Aluminum and sulfur Impurities in Electropolishing Baths.

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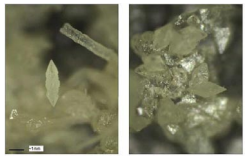
SRF

Introduction :

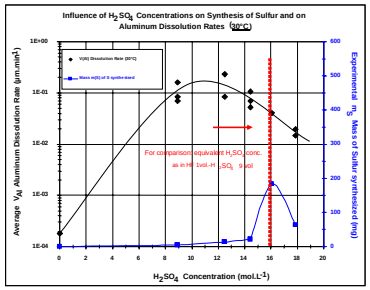
- This study highlights the impurities formation in Electropolishing bath (mixture of sulfuric and hydrofluoric acids) when aluminum is chosen as cathode material. Such impurities could partially explain the performances' discrepancies observed on electropolished cavities.
- These products might be aluminum derivatives, sulfur S and hydrogen sulfide H_2S. We have distinguished two cases: with or without applied voltage. Furthermore, parameters such as temperature and acid concentrations are also taken into account.



Sulfur crystals gathered from an aged EP solution

1) Without bias :

Aluminum corrosion, S and H_2S production



Al corrosion in pure H_2SO_4

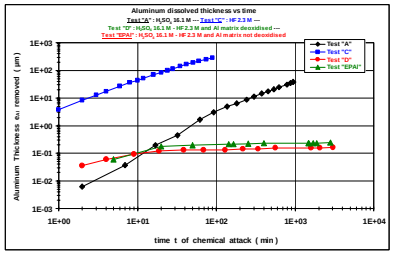
- *High content of H_2SO_4 promotes S production => decrease $[H_2SO_4]$?*
- *Al corrosion is higher at lower $[H_2SO_4]$,* ***=> ± acceptable !?****

Al corrosion in pure HF

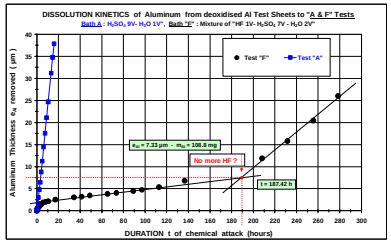
- *HF concentrations tested: between 2,29 et 22,87 mol/L*
- *$2Al + 6HF \longrightarrow 2AlF_3 + 6H_2$*
- *Al resulting removal rate: 4.15 et 7.60 µm/min*

** Al F_3 is fairly soluble in water : can be rinsed easily*

Inhibition of Aluminum corrosion



- *Corrosion is far more lower in mixtures of than in separated acids*
- *Corrosion and S production increase back when HF content is decreased (due to the reaction with niobium and evaporation)*



Test #	Volumic composition				Sulfur synthesis	
	HF	H_2SO_4	H_2O ad[d]	Total Volume	Time (minutes)	Sulfur mass (mg)
A	0 V	9 V	1V	10 V	955	184,2
E	0.1 V	9 V	0.9 V	10 V	5930	9.9
F	1 V	7 V	2 V	10 V	16705	29,1

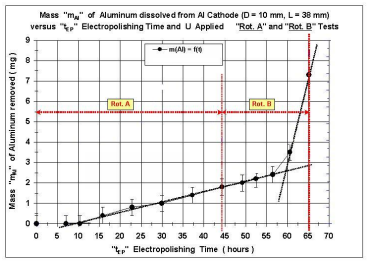
High H_2SO_4 content

Higher content of H_2O, lower content ofh SO_4

2) With bias:

Reduction of SO_4^{2-} at the cathode

Test\ sulfur production	M_S calculated (compared to Al dissol[n]) (mg)	Measured M_s (mg)
Rotanodes A	1,07	21,1
Rotanodes B	3,27	113,8



- slow but continuous corrosion of Al
- high quantity of S
- =>additional formation of sulfur at cathode :

$$SO_4^{2-} + 8H^+ + 6e^- \longrightarrow S + 4H_2O$$

NB : this ½ reaction is probably potential dependent and independent from cathode material

- Cathode corrosion under bias !?
- Corrosion becomes important when [HF] has decreased due to reaction + evaporation

Rinsing issues

Aluminum

- aluminum corrosion cannot be prevented, but it is rather slow.
- unlike Nb or Pt cathode, it doesn't form metallic particles but keeps in the form of Al^{3+} salts.
- should be easily rinsed with water, but needs to be checked

Sulfur

- It is a critical issue : S is not soluble in water
- deposits on cavities surface + sealing off tubes, filters…
- rinsing works in ethanol, but not very effective
- it is very effective in chloroform, but safety issues

Fluorine content

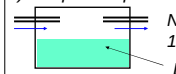
- generation of S is strongly dependent on [HF]
- HF is decreasing vs. time (reaction with Nb, evaporation..)

=> need for monitoring F^-, SO_4^{2-} content vs time :

ionic chromatography

- relationship between gloss decrease and F^- consumption ?
- what is the exact amount of available F^- ions (vs *FSO_3^-*) in the solution ?

1) F^- upon evaporation

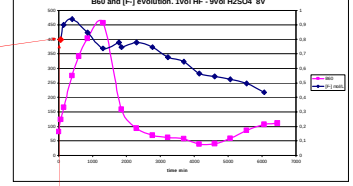


N_2, 3-6 l/mn, > 1week

EP 1-9 @ 30°C

=> Changes in [HF] < dispersion of results

2) F^- upon electropolishing



*Apparently : 1mole F^- + 1 mole SO_4^{2-} *missing*

=> $HF + H_2SO_4 \rightarrow HFSO_3 + H_2O$????

** SO_4^{2-} not shown*

Conclusion: sulfur generation and cathode corrosion are two issues to be considered with care. Increasing the HF content of EP solutions seems to improve both the EP solution lifetime (see ThP02 poster) and to reduce the sulfur generation. Rinsing procedure needs to be studied with care and to be improved.

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