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### ► **To cite this version:**

Jean-Pierre Guin, Vincent Keryvin, Ludovic Charleux, Jean-Christophe Sangleboeuf, Mariette Nivard, et al.. Plasticity at nanoindentation site in glass : a possible experimental benchmark for numerical modeling. CFM 2015 - 22ème Congrès Français de Mécanique, Aug 2015, Lyon, France. <hal-03444639>

**HAL Id: hal-03444639**

**<https://hal.science/hal-03444639v1>**

Submitted on 23 Nov 2021

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# Plasticity at nanoindentation site in glass : a possible experimental benchmark for numerical modeling

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## Abstract :

*We have recently developed a technique allowing for the 3 dimensional probing, with a nanometer scale resolution, of small volumes affected by plastic events at Nano indentation sites. This method is sensitive to the structural changes in the glassy network (number of SiO<sub>4</sub> units per ring, Si-O-Si inter-tetrahedral angle, connectivity loss) resulting from the densification. The plastic zone due to structural modifications has an enhanced reaction rate which, if coupled with precise atomic force microscopy measurements (AFM), may be used for probing and rebuilt the densified volume beneath nano indentation or nano scratch imprints made on a glass surface. Instrumented nano indentations and scratches (250  $\mu$ N to 10 mN range) were made on various glass compositions. The effect of the applied load on the size and the shape of the plastic zone size is reported and compared to numerical simulations.*

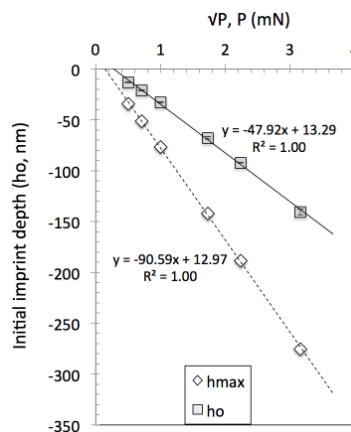
**Mots clefs : Nano indentation, glass , plasticity, finite element modeling**

## 1 Introduction

Although silica glass has an estimated theoretical strength of 35 GPa,[1] the extreme sensitivity of the glass surface to damage leads to considerably lower strength values reported in the literature for bulk samples (of the order of 20 MPa)[2]. Such surface imperfections or damages often result from the mechanical contact of the pristine glass surface with hard particles. Such an event may leave behind on the surface a permanent imprint and, providing the applied load is high enough, a well developed cracking system.[3] As the indentation test best mimics everyday life surface aggression, it makes this technique extremely popular and suitable for studying surface damage properties of materials in general.

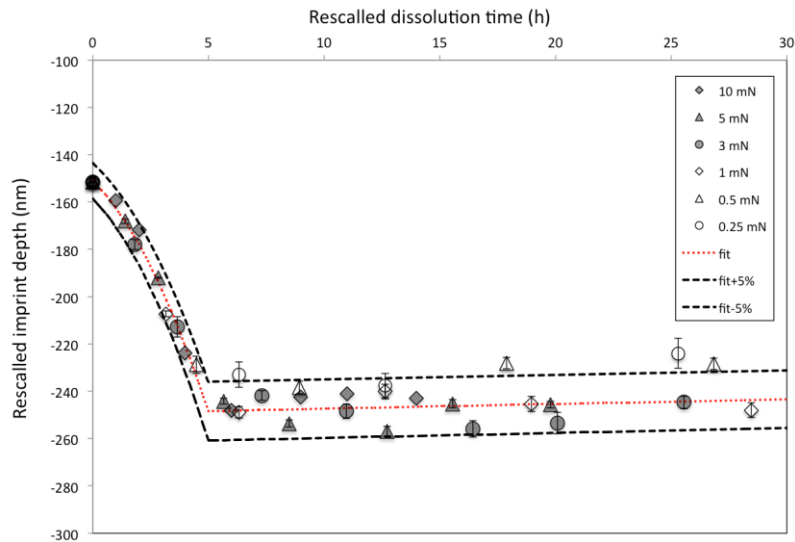
Previous works have demonstrated that under indentation testing, oxide glasses deform at first elastically then in a permanent way through two concomitant deformation mechanisms: a volume conservative one (shear flow) and a non volume conservative one (densification).[4-6] The relative importance of those two mechanisms is believed to play a major role and to trigger the occurrence of the well known median/radial crack system,[7] the most jeopardizing one regarding the mechanical

integrity of glass structures. Therefore, deformation mechanisms in glass are of first importance to understand the nucleation of crack systems under sharp contact conditions as it is shown by the number of recent publications in this field. To get the full picture scientists have developed a strategy, which relies both on a numerical approach and an experimental one. The main problem may be summed up by the fact that each numerical approach needs to be confronted to a physically sounded benchmark provided by experiment. We have recently developed a technique, which is able to probe, with a nanometer scale resolution, the volume affected by densification under nano indentations or scratches. Densification phenomenon involves structural changes in the glassy network (number of SiO<sub>4</sub> units per ring, Si-O-Si inter-tetrahedral angle, connectivity loss). Such structural modifications may modify the dissolution rate [8] of the densified glass (with respect to the one of the relaxed glass) either because the reactivity of the Si-O-Si bridge toward hydrolysis is favoured or because of a global connectivity loss leading to a lesser volumetric density of Si-O bonds to hydrolyse. This enhanced dissolution rate, if coupled with precise atomic force microscopy measurements (AFM), may be used for probing the densified volume beneath nano indentation or scratch imprints made on an oxide glass surface. In this work we report measurements made on both silica and window glass that were indented with loads ranging from 200  $\mu$ N to 10 mN. The data set is treated within the framework of indentation geometrical self-similarity as confirmed by the evolution of  $h_o$  (the final imprint depth after unloading) and  $h_{max}$  (the maximum penetration depth at the maximum indentation load  $P$ ) as a function of  $\sqrt{P}$  (figure 1).



**Figure 1 : Evolution of  $h_o$  and  $h_{max}$  as a function of indentation load  $P$  for Berkovich indentations made on silica glass.**

Following the hypothesis of indentation imprints performed within the geometrical self similar regime it can be shown that all the curves (Indentation imprint depth versus dissolution time) obtained for different indentation loads should superpose perfectly to each other (cf. figure 2) providing a proper load dependent rescaling factor is applied to both imprint depth and dissolution time. The 10 mN load is arbitrarily chosen as the reference load,  $P_1$  is therefore the load of the data set considered for rescaling. The result of such a data treatment is exposed in figure 2. One can note that from 250  $\mu$ N to 10 mN all the data points follow the same trend within a  $\pm 5\%$  error. This allows for a more accurate computation of the intersection between the two regions of the curve (i.e. the increase of the imprint depth and the plateau). This gives access to the depth beneath the indentation imprint at which the pristine glass is retrieved or the thickness of the plastic zone ( $d_d$ ) under the indentation imprint according to [8]. Because of the hypothesis of geometrical self-similarity the size of the plastic zone may be computed for any load. Further more the ratio  $h_d/h_{max}$  where  $h_d = h_o + d_d$  may be computed and directly compared to finite element simulations.



**Figure 2 : Silica Glass, Berkovich indentation :Evolution of the rescaled imprint depth as a function of rescaled dissolution time  $t$ . The load dependent rescaling factor is  $\sqrt{P_1}/\sqrt{10}$ . ( $P$ , mN) All the curves were re-scaled to the 10 mN one.**

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