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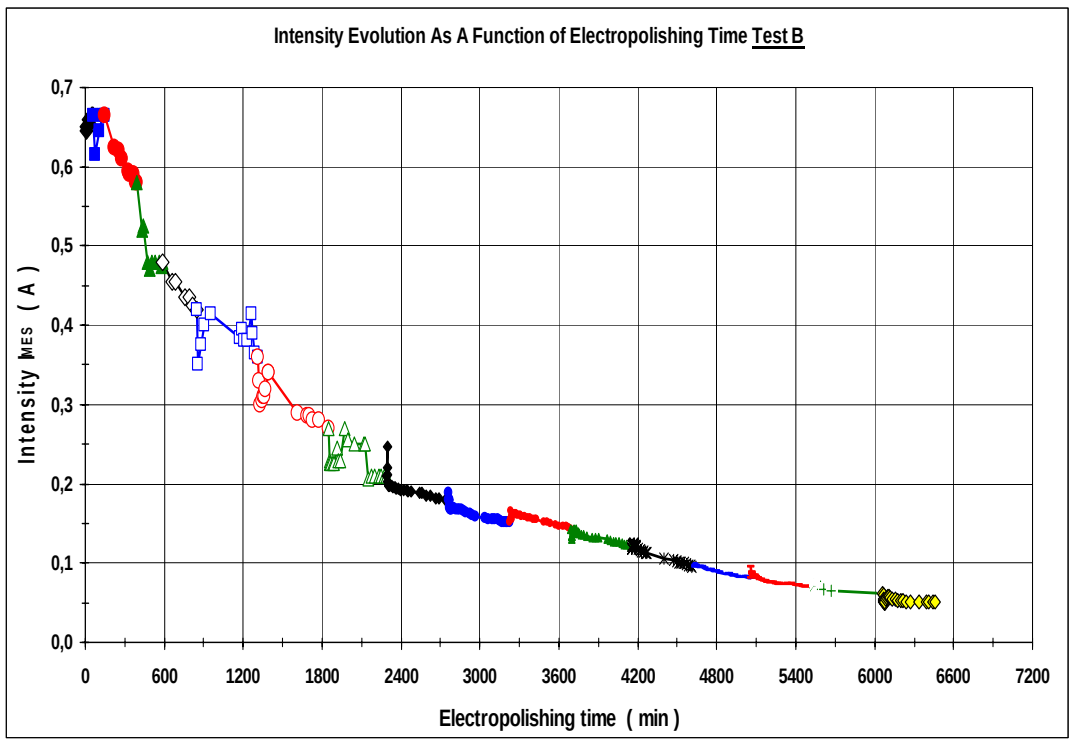
Optimization of Electropolishing

Main issues :

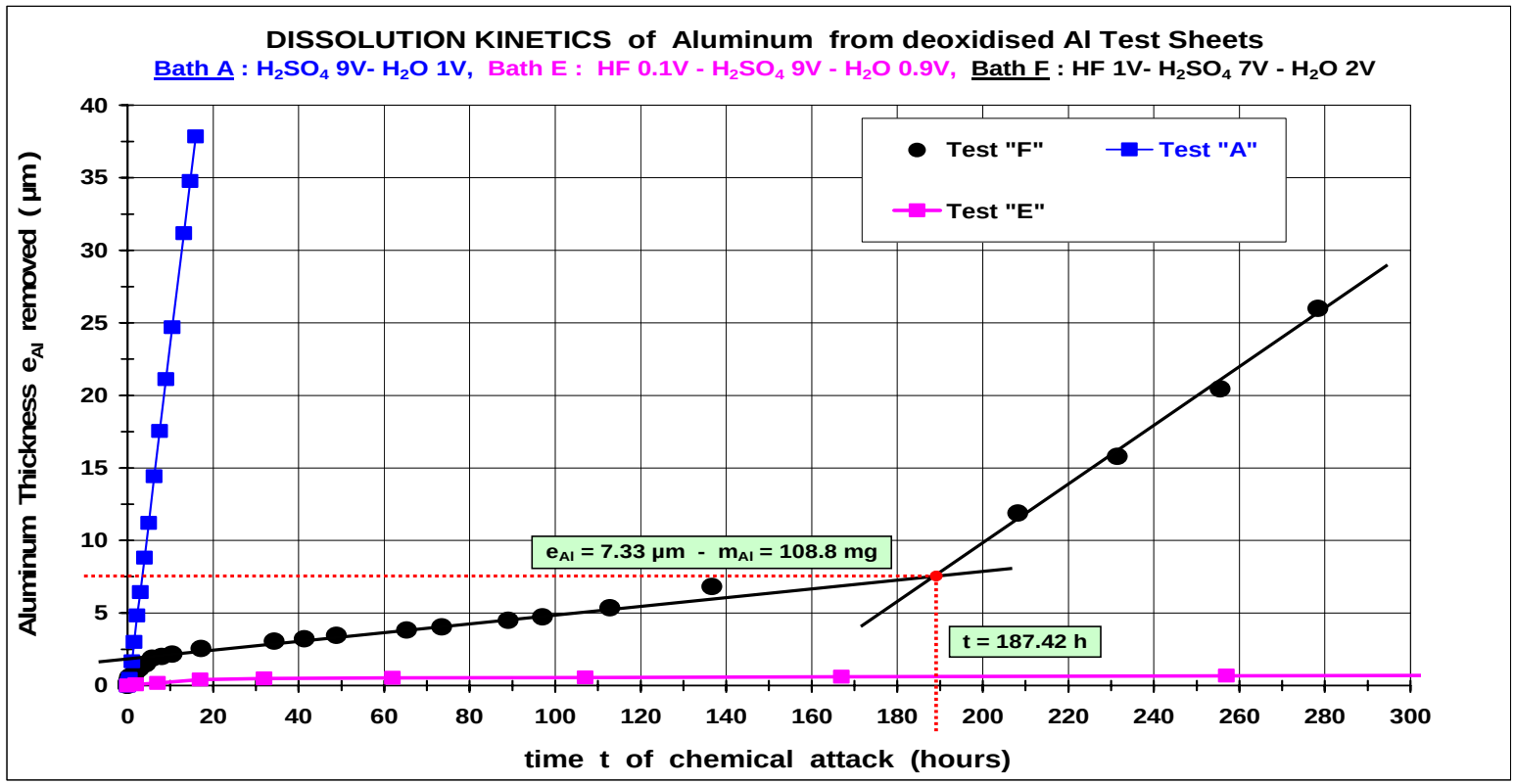
- ***Aging vs impurity content.***
- ***Efficiency (etching rate, life-time, surface state)***

Aging Of The Bath

- Drop in the polishing speed.
- Deterioration of samples' surface.
- Changes in intensity oscillations.
- Aluminum corrosion, S and H_2S production



Passivity of alumina (without bias)



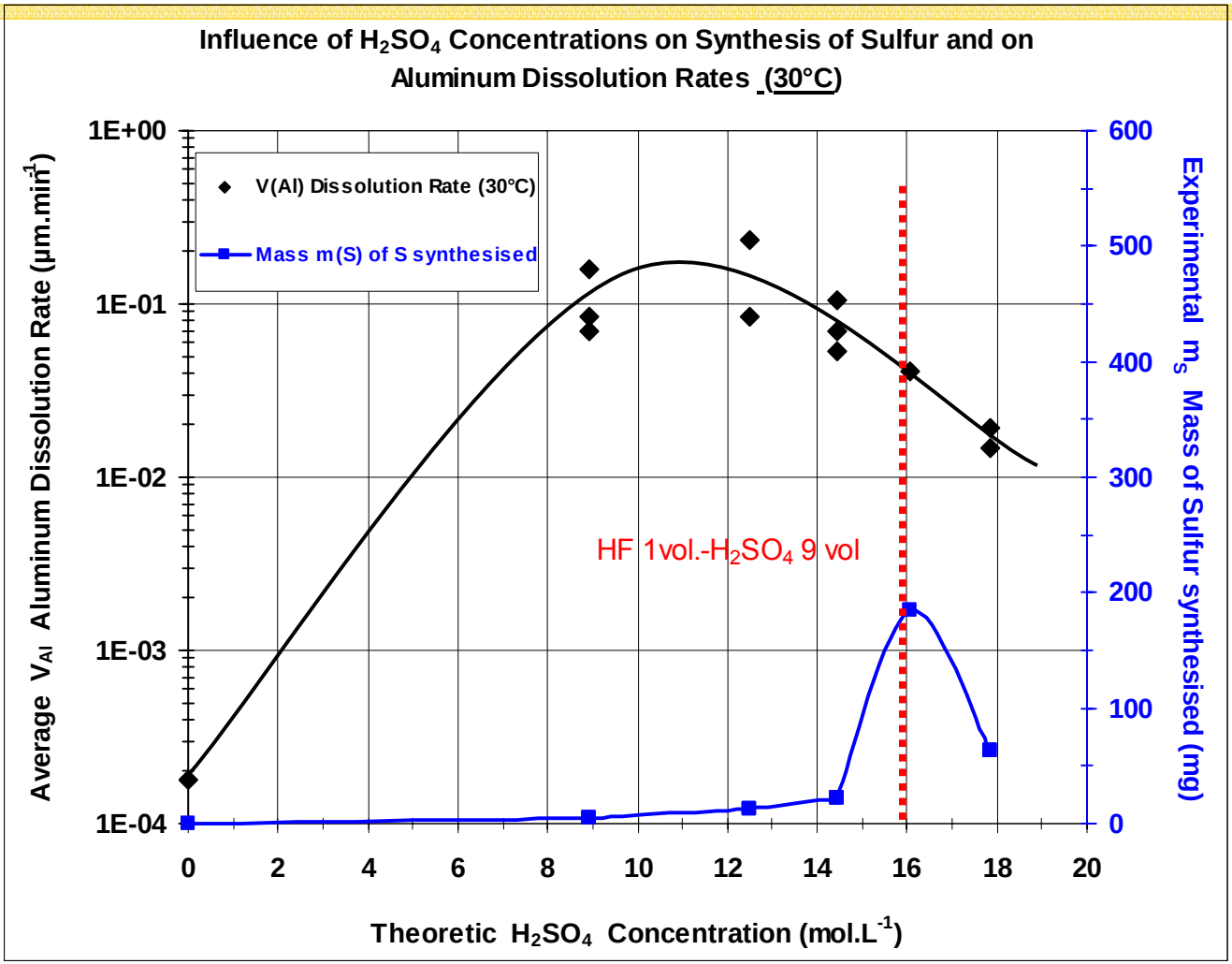
High H_2SO_4 content

Same H_2SO_4 Content, slight add^n of HF

Less H_2SO_4 content, higher of HF !?

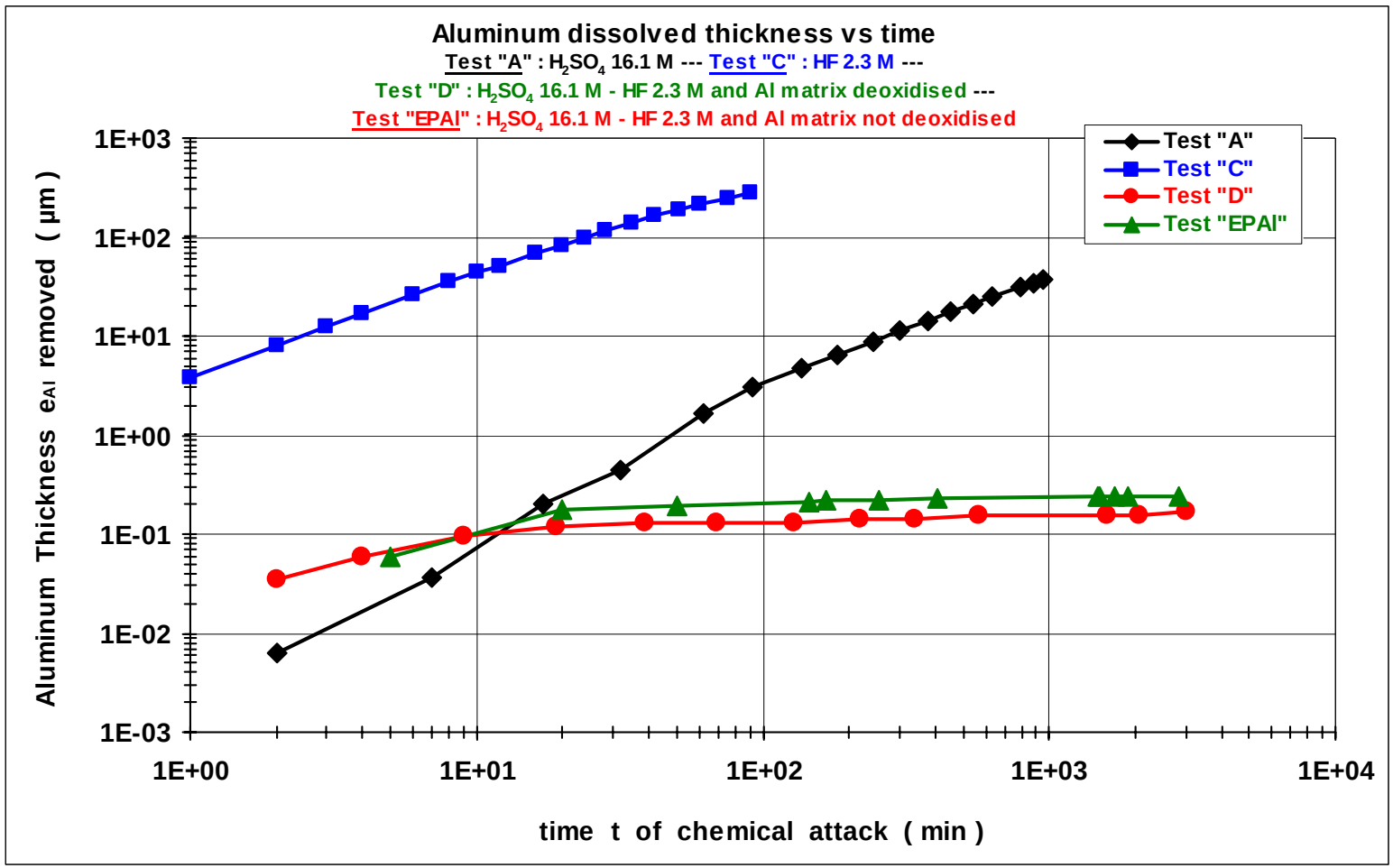
Test #	Volumic composition				Sulfur synthesis	
	HF	H_2SO_4	H_2O add	Total Volume	Time (minutes)	Sulfur mass (g)
A	0	9 V	1V	10 V	955	184,2
E	0,1 V	9 V	0,9 V	10 V	5930	9,9
F	1 V	7 V	2 V	10 V	16705	29,1

Aluminum corrosion, S and H_2S production



Lower content of H_2SO_4 reduces S production but increases Al corrosion (± acceptable !)

Passivation of Aluminum



Still corrosion is far more lower than in separated acids

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Aluminum is etched in each concentrated pure acids

- A few quantity of the other acid or dilution is enough to slow down drastically the dissolution of Al inside EP solution.
- Sulfur and H_2S is produced at high H_2SO_4 concentration. S appears also when HF is reduced due to evaporation or reaction => add HF !?

What is going on with bias ?

Reduction of SO_4^{2-} at the cathode under bias

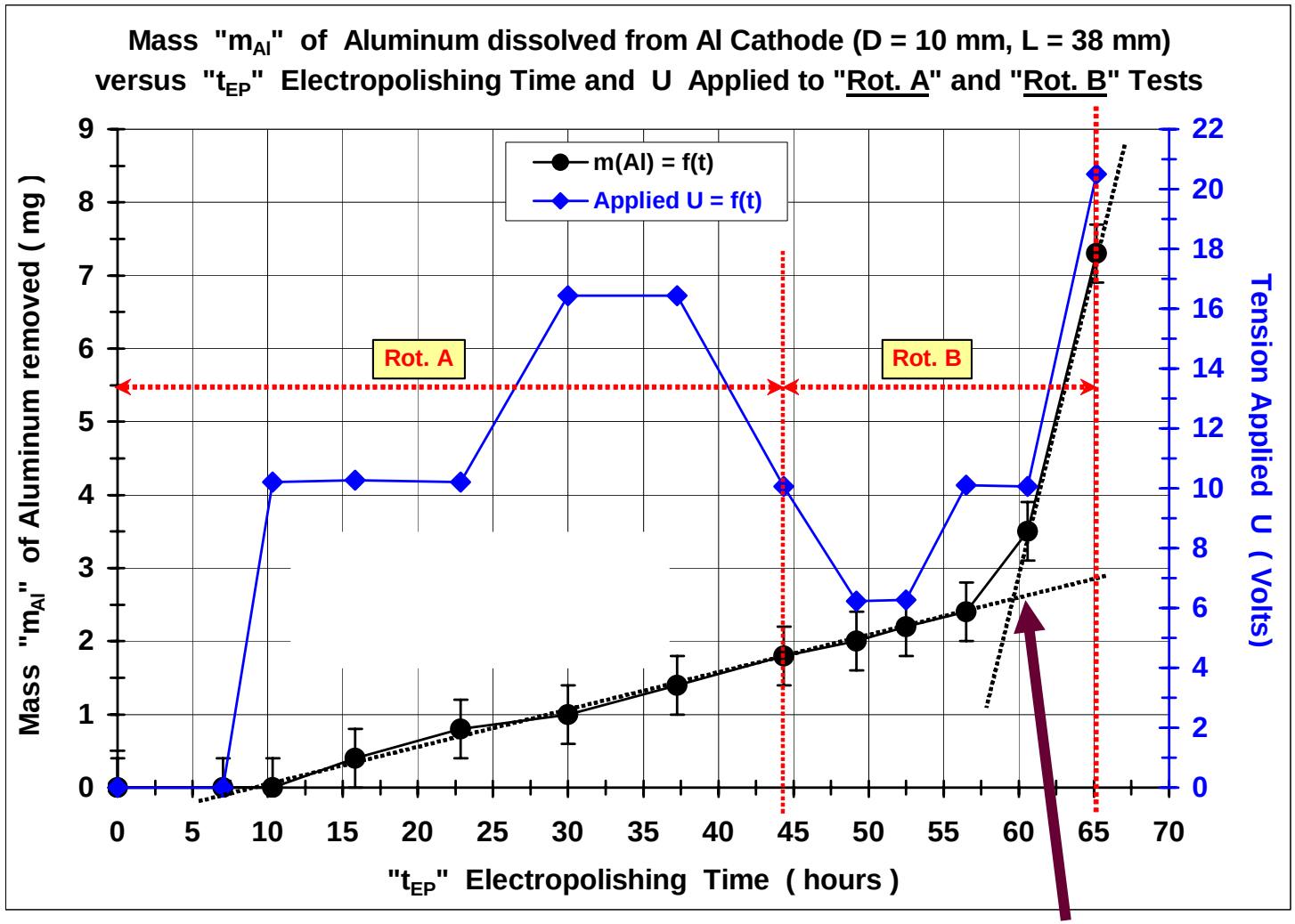
- slow but continuous corrosion of Al
- high quantity of S

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

Additional formation of sulfur at cathode

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

Cathode corrosion under bias



Corrosion becomes important when HF has reduced due to reaction + evaporation

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Without bias

- HF prevents S synthesis in presence of H_2SO_4
- H_2SO_4 hinders Al dissolution in presence of HF
- Active dissolution of Al and production of S when HF decreases (evaporation, reaction)

With bias:

- Synthesis of S and corrosion of aluminum cannot be hindered
- They can be reduced (↑ HF, ↑ H_2O,↓ H_2SO_4)

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S is not soluble in H_2O : rinsing process

- works in ethanol, but not very effective
- is very effective in chloroform, but safety issues

Rinsing process must be improved

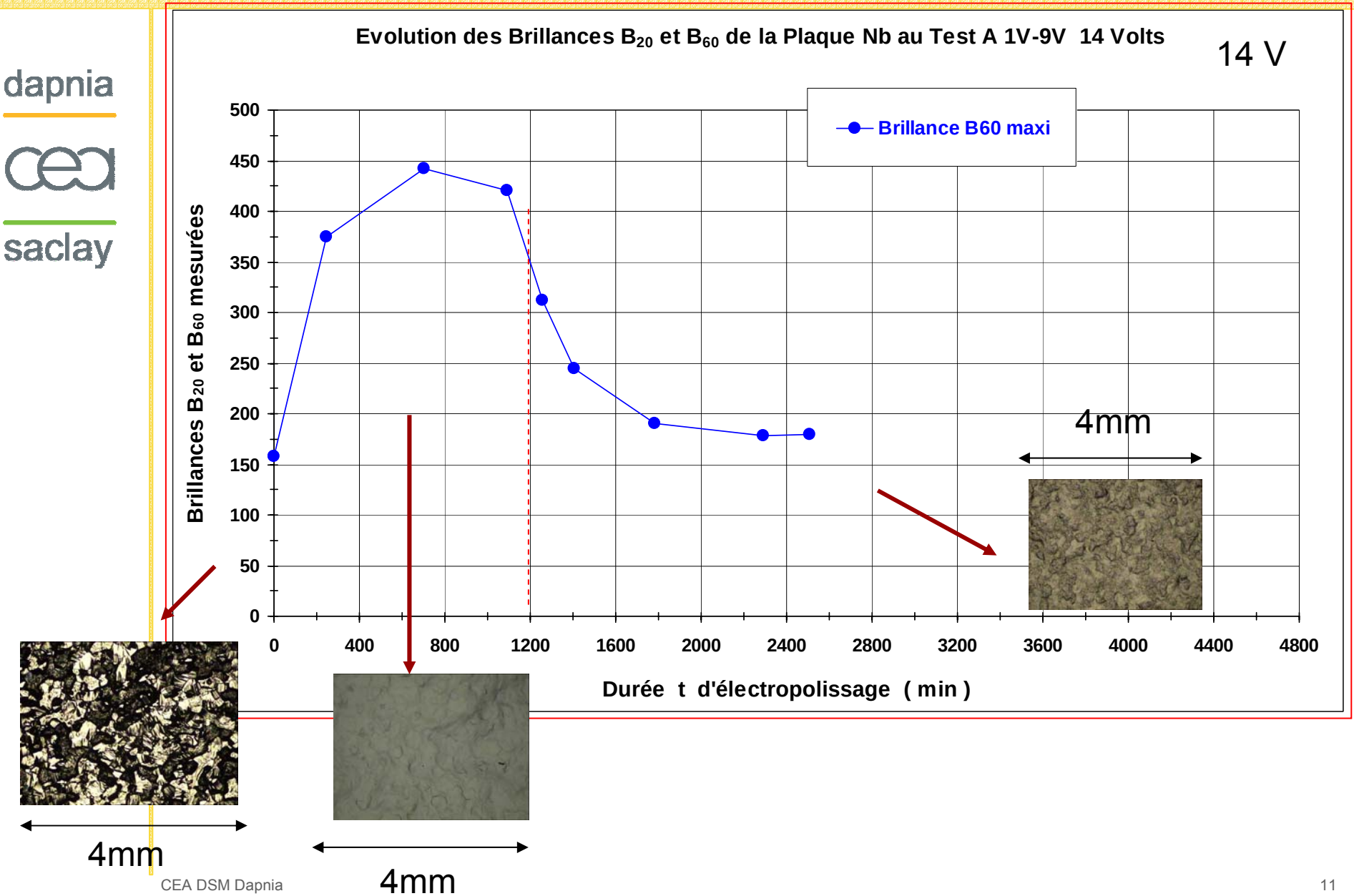
Aluminum :

- Is slightly dissolved in acidic mixture
- keeps in the form of Al^{3+} salts

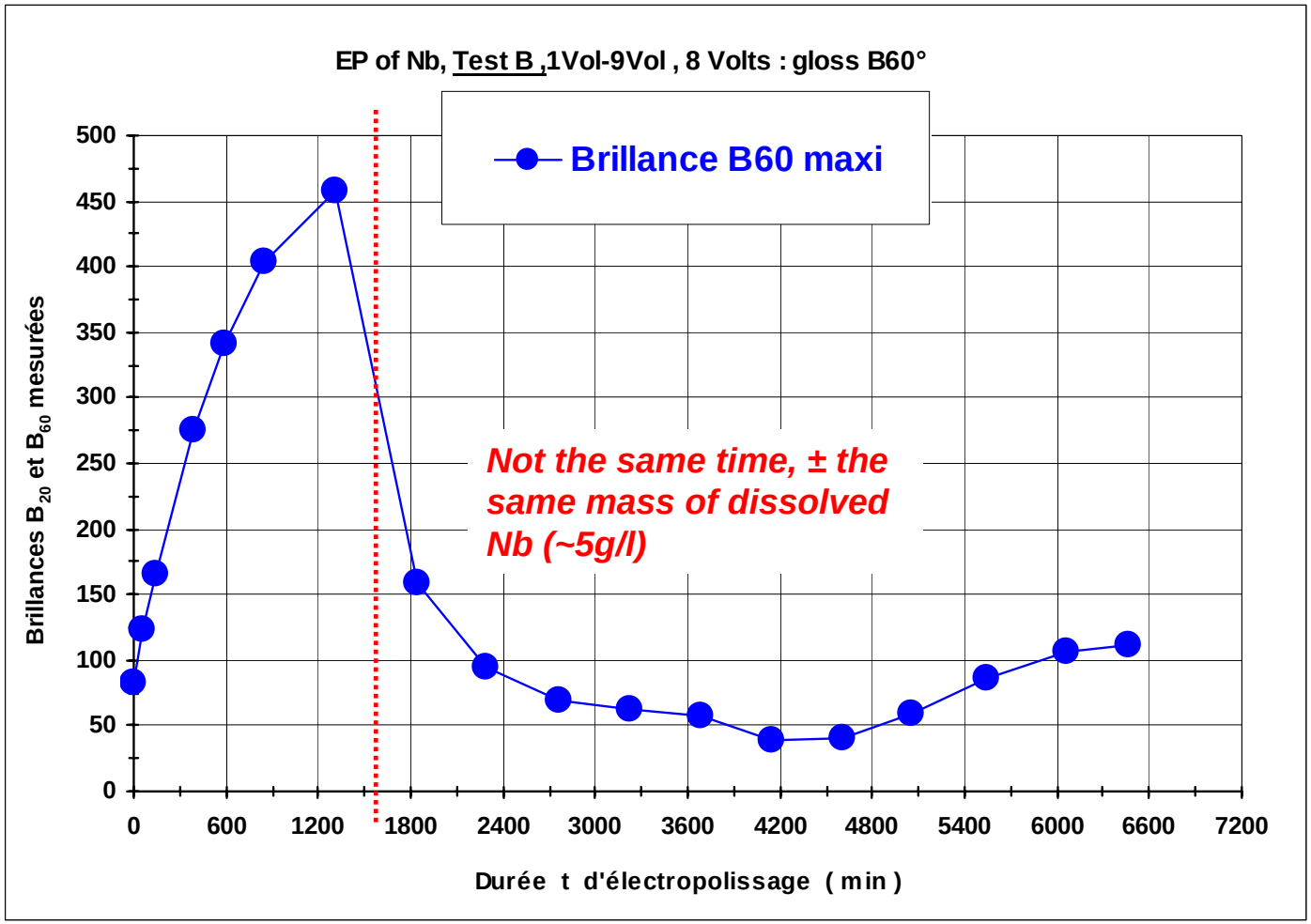
Rinsing process must be improved

+ Change in EP bath composition : ↑ HF, ↑ H_2O

Aging effect on samples' surface

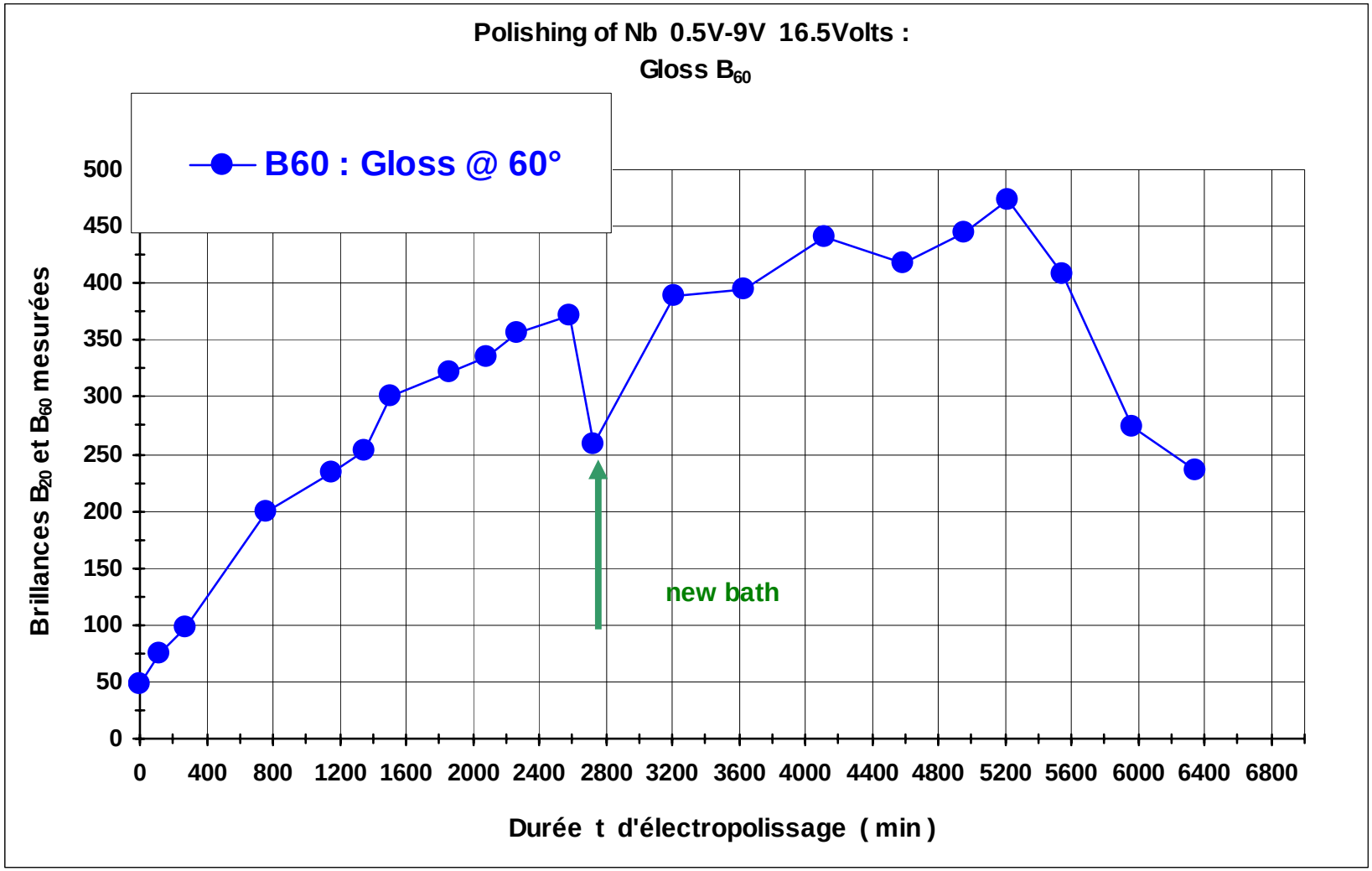


Aging of bath 2)



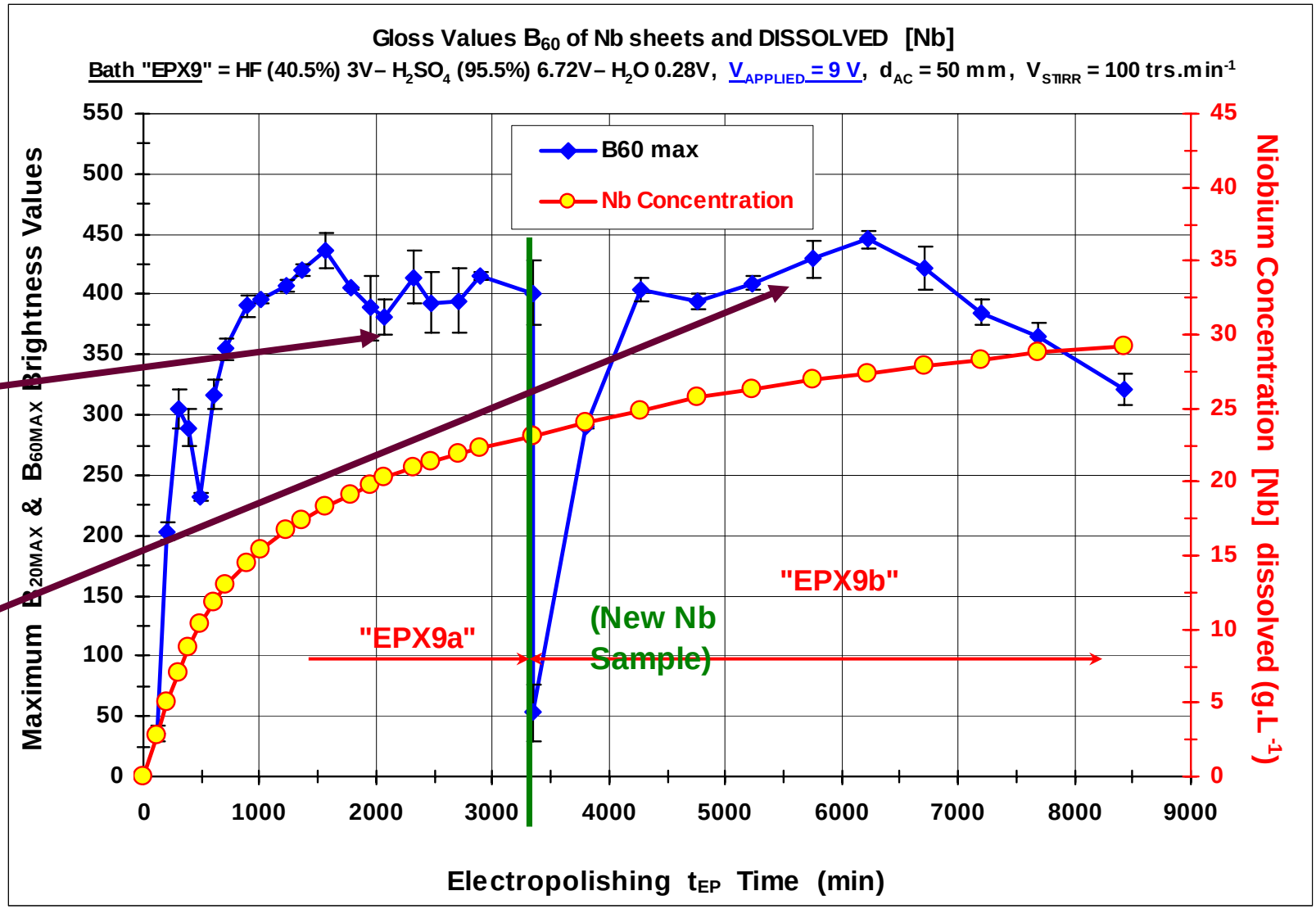
Does the Nb content of the solution matters ? (EP bath should be changed more often ?)

Changing for a fresh mixture (same sample)



Gloss can be further improved with a fresh bath

Higher HF content / Changing for a fresh sample



Nb content ~ 23g/l

Nb content ~ 28g/l !!!

Enhance HF and or H_2O ?

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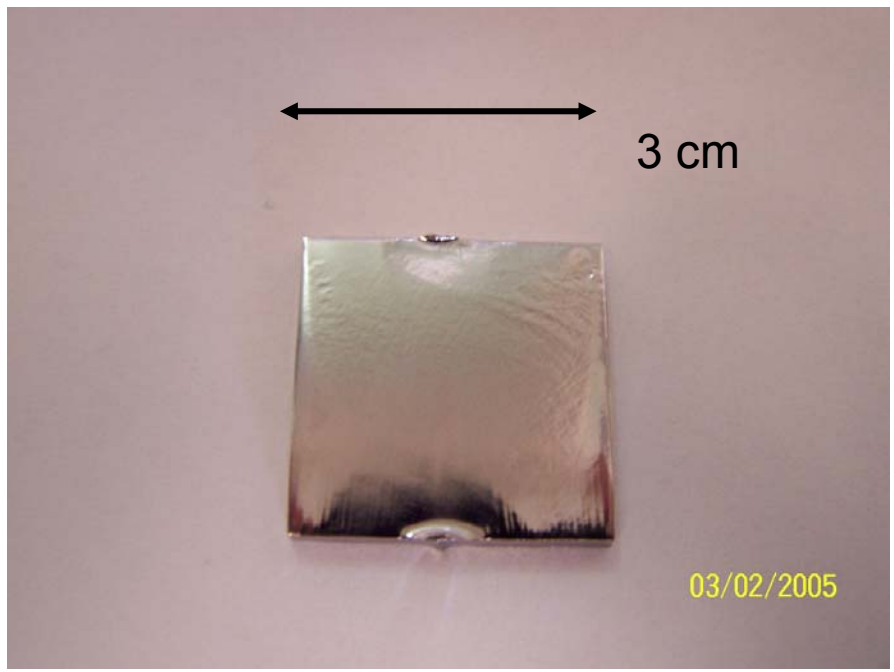
e.g. : HF 3 Vol. + H_2SO_4 6.72 vol + H_2O 0.28 Vol :

- Not « polishing », but « active dissolution *»
- High dissolution rate, lower potential
- Long lasting (Nb >> 25 g/ l and still B_{60} = OK)
- Very glossy, but some ripples

Acceptable ?

* No plateau in th E=f(V) curve



Possible compositions

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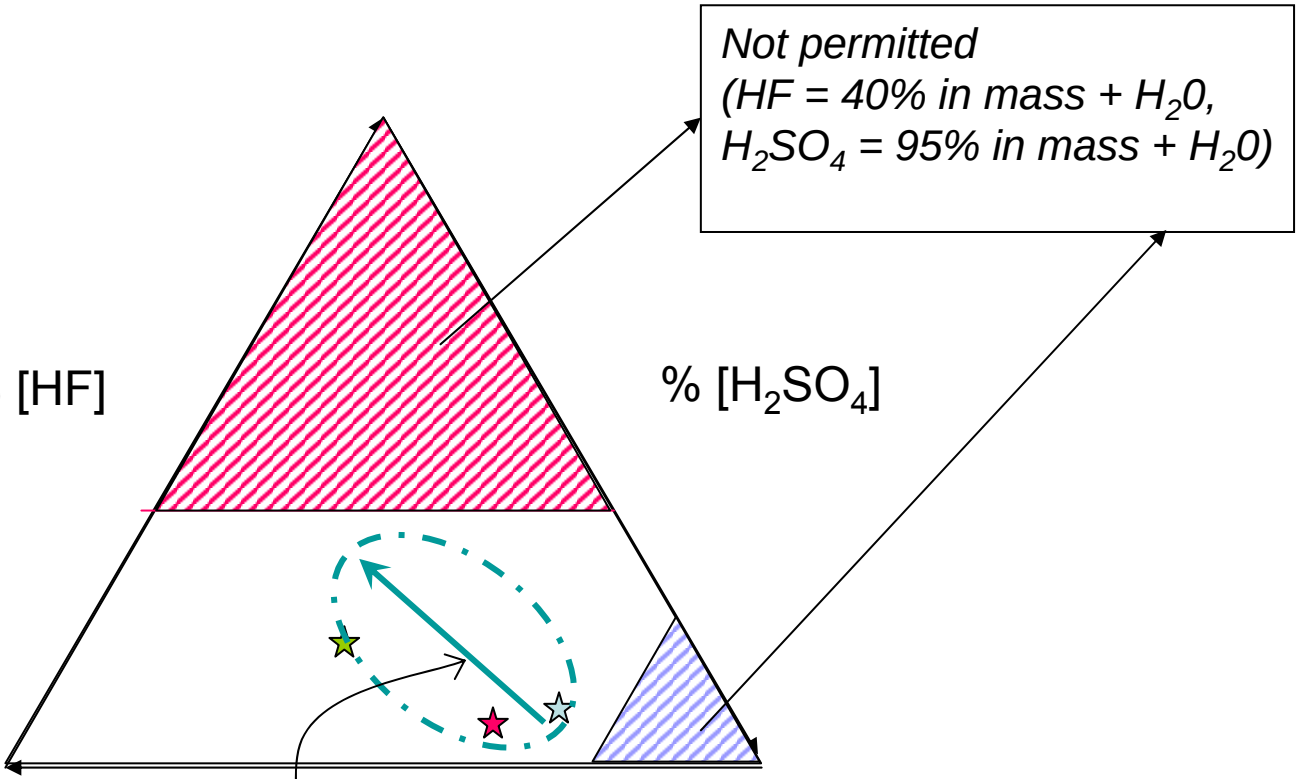

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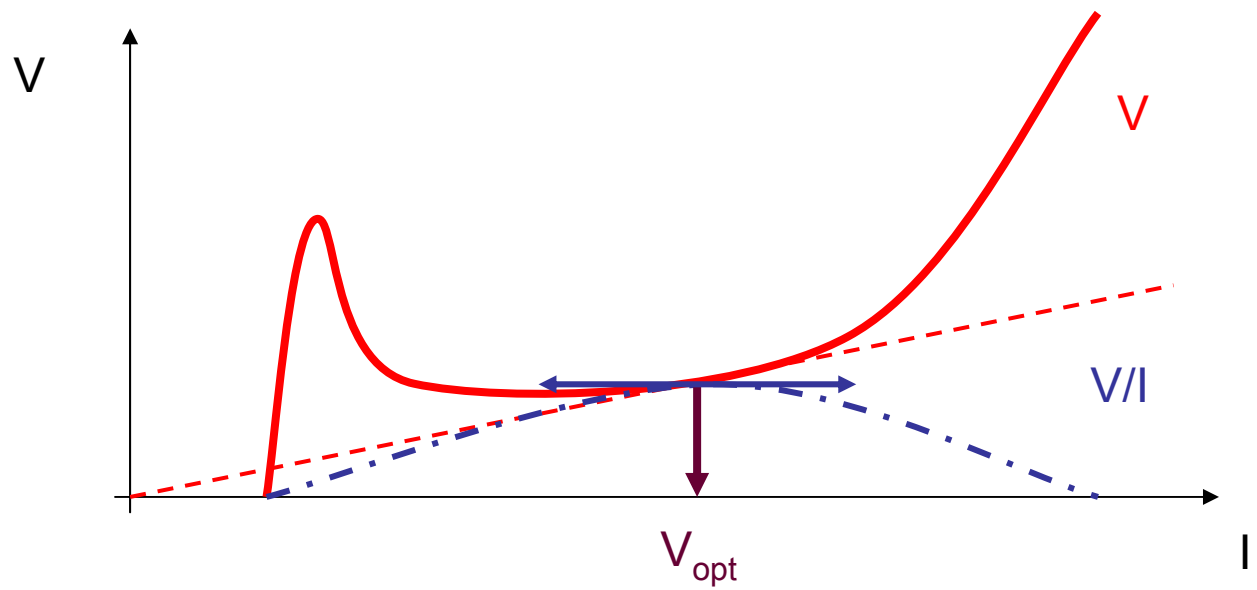
Literature says :

Polishing = plateau in the I=f(V) curve + diffusion limitation

=> 1st order criteria : « ideal V » @ the highest cell impedance



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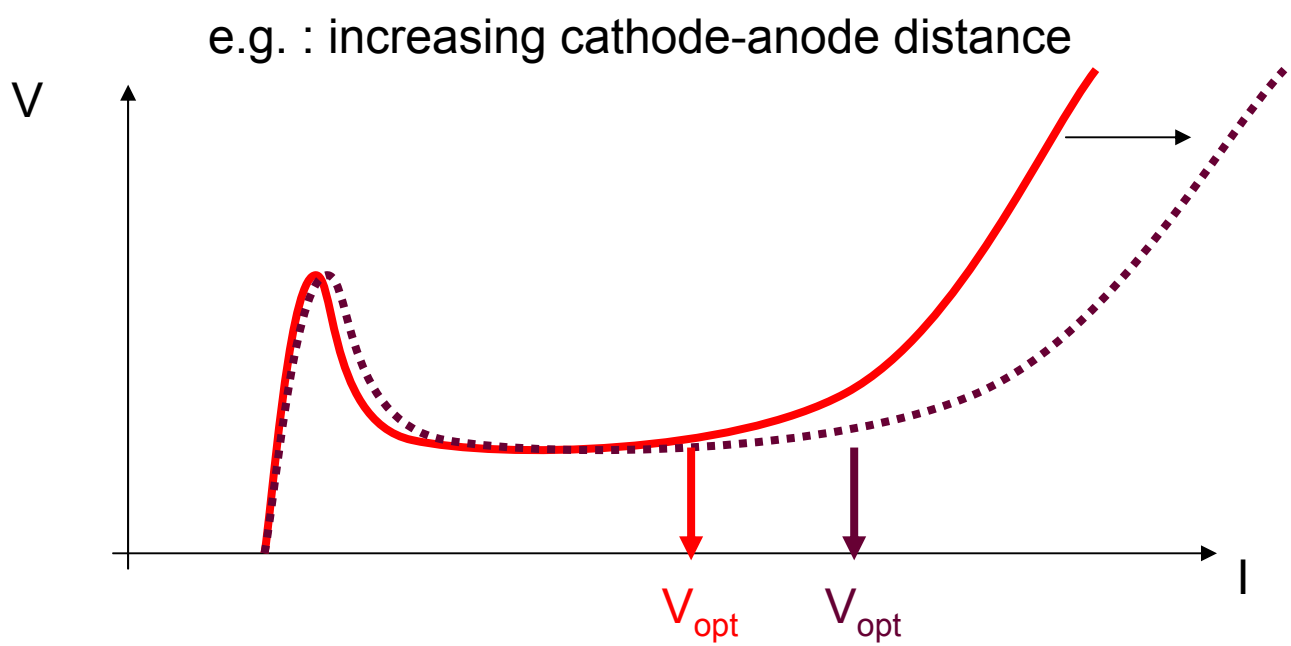
The I=f(V) curve depends on the characteristics of the «Circuit» (Geometry, field repartition, …)

=> The I=f(V) curve must be determined in the real situation !!!!!!



Conclusion

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- Increasing HF, H_2O and decreasing H_2SO_4 seems interesting in term of increase life time and efficiency of polishing bath, as well as reducing Sulfur content. It also allows to reduce bias, and possibly power.
- Al corrosion cannot be hindered, but is low and can be acceptable.
- Decision must be made between medium surface state but long lasting EP bath composition, and better surface state with lower life time etc.

=> test cavities with new bath composition !