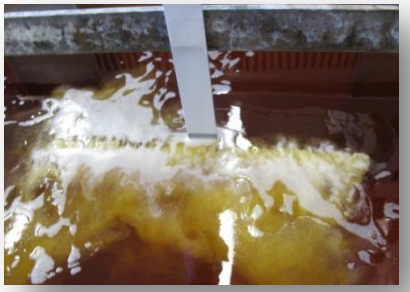
Pre-Silvering : $AgCN + KCN \longrightarrow K[Ag(CN)_2]$ **Double silver cyanide (complex)**

Double silver cyanide enables the lowering of the potential difference between the metal surface (nickel) and silver in solution and prevents the displacement deposition.

Formulation
[AgCN] : 4 g/L
[KCN] : 90 g/L

Concentration
Ag^+ free low





Pre-silvering

Silver plating

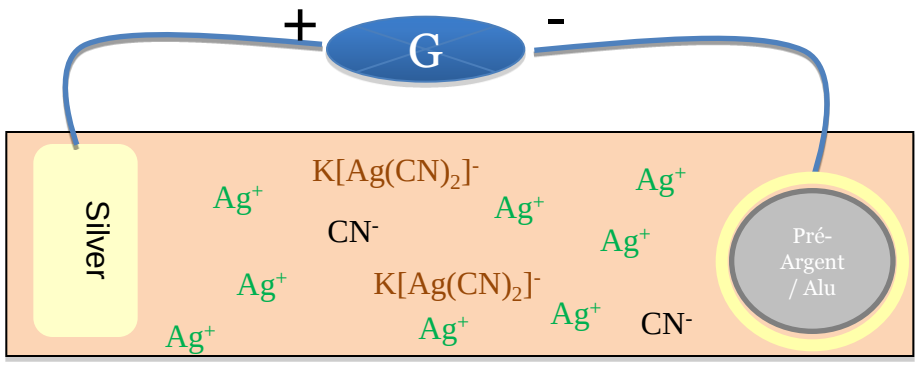
Silvering :

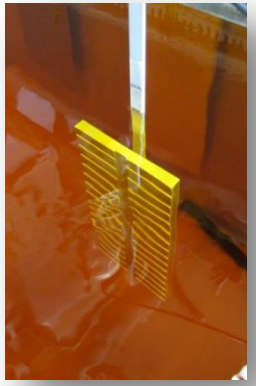
$$AgCN + KCN \longrightarrow K[Ag(CN)_2]$$

Double silver cyanide (complex)

Formulation
[AgCN] : 37 g/L
[KCN] : 130 g/L

Concentration
Ag^+ free
high

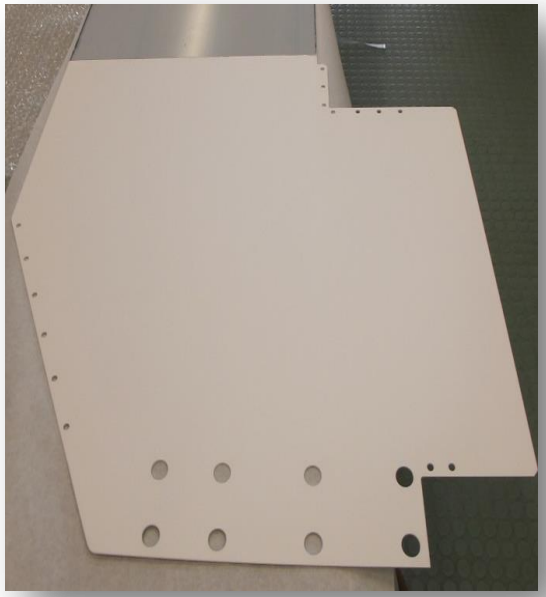




Silvering Contact Streap (AD)

The silver in a uniform power distribution and allows to obtain a deposit until 30 microns

Strip-line AD



Improvement and development

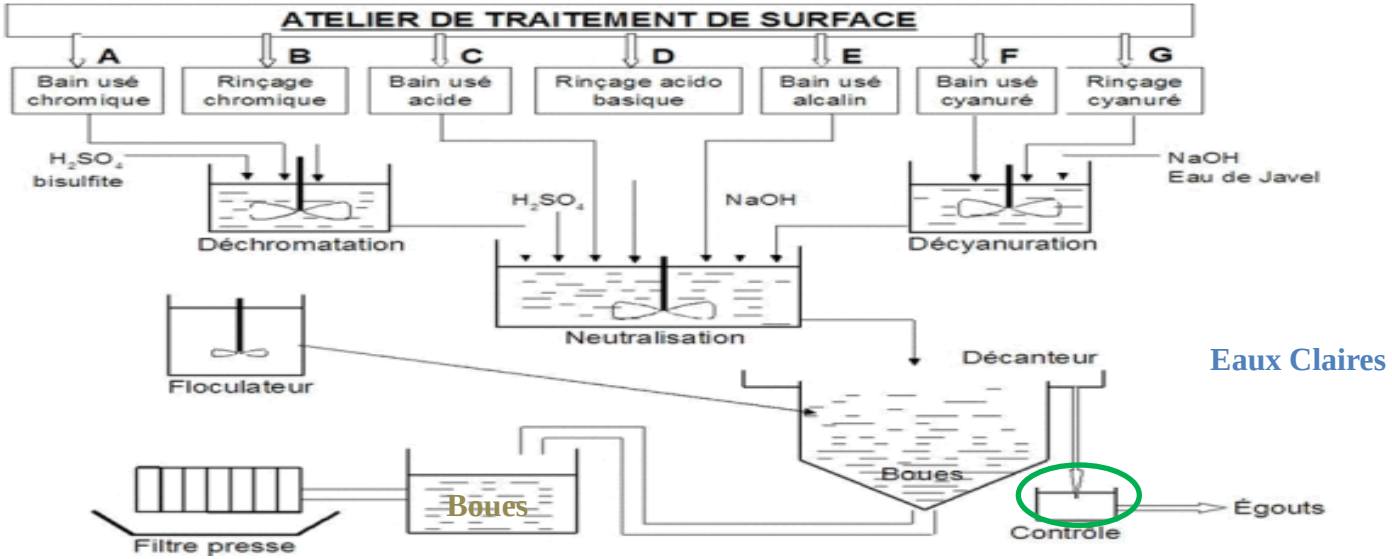
Analysis and water treatment plant

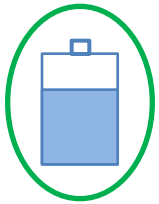
Workshop 118

Workshop 102

Effluent

Effluent



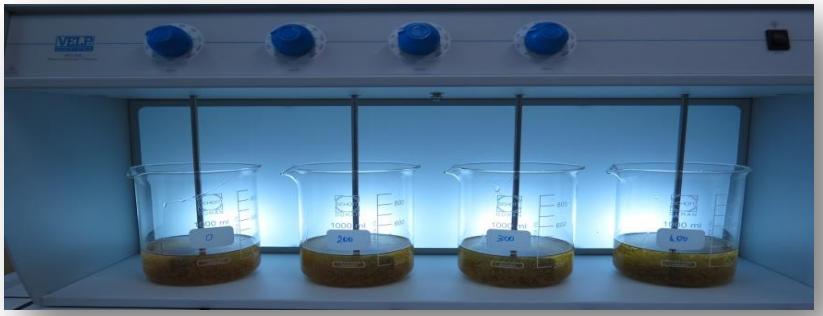


Spectromètre d'Emission Optique **(ICP-OES)**

Optimization of the treatment plant

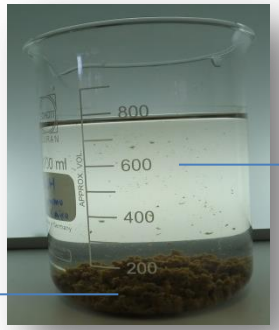
Following overruns on copper water discharges :

- Contact providers specialized in water treatment product
- Purchase of a test bench (Jar-test)
- Testing of different organic precipitants



Essai en Jar-test de précipitant en neutralisation

- Supernatant by ICP analysis and determination of the most appropriate methods to our effluent.



Effluents after decantation

Metal hydroxide sludge

Analysis of the supernatant

Optical Emission Spectrometer **(ICP-OES)**

Qualification of new processes for copper chemical degreasing

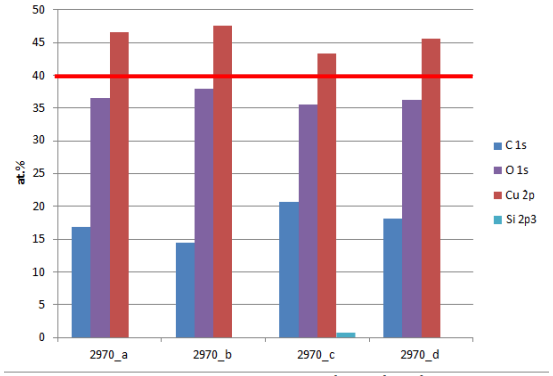


Figure 1: Concentrations atomiques relatives de surface

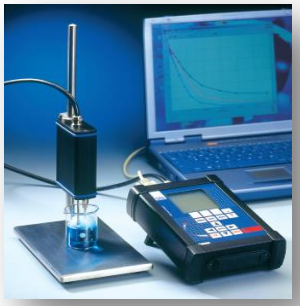
- Contact suppliers specialized in surface treatment (new processes)
- Contamination of samples of copper and chemical degreasing samples according to procedure

https://edms.cern.ch/document/997363/3

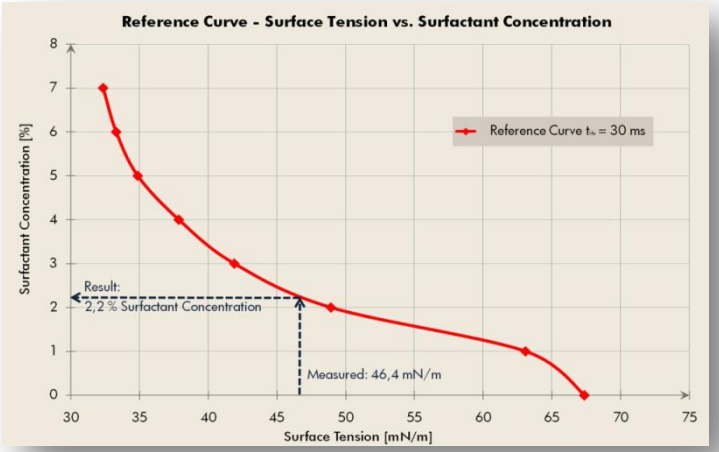
- *Qualification of processes by measuring the residual carbon surface (XPS)*

Analysis methods surfactants in treatment baths surface

Monitoring the concentration of surfactants in chemical degreasing baths by measuring surface tension.



science line t60



Improvement and Development (Workshop)

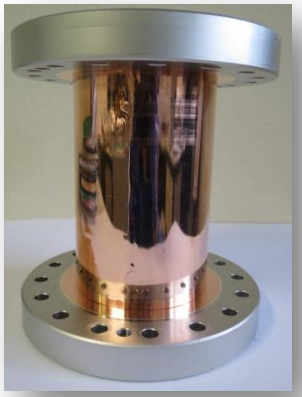
Etching for the thinning of the sample 3 (LHCb Velo) :

Establishment of a procedure for etching chambers VELO (LHCb)

Echantillon 3 (LHCb Velo)

Achieving chamber with integrated copper electroforming layer NEG:

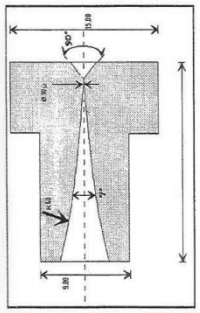
Dévelopment of new sequences copper plating by pulsed current



Tube électroformé

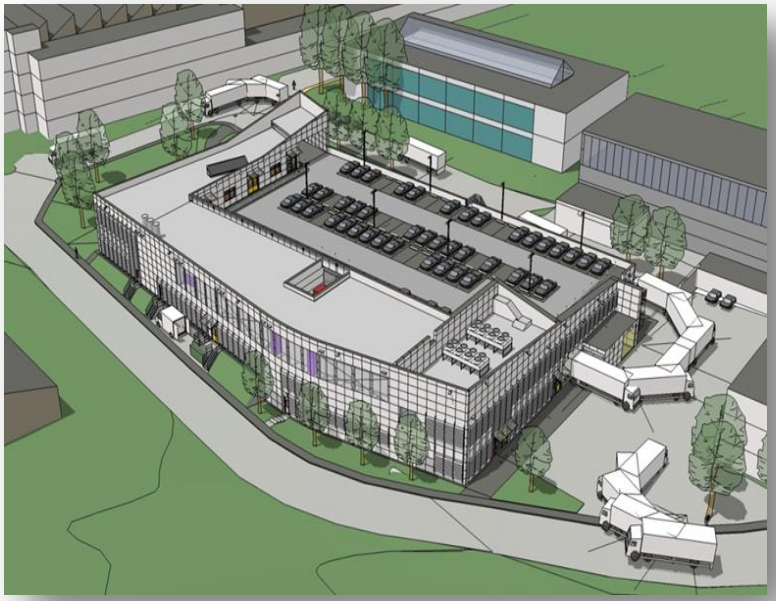
Fabrication by electroforming nozzle gas diffusion :

Electroformages of micronozzles for injecting a hydrogen micro-jet in the CERN accelerators



Microbuses

Building new surface treatment (107) planned for 2016



THANK YOU FOR YOUR ATTENTION!