

## Spectroscopic and electrochemical studies of the chemical oxide growth in SC-1 and SC-2

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As well known, the SC1 and SC2 solutions oxidize the Si surface. The growth of the chemical oxide was followed in situ by impedance spectroscopy (EIS). Besides, the surfaces have been characterized ex situ by ATR and ARXPS. A brief discussion of the results is hereafter presented, using the analytical method mentioned above.

The Nyquist plots have been recorded during more than 3 hours in our electrochemical cell under OCP [1]. The diameter of the semicircles increases with time in both SC-1 (Fig 1) and SC-2 indicating that a silicon oxide is growing up on the surface. Experimental results fit an almost ideal RC circuit in the case of SC-1 solutions. After 3h, the capacity has reached a plateau of  $1\mu\text{F}/\text{cm}^2$ . The capacity of the oxide layer for SC-2 has been expressed as a non-ideal frequency-dependent capacitor called constant phase element (CPE), which continuously increases with time. The CPE behaviour in SC-2 is generally attributed to a non homogeneous structure of the surface.

Chemical surface bonds were evidenced with ATR and ARXPS spectra. In the case of SC-1, the absorption for the Si-H vanished rapidly, and moreover the longitudinal and transversal modes of the Si-O-Si bonds vibrations (LO and TO) increased with time, indicating that a silicon oxide has grown on the surface. In contrast with SC-1, in SC-2 solutions the absorption remained important in the Si-H region and the LO vibration was not visible. After 3h immersion, the Si atoms back bonds were oxidized leaving  $\text{O}_3\text{Si-H}$  bonds on the surface together with Si-O-Si surface groups. These results suggest that the Si oxide coverage formed in SC-2 was less uniform than in SC-1.

The obtained XPS data (table 1) report the amount of suboxides at different time in SC-1 and SC-2. From the XPS estimated silicon oxide thickness, the coverage proved to be less than a monolayer for short SC-2 treatment [2]. At longer time in SC-2 as well as in SC-1, the results were in agreement with a complete coverage.

Finally, the results show a good correlation between in and ex situ analysis. The values of the AC-circuit components are quite consistent with the TO/LO splitting and surface coverage deduced from ATR and XPS data.

### References

[1] V. Bertagna, F. Rouelle, and M. Chemla, *J. Appl. Electrochem.*, **27**, (1997), 1179.

[2] Z. H. Lu et al., *Appl. Phys. Lett.*, **63**, (1993), 2941.

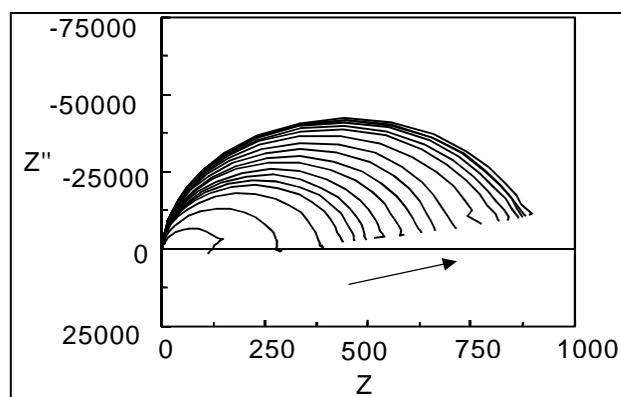


Fig 1. Nyquist plots for the oxide growth in SC-1.

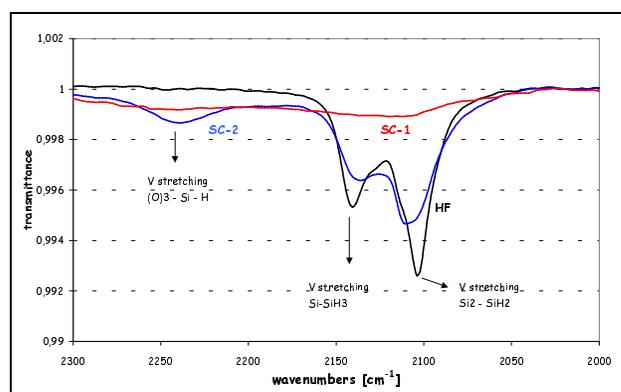
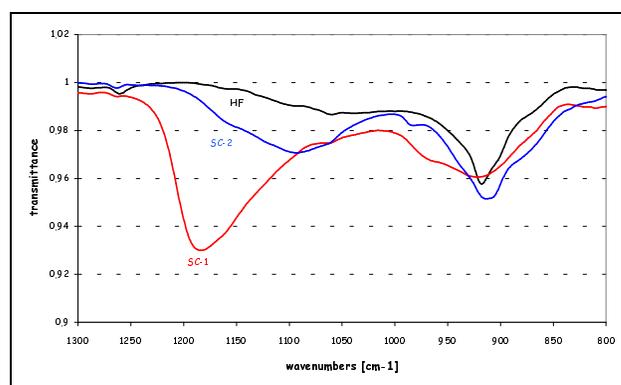


Fig 2&3. ATR spectra data in p polarisation for SC-1 and SC-2 oxides at 7 minutes.

	Thickness [Å]	Percentage of different suboxide /Si total				
		Si <sup>0+</sup>	Si <sup>1+</sup>	Si <sup>2+</sup>	Si <sup>3+</sup>	SiO <sub>2</sub>
HF	-	77.6	4.2			
SC1 7'	2.7	29.2	2.5	1.2	1.7	12.8
SC1 3h	3.8	24.6	3.2	1.7	1.5	12.8
SC2 7'	0.45	67.1	8.4			3.3
SC2 3h	2.8	33.1	3.3	1.5	0.7	15

Table 1. XPS data at take off angle of 18° for SC-1 and SC-2 oxides.