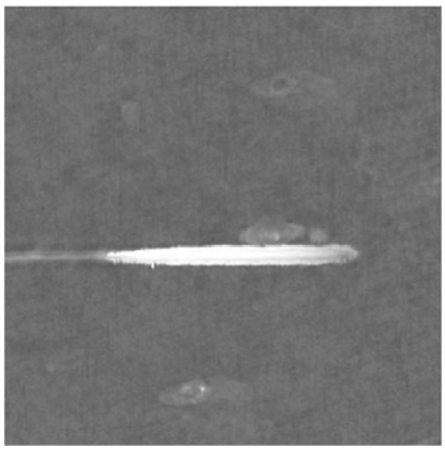
ANR Postdoc Position at LCVN/LPMCN (2008)

Investigation of the wetting properties of the fracture surfaces in silica glass by colloidal probe microscopy

The presence of a liquid condensed phase inside the nanometrically sharp cracks in glasses plays an important role in the physics and chemistry of crack propagation, as well as in many industrial problems related to the strength and lifetime of glass products. The slow crack propagation in glasses is commonly explained by the stress-corrosion theory [1]: water molecules that move in the crack cavity effectively reduce the bond strength at the strained crack tip and thus enhance the subcritical crack propagation velocity, which is ruled by the rate of a thermally activated chemical reaction. However, the details of the local environmental condition at the crack tip in moist air are very complex and still need careful investigation.

We have recently reported direct evidence of the presence of a submicrometric liquid condensate at the crack tip of a pure silica glass during very slow propagation [2]. These observations were based on *in-situ* atomic force microscopy (AFM) phase imaging techniques applied on double cleavage drilled compression (DCDC) glass specimens in controlled atmosphere [3]. By measuring the variations of the length L of the condensed phase (Fig. 1) as a function of the crack opening (determined by the stress intensity factor K_I), we showed that the condensation length is determined by an equilibrium condition and we were able to measure the critical condensation distance Hc as a function of relative humidity [4].



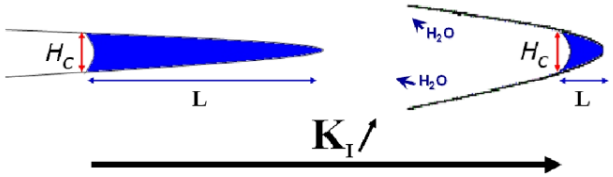


Figure 1 – (Top) Typical AFM phase image of a crack propagating from left to right (1x1 μm). The light contrasted area corresponds to the condensate. (Bottom) The variation of the condensation length as a function of the stress intensity factor is determined by a critical distance Hc between the crack walls.

The measured values of the critical condensation distance are of the order of 10 nanometers, which is quite large in relation to what we expected theoretically. However, the physico-chemical properties of the liquid condensate are complicated by the high degree of confinement, the presence of an elevated local stress and the development of surface charges caused by the corrosion process. A deeper investigation of the wetting properties of the fresh fracture surfaces is thus needed and will be the objective of the post-doctoral work.

In spite of the excellent lateral resolution, the AFM phase imaging techniques used to observe the condensed phase can presently provide only qualitative information on the local variations of the wetting properties due to the lack of control on the exact shape of the AFM tip and on the detailed laws of the tip-surface interactions. The use of a Surface Force Apparatus, measuring the force between a flat surface and a spherical bead of millimetric diameter allowed the LPMCN group for very precise measurements of the wetting and condensation properties [5-7]. However, the large size of the probed area involves averaging on a greater degree of heterogeneity (in the roughness and cleanness of the surface) that alters the overall

interaction. A promising solution is to combine the two techniques by using a micrometric colloidal probe with well controlled sphericity glued at the end of an AFM cantilever.
The main objective of the post-doctorate is the achievement of a quantitative interpretation of these intermediate scale measurements and the characterization of the evolution of the wetting properties of the fresh crack surfaces as a function of time (after fracture), applied stress and environmental conditions. This understanding will then be used to model both the condensation inside the crack tip and the capillary interactions between the AFM tip and the glass surface.

[1] S. M. Wiederhorn, "Influence ofWater Vapor on Crack Propagation in Soda–Lime Glass", J. Am. Ceram. Soc., 50, 407–14 (1967).
[2] L. Wondraczek, M. Ciccotti *et. al*, "Real-time observation of a non-equilibrium liquid condensate confined at tensile crack tips in oxide glasses", J. Amer. Ceram. Soc. 89(2), 746-749 (2006).
[3] M. Ciccotti, M. George *et. al*, "Dynamic condensation of water at crack tips in oxide glasses", J. Non-Crist. Solids. 354, 564-568 (2008).
[4] A. Grimaldi, M. George, et. al, "The Crack Tip: A Nanolab for Studying Confined Liquids", PRL, 100, 165505 (2008).
[5] E. Charlaix, J. Crassous, "Adhesion forces between wetted solid surfaces", J. Chem. Phys. 122, 184701 (2005).
[6] J. Crassous, E. Charlaix, J.L. Loubet, "Capillary condensation between high energy surfaces", Europhys. Lett. 28(6), 415-420 (1994).
[7] F. Restagno, J. Crassous *et. al*, "A new Surface Force Apparatus for nanorheology", Rev. of Scient. Instrum. 73(6), 2292-2297 (2002).

Profile : the candidate is expected to possess some experience in one of the following fields: Atomic Force Microscopy, capillary forces and wetting phenomena, surface science.

Period : 12 months (start in 2008)

Location: Laboratoire des Colloïdes, Verres et Nanomatériaux (UMR 5587 - Université de Montpellier II - Place Eugène Bataillon - 34095 Montpellier cedex 5, France).
The first 3 months will be spent in the Laboratoire de Physique de la Matière Condensée et Nanostructures (UMR 5586 - Université Lyon 1 - Campus La Doua - 6 rue Ampère - 69622 Villeurbanne Cédex).

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Fundings : ANR CORCOSIL (2007-2010) will fund the present post-doctoral position, as well as a last generation AFM that recently upgraded the LCVN experimental setup.

ANR Postdoc Position at CEA Saclay/LCVN (2008)

AFM investigation of the nonlinear process zone at the crack tip in glasses

Recent in situ AFM observations have shown that fracture of silicate glasses proceeds through the nucleation, growth and coalescence of damage cavities ahead of the main crack tip, while classical models predict the successive breaking of atomic bonds right at the crack tip, like in cleavage [1,2] (Fig. 1). However, experiments performed up to now are confined to the free surface of the specimen. The conditions at the surface are different from the ones in the bulk for what concerns the mechanical and environmental state.

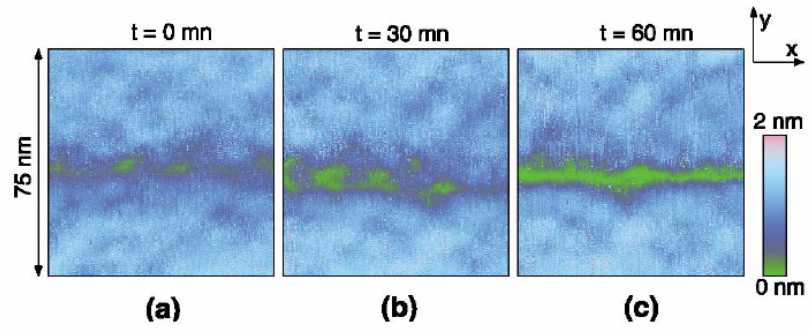

Fig. 1: AFM topographic image of the glass surface showing a crack slowly propagating from left to right. In green we can observe the formation of cavities of nanometrical size before the arrival of the fracture.

The post-doc activity aims at realizing cartographies of damage induced by cavities in the bulk of the specimen by coupling experimental analysis of the post-mortem fracture surfaces by AFM and statistical studies of the fracture surface roughness. The 3D distribution of damage within the process zone will be estimated through the FRActure Surface Topography Analysis (FRASTA) technique, originally introduced to study damage in metallic alloys [3]. The method consists in analysing the mismatch between the two opposite fracture surfaces corresponding to the two halves of the sample (Fig 2). Lateral resolution about 5 nm and vertical resolution about 0.5 nm will be reached through AFM, which should allow detecting the remnants of damage cavities formed during slow crack growth. It should be emphasized that the resolution of our apparatus is very close to the sizes of the non-matching zones in the fractured surfaces expected from the in situ observations performed at the free surface of the specimens. Therefore, special attention will be paid to develop clear statistical estimators so that cavity remnants can be unambiguously detected and separated from the inherent noises of our experimental setup.

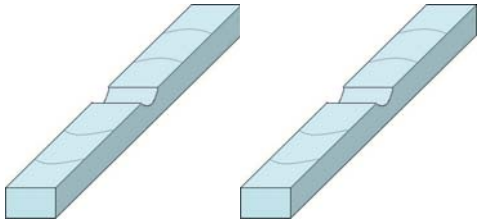

Fig. 2: In the *post-mortem* experiments the two halves of a fractured specimen are mounted side to side in order to examine corresponding fracture surfaces.

The post-doc will also investigate the scaling properties of the post-mortem fracture surfaces (Fig 3). It has been shown recently that fracture surfaces exhibit two scaling regimes [4]: at large length scales, roughening due to disorder can be explained within a purely linear elastic theoretical framework; at length scales smaller than the process zone size, a completely different regime is observed. Hence, analyzing fracture surfaces gives a good estimate of the fracture process zone in a material. The variations of the scaling properties as a function of the loading conditions and environmental parameters will provide important complementary confirmation of the behaviour of the nonlinear deformation zone at the crack tip of glasses.

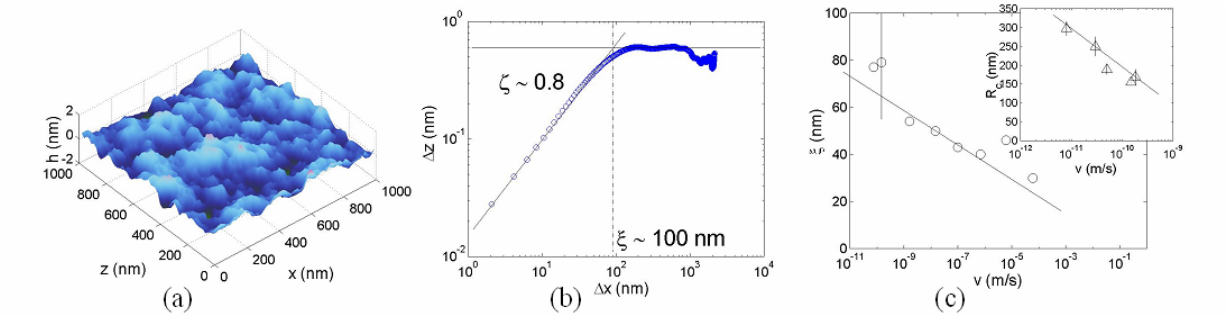

Fig. 3: (a) Morphology of the fracture surface in glass. (b) height-height correlation calculated from the fracture surface. This function exhibits self-affine properties characterized by a roughness exponent ζ~0.8 up to a cut-off length ξ~100 nm. (c) Variation of the cutoff length ξ as a function of the crack growth velocity (taken from [4]). The inset shows the variation of the process zone size Rc with the crack growth velocity. The similarity between the two curves suggests that the process zone size Rc sets the cut-off length.

The developed techniques will also be applied to the study of the fracture surfaces of glass ceramics in order to test the effect on the nonlinear process zone of the increasing degree of heterogeneity. Glass ceramics can be obtained by applying calibrated thermal treatments to a 'mother' glass resulting in the nucleation and growth of crystalline phases of nanometric size.

[1] F. Célarié, S. Prades, et al., "Glass breaks like metal, but at the nanometer scale", Phys. Rev. Lett., 90, 075504 (2003).
[2] D. Bonamy, S. Prades, et al., "Nanoscale damage during fracture in silica glass", Int. J. of Fract., 140, 3-14 (2006).
[3] T. Kobayashi, D.A. Shockey, "A Fractographic Investigation of Thermal Embrittlement in Cast Duplex Stainless Steel", Metall. Transac., 18A, 1941 (1987).
[4] D. Bonamy, L. Ponson, et al., "Scaling exponents for fracture surfaces in homogeneous glass and glassy ceramics", Phys. Rev. Lett., 97, 135504 (2006).

Profile : the candidate is expected to possess some experience in one of the following fields: Atomic Force Microscopy, fracture mechanics, fractal analysis.

Period : 12 months (start in 2008)

Location: The post-doctorant will spend 6 month in each of the two following laboratories:
1) Laboratoire des Colloïdes, Verres et Nanomatériaux (UMR 5587 - Université de Montpellier II - Place Eugène Bataillon - 34095 Montpellier cedex 5, France).
2) Service de Physique et de Chimie des Surfaces et Interfaces (CEA-Saclay - DSM/DRECAM/SPCSI - Centre de Saclay - 91191 Gif-sur-Yvette cedex – France)

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Fundings : ANR CORCOSIL (2007-2010) will fund the present post-doctoral position, as well as a last generation AFM that recently upgraded the LCVN experimental setup.