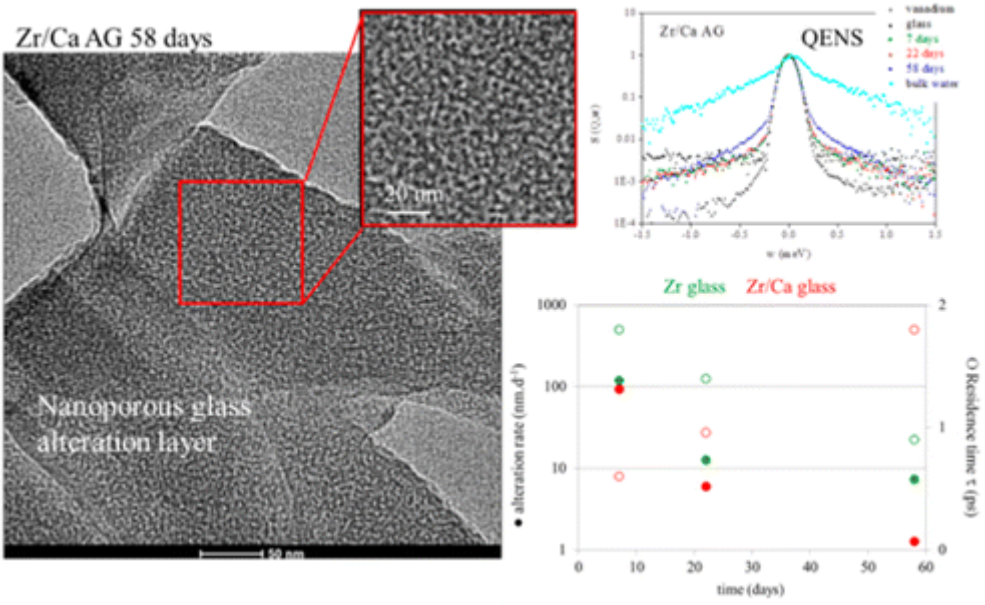
Advanced characterization

Water Dynamics in Nanoporous Alteration Layer Coming from Glass Alteration: An Experimental Approach

JOURNAL OF PHYSICAL CHEMISTRY C 119 (28) 15982-15993, 2015 DOI: 10.1021/acs.jpcc.5b03073

Collaboration: ICSM Marcoule, CEA Marcoule, Univ. Aix Marseille
Involved PNM researcher: Julien Cambedouzou

Protons dynamics in borosilicate glasses of various compositions (SiO2/B2O3/NaO/CaO/X, with X = Al2O3 or ZrO2) at various stages of alteration have been characterized at a picosecond scale using quasielastic neutron scattering (QENS). The results obtained were compared to the one from porous silica-based material (MCM41) having pore wall surfaces with Si–OH, Al–OH, or Zr–OH terminal groups and pore sizes around 2.3 nm. The composition and the nanoporosity of the alteration layers were characterized using solution analysis, small angle X-ray scattering and transmission electron microscopy. The strength hydrogen bonds of confined water were studied using thermogravimetric analysis and differential thermal analysis. The results showed that the free water bonding and the mobility of protons depend on the altered glass composition and that the residence time of water obtained from QENS analysis is apparently correlated to the alteration rate of the glass. Moreover, whereas the composition of the alteration layer can partially explain this result, the presence of ions in the leachate filling the gel nanoporosity seems to be the main factor affecting the water/protons mobility. The latter result is really important since the ion solvation and the ion adsorption on the surface can strongly impact the hydrolysis rate of the pore wall of the gel and can also modify the kinetics of dissolved elements recondensation into the nanopore.



Advanced characterization

Nanoporosity, aging or swelling of supported thin films

Collaboration: DM3/MCP, IP2, IATE Montpellier, ST Microelectronics Crolles, CEA Grenoble
Involved PNM researcher: Vincent Rouessac

Ellispometry coupled with gas sorption:

A. J. Kinfack Leoga, L. Youssef, S. Roualdes, V. Rouessac, *Thin Solid Films*, 660 (2018) 506-515.
M. Lépinay, D. Lee, R. Scarazzini, M. Bardet, M. Veillerot, L. Broussous, C. Licitra, V. Jousseaume, F. Bertin, V. Rouessac, A. Ayral, *Microporous Mesoporous Mat.* **228** (2016) 297.
M. Haacké, R. Coustel, V. Rouessac, S. Roualdès, A. Julbe, *Plasma Processes and Polymers.* **13** (2016) 258.
V. Rouessac, A. van der Lee, F. Bosc, J. Durand, A. Ayral, *Microporous Mesoporous Mat.* **111** (2008) 417.
W. Puyrenier, V. Rouessac, L. Broussous, D. Rébiscoul, A. Ayral, *Microporous Mesoporous Mat.* **106** (2007) 40.
W. Puyrenier, V. Rouessac, L. Broussous, D. Rébiscoul, A. Ayral, *Microelectronic Engineering* **83** (2006) 2314.
V. Rouessac, R. Coustel, F. Bosc, J. Durand, A. Ayral, *Thin Solid Films* **495** (2006) 232.
P. Revol, D. Perret, F. Bertin, F. Fusalba, V. Rouessac, A. Chabli, G. Passemard, A. Ayral, *Journal of Porous Materials* **12** (2005) 113.

QCM coupled with gas sorption:

V. Thoury-Monbrun, S. Gaucel, V. Rouessac, V. Guillard, H. Angellier-Coussy, *Carbonhydrate Polymer*, 190 (2018) 307.
V. Monnier, E. Dubuisson, N. Sanz-Menez, B. Boury, V. Rouessac, A. Ayral, R. B. Pansu, A. Ibanez, *Microporous Mesoporous Mat.* **132** (2010) 531.
T. Mérian, D. Debarnot, V. Rouessac, F. Poncin-Epaillard, *Talanta* **81** (2010) 602.
V. Rouessac, P. Tuluc, J. Durand, *Journal of Membrane Science* **230** (2004) 49.

Two coupled techniques installed at IEM can give some information on thin deposited films. Quartz crystal microbalance coupled with gas sorption (water, ethanol, …) allows to follow the film mass uptake as a function of the pressure in steady state due to vapor sorption or dynamically as a function of time after a sudden increase of the vapor pressure. The film should be deposited on a dedicated piezoelectric quartz support.

Spectroscopic ellipsometry (extended visible light) coupled with vapor sorption allows to characterize the open nanoporosity of films (thickness 100nm-1µm) deposited on any polished reflective support (polished Si wafer, stainless steal,…) and the evolution of thickness due to capillary condensation in rigid mesopores or due to sorption in flexible polymeric films.
By following the refractive index, it is also possible to have information of any short time aging of the layer submitted to the vapor.