STUDY OF THE GLASS DEFORMATION REGIME TRANSITION USING INDENTATION AND SCRATCH TEST

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Abstract

The scratch tests are often used to study the friction and the wear phenomena thus to identify locally the materials mechanical proprieties. In this context, our study aims to contribute to the understanding of the glass deformation regime transition using the scratch and the indentation. The characterization of the scratching resistance and the comprehension of the scratching damage on the basic of structural considerations and mechanical analysis were done. Tests of progressive linear scratching and instrumented indentation, were performed on two glasses nuances using different indenters and environments. The obtained results show a similarity of the scratch behavior of the two glass types by comparison of their deformation. However, the obtained rupture critical load of the borosilicate and the soda lime glasses were about 4.16 N and 5.34 N respectively. In addition, the humid environment has a notable effect on the deformation regime in scratching.

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Keywords: Glass, Hardness, Indentation, Scratch

1- INTRODUCTION

The surfaces mechanical characterization constitutes today a major importance for the development of certain industrial sectors, thus for example optical industry [1] and auto industry [2] are, for evident reasons very imploring of anti-scratch films. More materials are covered of thin films which allow, inter alias reasons, to improve the surface mechanical properties, the measurement of these mechanical properties is generally difficult, and one of the used tests for this purpose is the scratch test. In order to analyze this type of test mechanically; it is important to simplify the problem while trying to understand, at first the scratch behavior of a massive material, thus the interest will be for the mechanics of the scratch test of any material. In addition, since the brittle materials grinding and polishing as the ceramics and the glasses are necessary for the precision instrumentation improvement, their study using scratch test is possible. Indeed, recently it was shown that brittle materials as glasses can be finished without causing of rupture, this process called grinding in ductile mode [3]. In this process, understanding of the material removal can be do using the scratch tests, to explain the abrasive grains actions on the polished or ground surface [4]. Glass is a material particularly sensitive to the surface damage by indentation and scratch. This mechanical damage results not only in the visible deterioration of the surface and its optical properties, but also by the structure failing (lens, window…) which can appear catastrophic under load. The glasses scratching resistance study is interesting to understanding the causes generating the optical losses and the windows or lenses rupture caused by the surfaces damage during their use. In addition, the glasses service duration depends usually on their surface damages which propagate dramatically until rupture. The damages are generally in the scratches or abrasions form, thus, the scratch process study appears suitable to understand and thus prevent glasses surface damage. However, in spite of the technological and scientific importance of the surface mechanical damage, as well on the level of the glass object realization, as with those of handling and use duration, the subjacent mechanisms stay unknown. In addition, the scratching problem is few studied. This can certainly because of its complexity and its apparent technological character. Some works have studied the relationship between the oxide glasses composition and their physical properties and thermomechanics, as the density, thermal dilation coefficient, elastic model, Poisson's coefficient, hardness [5] and tenacity [6]. However, the correlation between surfaces damages resistance by scratching and the glasses properties [7], [8], their compositions [8], [9] or environment effect [7], [10] is not well established and only some articles were interested. In this work a practical simulation was done using the scratch and indentation tests to reproduce the action of the grains on the glass surface and to study its mechanical behavior, especially the deformation regime transition since it is the most important phenomenon responsible of the mechanical action in the glass finishing process.

2- EXPERIMENTAL STUDY

2.1- Materials and characterization

The experimental tests were done using two types of glass; the soda lime and borosilicate glass. The chemical composition illustrated in table 1 was determinate by the X fluorescence for the soda lime glass and the bolometric technique for the borosilicate glass.

Table 1. Chemical composition of the glass samples

Éléments %	Soda lime	Borosilicate
SiO_2	72,851	69,734
Al_2O_3	1,354	2,549
Na_2O	12,729	6,761
K_2O	0,478	3,071
CaO	8,249	1,508
MgO	4,097	--
Fe_2O_3	0,098	--
B_2O_3	--	12,086
ZrO_2	--	0,399
SO_3	0,151	0,033
BaO	0,007	2,318
TiO_2	0,469	--
ZnO	--	1,115

The samples were prepared in parallelepiped shape which dimensions are about 40×20×5 mm^3 for the soda lime glass and 40×20×3 mm^3 for the borosilicate glass. The two glasses were treated at a temperature about 530°c for 45 minutes. Young module was measured using the impulse excitation technique (IET) (type RFDA-HT1750), where samples of 3×4×45 mm^3 in dimensions were prepared. The tenacity of the two types of glass was obtained on samples having rectangular section of 3×4 mm^2 using the single edges V-Notched Beam method. The tenacity was calculated using the formula presented in the study [11]. The samples resistance was done by three points flexion technique using the universal machine (type Zwick Z100) where the distance between points was 15mm.

In addition to the tenacity and the elastic module, the micro hardness, the refractive index and the density were measured. The mean results of five measurements in any measured characteristic are illustrated in table2.

Table 2. The measured characteristics of the glasses samples

	Soda lime	Borosilicate
$Hv_{0,5}$	639±10	760±12
IET E(GPa)	74±2	63±2
K_1c ($MPam^{1/2}$)	1,056±0,20	0,818±0,11
Refractive index	1,517±0,01	1,473±0,01
Density (g/cm^3)	2,46±0,02	2,23±0,02
Tg (°c)	560±2	592±2

2.2- Indentation and scratch device

The indentation and scratch tests were performed using the instrumented indentation device (type MCT, CSM instruments, Switzerland) [12]. The device is very precise, it’s used to determine the mechanical proprieties of the thin films, the hardness and the elastic module of the softest, hardest, most ductile and brittle materials, and the scratch tests. The indentation and scratch tests were performed using Vickers and Rockwell indenters respectively. The Rockwell indenter diameter was about 0,2mm.

During the indentation tests the charge was fixed at 5 and 10 N. The loading was maintained 30 s. The loading and unloading rate was about 2 N/min. The scratch tests were done in progressive mode; the applied normal force was fixed between 0,3 N and 20N and the loading speed was about 19,7 N/min. The scratch length was about 5 mm, where the sample movement direction was parallel to the surface, at a constant speed fixed at 5mm/min.

In order to study the influence of the test environment on the glass behavior, scratch tests were performed in two different environments (dry and humid). The humid environment was created by adding of distilled water on the contact surface (indenter/glass). The results reproducibility was verified by the tests repetition for 5 times and mean results were considered.

3- RESULTS AND DISCUSSIONS

3.1- Instrumented indentation tests

The penetration depth variations according the load of the two kinds of glass are illustrated in figure1. The results were obtained during indentation test done using the maximal force fixed at 10 N. The results show that the airs under the curves are different according to the glass nature. Indeed, it is more important in the case of soda lime glass, this can be explained by the fact that under the same indentation load, the soda lime glass plastic deformation energy is higher than the borosilicate [13]. We notice that the residual penetration depths after unloading of the soda lime and borosilicate glasses are respectively 5,8 µm and 4,9 µm. The beginning slope of the unloading indentation curve is used to determine the elastic module of material [14], [15]. Because of the curves difference of the two tested glasses, the elastic modules of the soda lime and the borosilicate glasses are 76,20 ± 6,62 GPa and 60 ± 5,5 GPa respectively. The comparison of the obtained results and those obtained by the resonance method (IET) show that the latter are more reproducible with lower than 2% of dispersion. Nevertheless, the first elastic module values remains acceptable. Indeed, the instrumented indentation is based on the measured in extremely sensitive location to the local defects (microscopic cracks), however, the resonance method (IET) provides values on all the sample volume. The technique of instrumented indentation gives highly reliable results of hardness because of the very weak standard deviations of the measured values. The found Vickers hardness for the two glasses is about 6,58±0,062 GPa and 7,91±0,045 GPa for the soda lime and borosilicate glasses respectively.

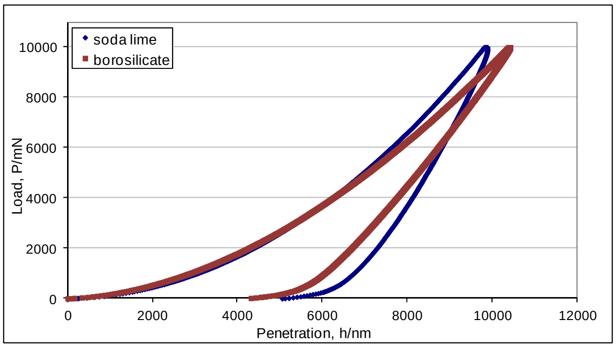

Figure 1. Indentation curve of the two glasses.

3.2- Scratch tests

3.2.1- Influence of the load

To understand the load influence on the surface damage behavior during scratch, tests were done with progressive load. Theoretically, during the Vickers indentation tests, the cracks occurrence depends strongly on the applied load and this one can appear during the loading or unloading [14]. The obtained results of the progressive mode scratching performed on the two types of glass (see fig.2) show the existence of three different damage modes:

a. Micro-ductile mode I: Characterizing the scratching beginning in which permanent traces are created without visible damages. The optical observation reveals the presence of residual plastic stripe only (plastic ploughing) (see fig. 2a).
b. Brittle mode II: When the normal load reaches the critical load of cracks initiation ($Fc_{I/II}$), the first circular crack (closed Rings) is formed in the back of the contact zone on the scratch axis. The micrographics in figure 2b show important damages in the form of side cracks reaching the surface and the radial cracks (chevrons cracks), these results are in concordance with those obtained in the study [9].
c. Micro abrasive mode III: As the normal load reaches a certain load called critical load of rupture ($Fc_{II/III}$), the scratches deformation passes from the brittle damage mode to the micro abrasive mode characterized by the presence of many debris and small emerging side cracks (Chipping) (see fig. 2C).

In order to determine the critical rupture load of the glass, figure 3 illustrates the variation of the acoustic emission according to the normal applied force for the soda lime glass. Results show rushed increases in the acoustic emission corresponding to an applied load about 5,34 N which is corresponding to the critical load ($Fc_{I/II}$) of the passage to the brittle mode II. This phenomenon can be explained by the principle that the rupture or the chipping of the glass surface generates discreet acoustic emission with great amplitude produced by the rough release of stored elastic energy. These increases indicate the rupture beginning of the glass and the passage to the brittle mode II. The critical force can be also determined by the microscopic visualization of the scratches, by determining the position of the first radial cracks and the corresponding load.

The progression of the normal applied load contributes to the damages increasing. When the applied load reaches 9,51N the acoustic emission attain its maximum (99,5%), this indicates the rapture critical force ($Fc_{II/III}$) value which is responsible of the complete rupture of the soda lime glass surface.

In addition, the variation of the apparent friction coefficient (Ft/Fn) versus the normal applied force during the scratch tests is illustrated in figure 3. It is noticed that the apparent friction coefficient decrease according to increasing of the normal force. This can be explained by the fact that the evolution of glass scratch resistance force (tangential force) is less important than the normal force which decreases their ratio.

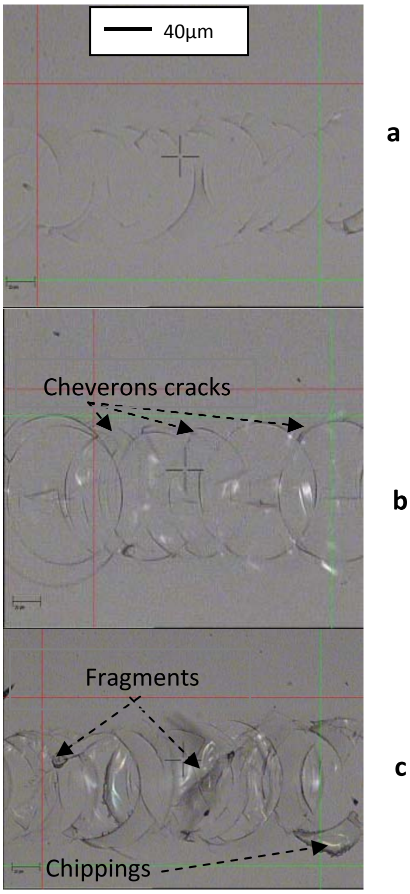

Figure 2. Scratch micrographics of the sodalime glass.
a-Micro-ductile regime, b-Brittle Régime, c- Micro-abrasive regime

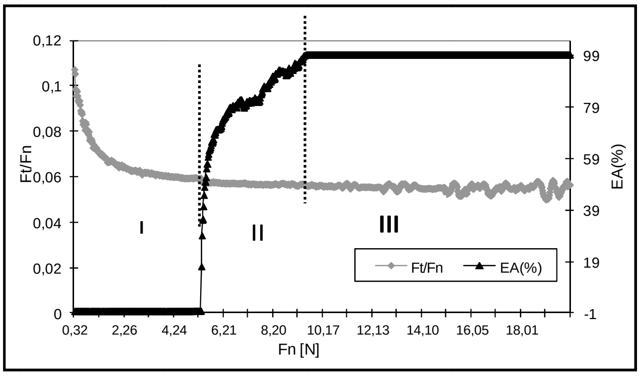

Figure 3. Variation of the acoustic emission and the force ratio vs the applied normal force of the soda lime glass

The penetration depth variation is shown in figure 4. It is shown that during the application of lower load the penetration depth is weak which can be explained be the fact that the soda lime glass deformation is purely elastic and an almost complete covering results just after the indenter extraction. As the normal load progresses, the penetration depth evolves until its maximum value about 8,80µm at the end of the test.

However, when chipping occurs, the scratch depth and the tangential force are affected by a typical oscillatory behavior; these oscillations can be related to the progressive accumulation of the damages inside the scratch according to the normal load increasing. The consideration of the inflection point on the penetration depth curve, as a sign of the transition from the deformation process to the rupture one, allow the determination of the rupture critical load graphically [16].

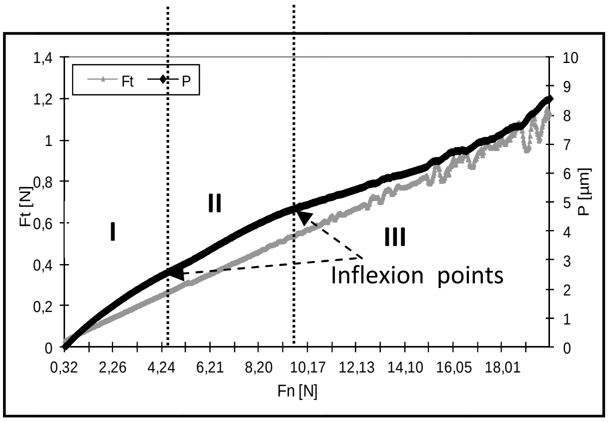

Figure 4. Variation of the tangential force and the penetration depth vs the normal applied force of the soda lime glass.

3.2.2- Effect of the glass chemical composition

The figure 5 presents a comparison of the scratch behavior of two glass types. The quantitative damage evaluation shows that the residual penetration depth of the soda lime glass is slightly larger than that of borosilicate glass, which are respectively 8,8 µm and 8,2 µm. Nevertheless, the inspection of the acoustic response during the two scratch tests illustrate that the borosilicate glass damage appear for lower applied loads. The results are in concordance with those of the tenacity of two glasses measured by the SEVNB method (see tab. 2). This difference of results between the two glass types can be due to the difference of their chemical composition and their mechanical behavior.

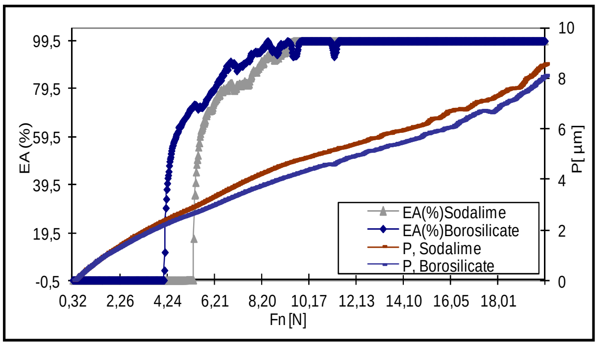

Figure 5.Variation of the acoustic emission and the penetration depth vs the normal applied force of the soda lime and borosilicate glasses.

3.2.3- Scratches interaction

Scratches interaction is a phenomenon which always exists in several situation especially when during the glass surface finishing (lapping and polishing) [4]. In order to highlight this phenomenon, adjacent scratches about 50 µm were performed on the two glass nuance surfaces (see fig.6), the scratches were repeated five times in each type of glass.

The micrographics show the existence of an interaction between the radial cracks of adjacent scratches at the end of the test on the borosilicate glass surface. However, the phenomenon was not noticed during the soda lime glass scratching which can be due to its great tenacity relatively to the borosilicate glass. It is also noticed that the mutual effect of adjacent scratches is not only on the surface but also generate significant difference damage mode.

Table 3 presents the average critical loads values of the four adjacent scratches obtained for the two glasses. It is noted that the adjacent scratches were made in order (A, D, C, B).

It was noted that the mode transition load are smaller in the case of borosilicate glass and the scratches critical loads B and C are smaller than those of A and D, which can be due the constraints caused by these latter.

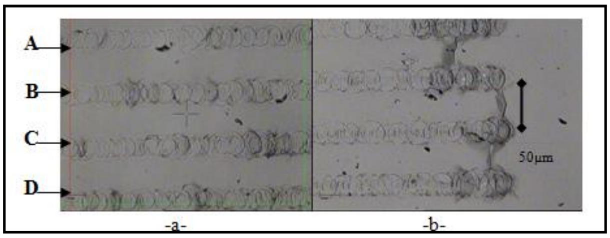

Figure 6. Micrographics of the scratches interaction.
a- Soda lime glass, b- Borosilicate glass
Scratch order: scratch A, scratch D, scratch B, scratch C

Table 3. Load of the damage regime transition of adjacent scratches.

	Soda lime		**Borosilicate**	
	FcI/II	**FcII/III**	**FcI/II**	**FcII/III**
Scratch A	5,31±0,18	9,52±0,66	4,12±0,20	8,41±0,79
Scratch B	4,78±0,43	8,12±0,41	3,74±0,73	7,58±0,41
Scratch C	4,44±0,65	8,31±0,18	3,86±0,69	8,52±0,59
Scratch D	5,5±0,26	9,73±0,74	4,19±0,26	8,63±0,84

3.2.4. Effect of the test environment

The results of the force ration during scratch tests performed in two different environments are shown in figure7. The results illustrate that the weaker force ration is obtained in the humid environment. This can be explained by the fact that the existence of the water can react chemically with the glass. This reaction produce a hydrated layer on the glass surface which decreases its hardness and then the indenter penetration is easer which results a weaker tangential force (the scratch resistance force) [4]. The regime transition load evolves according to the hygrometry rate. Indeed, the damage, the chipping and the micro abrasion regimes appears at higher normal loads when the hygrometry rate decreases [19]. Results in figure 7 permits the deduction of the soda lime glass critical loads, which are about 5,34 N ($Fc_{I/II}$) and 9,52 N ($Fc_{II/III}$) in the dry environment and about 4,25 N ($Fc_{I/II}$) and 7,82 N ($Fc_{II/III}$) in humid environment.

The scratches morphology in the two environments was characterized using the mechanical profilometer Form Talysurf (Taylor-Hobson Ltd). The micrographics are shown in figure 8. It is noticed that the scratches damages in humid environment are bigger in dimensions as those created in dry environment. These results traduce the penetration depth difference which was found about 8,8 µm for the scratches created in dry environment and 19,25 µm for those found in humid environment.

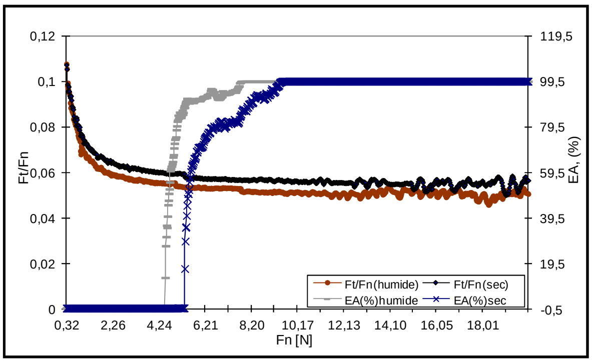

Figure 7. Variation of the force ratio and the acoustic emission vs the normal applied force during scratch of soda lime glass in dry and humid environments.

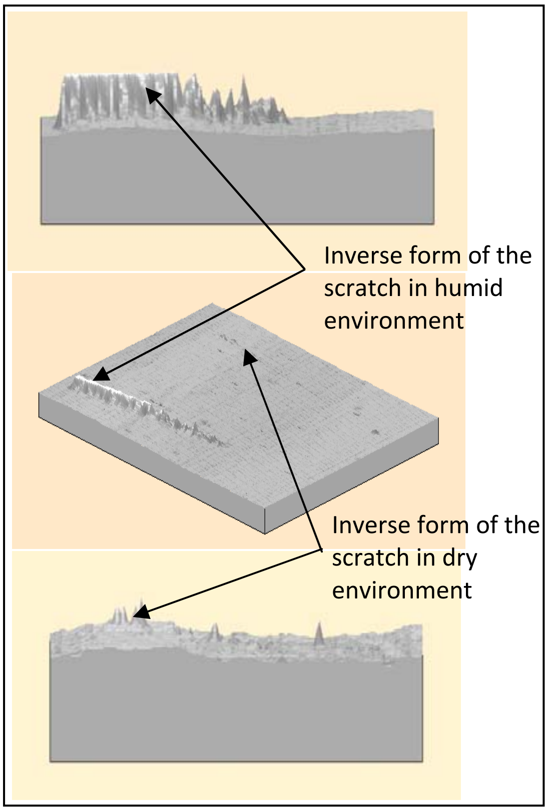

Figure 8. Mechanical profilometer micrographics of scratches performed in dry and humid environment on the soda lime glass.

4- CONCLUSION

The mechanical behavior of two glass nuances was investigated to explain the glass deformation regime transition using the scratch and indentation tests. The influence of various parameters has been briefly studied and some conclusions were drawn. The glass deformation nature changes by a transition from a micro-ductile regime to a brittle regime followed by a micro abrasion. The last two regimes are more damaging the glass which disrupts it mechanically. The nature of the glass has not a significant influence on its mechanical deformation during its indentation or scratching. However, the glasses critical loads decrease according to their tenacity. The interaction between radial cracks is very common when the scratches are relatively adjacent, and develops and facilitates the transition between damage regimes of the second scratch at lower loads which can be related to the constraints caused by the neighboring scratches. The scratch test environment has a significant effect on the deformation degree which is more important in the humid environment. The damage regime transition is also possible at lower loads than the dry environment case. This result explains the ability and the lower working time of the borosilicate glass during its finishing relatively to the other kinds of glass.

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