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HF etching effect on sandblasted soda-lime glass properties

M. Kolli [a,*], M. Hamidouche [a], N. Bouaouadja [a], G. Fantozzi [b]

[a] *Laboratory of Non Metallic Materials, DOMP, Faculty of Engineering Sciences, University of Setif, Setif 19000, Algeria*
[b] *Laboratory MATEIS (UMR CNRS 5510), Bat. Blaise PASCAL, INSA, 20 Avenue Albert Einstein, Villeurbanne 69621, France*

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Abstract

Our objective in this work is to study the HF etching chemical treatment effect on the mechanical and optical properties of soda-lime glass eroded with 200 g fixed sand mass. We followed the evolution of these properties in relation to the chemical attack duration.

The results show a clear improvement of the measured properties. The strength of the eroded samples is 44.23 ± 0.91 MPa. It increases up to 57.73 ± 1.76 MPa after 15 min of treatment and reaches 181.43 ± 23.69 MPa after 1 h. This last value is much higher than the as received glass strength (117.5 ± 10.48 MPa). The optical transmission of the eroded samples is about 18.5%. During the first 2 min of the chemical treatment, an important drop of the optical transmission (12%) was observed. However, improvement of the transmission was achieved for longer chemical treatment durations. After 8 h of treatment, the optical transmission increases up to 57%. Microscopic observations show that the HF attack causes the opening and the blunting of the surface cracks. In general, the surface state is improved during the chemical treatment.

Keywords: Glass; HF; Strength; Optical properties

1. Introduction

The glass mechanical and optical properties are strongly influenced by its surface quality.[1,2] In their different applications in Saharan regions, ordinary glass products (glazes, cars windshields, solar panels protecting glass, ...) are exposed to inevitable sandblasting effect caused by frequent sandstorms. The surface flaws induced by sand particles impacts lead to a deterioration of the glass strength and optical transmission.[3–7]

Glass strengthening has been abundantly studied. The numerous methods that were cited in literature are based on different concepts.[8] Thermal strengthening[9–11] and ionic exchange[12,13] make the surface flaws under compressive residual stresses. An applied loading on a strengthened glass by such methods has to overcome the residual stresses before putting the surface in a tensile state and eventually reaching a critical stress leading to fracture.

Coating methods, usually used for obtaining specific glass characteristics (optical, electronic, ...), can also be used for strengthening damaged glasses.[14–17] Not only they strengthen the glass by flaw filling, they constitute a protection against eventual damages (mechanical, chemical, ...) as well.

The etching technique using hydrofluoric acid (HF) is also well-known as an efficient way for strengthening glass.[18–21] HF etching of silica leads to the formation of the hexafluorosilicic acid in accordance with the following chemical reaction[22–24]:

$$\underset{(s)}{SiO_2} + \underset{(l)}{6HF} = \underset{(1)}{SiF_6H_2} + \underset{(1)}{2H_2O} \quad (1)$$

Another product that can result from the HF etching of silica at moderately high temperature (~60 °C) is the tetrafluorosilicate gas:

$$\underset{(s)}{SiO_2} + \underset{(1)}{4HF} = \underset{(g)}{SiF_4} + \underset{(1)}{2H_2O} \quad (2)$$

The HF glass etching enables to reduce the surface cracks length and blunt the crack tips.[19,20,25] After a sufficient etching period, the cracked layer can be entirely removed. In this case, the glass strength will depend more on eventual bulk flaws or damages caused by the presence of the chemical reaction products.[19] For an indeterminate etching duration, the chemical dissolution will end when the HF acid is entirely converted to

* Corresponding author.
E-mail address: kolmus_eulma@yahoo.fr (M. Kolli).

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Table 1
Mean chemical composition of the glass used.

Oxides	SiO_2	Al_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	Fe_2O_3	Others
% Mass	69.14	1.77	8.33	3.97	13.2	0.83	0.69	0.20	1.87

Table 2
Glass main physical and mechanical characteristics.

Characteristics (units)	Values
Poisson's ratio	0.22
Transition temperature (°C)	530
Density (g/cm^3)	2.45
Refraction index	1.52
Strength by concentric biaxial bending (MPa)	117
Young's Modulus (GPa)	72
Linear expansion coefficient (C^{-1})	8.9×10^{-6}

the hexafluorosilicic acid and/or tetrafluorosilicate gas.

In the present work, we examined the effect of HF etching on the strength and the optical transmission of a glass previously damaged by sandblasting.

2. Experimental procedure

2.1. Glass characteristics

A soda-lime sheet glass of 4 mm thickness was used for our experimental tests. Its chemical composition and its main physical and mechanical characteristics are respectively given in Tables 1 and 2. Samples of square shape (50 mm × 50 mm) were cut from the same glass sheet. In order to relax any eventual residual stresses, they were submitted to an annealing treatment at 550 °C during 15 min with a heating temperature rate of 3 °C/min and a cooling temperature rate of 2 °C/min.

2.2. Sandblasting tests

The glass samples were eroded with a sand coming from the region of Ouargla (south of Algeria). Fig. 1 shows a sample of grains from the used sand. We can notice that the shape is quite

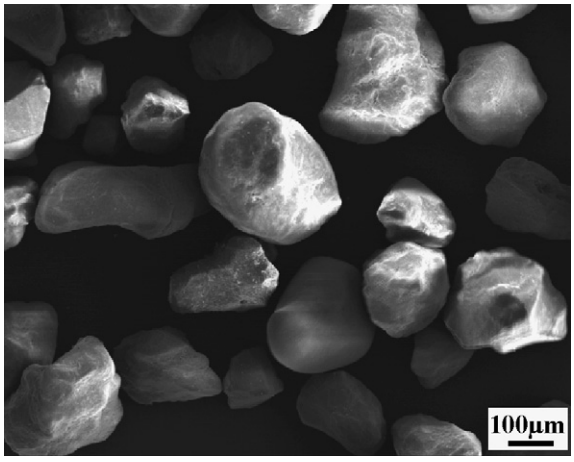

Fig. 1. Sample of sand grains used for sandblasting experiments.

Table 3
The granulometric distribution of the used sand.

D (µm)	<125	125–280	280–355	335–450	450–500	>500
Mass (%)	7.17	76.44	11.58	3.75	0.64	0.40

variable (quasi spherical, sharp, elongated, ...). We therefore can expect two types of contact between the projected sand particles and the glass surface: one caused by sharp particles that can be assimilated to Vickers indentation and the other caused by nearly spherical particles comparable to Hertzian indentation. In fact, generated flaws observed on surface are in general much more complex because of the variable grains shape and the interactions that occur between close singular flaws.

The sand granulometric distribution was evaluated on a sample of 100 g using a series of sieves. The obtained distribution is presented on Table 3. We can see that the greatest part of the grains (∼76%) is within the diameter interval (125–280 µm).

Mineralogical analysis showed that sand grains contain essentially quartz and tourmaline and to a lesser extend other minerals such as *ilmenite*, limonite, gypsum.[3] The sand Vickers microhardness measured by Bousbaa et al.[3] on a sample of 30 grains with a load of 0.6 N is 14.49 ± 3.28 GPa. This relatively high value is probably related to the high hardness of the main mineralogical constituents (quartz, tourmaline). In literature, much dispersed silica sand hardness values were reported: 11 GPa by Feng and Ball,[26] ∼13 GPa by Shipway and Hutchings[27] and Wheeler and Wood[28] and 35.3 GPa by Yabuki et al.[29]

The sandblasting operation was undertaken with a horizontal type sand blower apparatus. Fig. 2 shows a schematic representation of the used equipment. A known quantity of sand is placed in the sand hopper equipped with a flow rate control device. During the tests, the sand is projected by air flow on the target surface at a predetermined incident angle with a controlled velocity.

The chosen experimental parameters are presented in Table 4. These conditions led to a sharp decrease of glass strength and optical transmission whose values are respectively 44 MPa and 18.5% in comparison to those of the as received glass (117 MPa and 91.5%). This strongly deteriorated state enables to better

Table 4
Sandblasting tests conditions.

Parameters	Values
Impact angle (α)	90°
Projected sand mass (Mp)	200 g
Projection velocity (V)	16 m/s
Sand mass rate	1.6 g/s
Distance tube-target (x)	100 mm

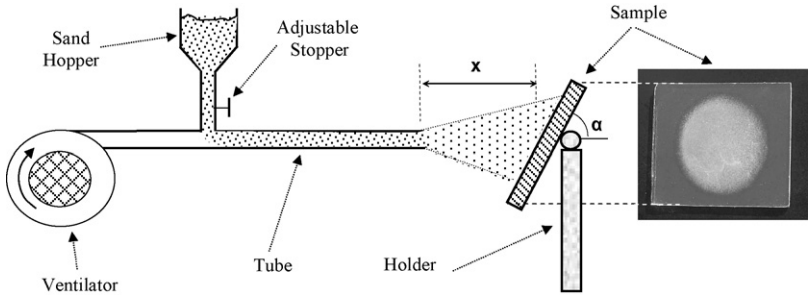

Fig. 2. Schematic representation of the used sandblasting apparatus.

detect any improvement on the glass properties after a chemical treatment with the HF acid.

2.3. Chemical treatment

Every sandblasted glass sample was immersed in a solution containing 150 ml of hydrofluoric acid (HF 5%) for variable periods up to 8 h. The chemical reaction occurs at ambient temperature and atmospheric pressure without stirring. After every chemical treatment, the samples were washed with distilled water and dried at a temperature of 60 °C in a stove before their characterization.

2.4. Samples characterization

The strength characterization was made by concentric biaxial bending tests (Fig. 3). This technique presents numerous advantages in comparison with the 3 and 4 points bending. It enables to avoid the edge flaws effect and to determine therefore the intrinsic strength. The samples were prepared in a squared or circular shape without worrying about polishing the edges.

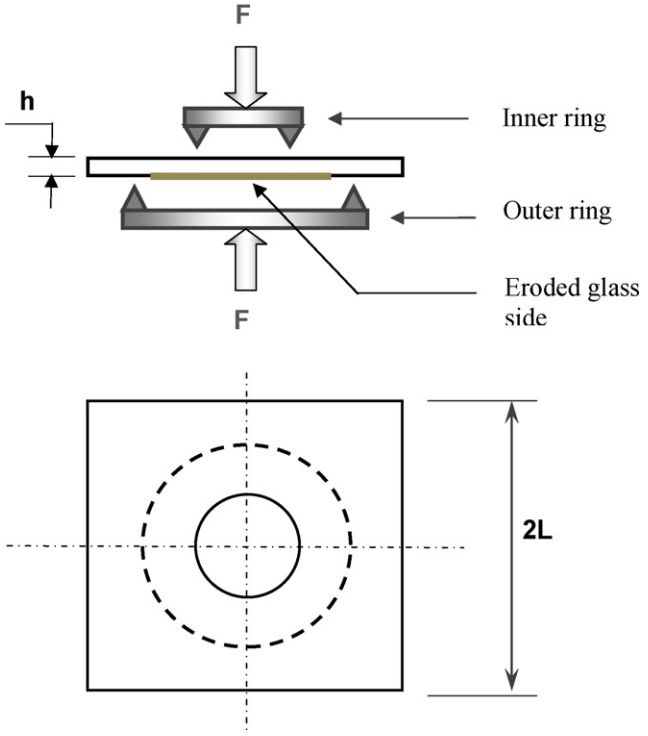

Fig. 3. Principle schema for bending test using concentric rings.

For our tests, we used inner and outer rings having diameters of 16 mm and 36 mm respectively. The eroded surface is put on the tensile side (Fig. 3). The loading rate was maintained constant for all tests at 1 mm/min. The reported values represent the average values of three tests for each glass state.

The strength was evaluated according to the following relation[30,31]:

$$\sigma = \frac{3F_R}{2\pi h^2}\left[(1+\upsilon)ln\frac{r_1}{r_0} + (1-\upsilon)\frac{r_1^2 - r_0^2}{2r_2^2}\right] \quad (3)$$

where F_R is fracture stress, υ is glass Poisson's ratio (0.22), h is sample thickness (4 mm), and r_0, r_1 are the inner (8 mm) and outer (18 mm) rings radius.

$$r_2 = \frac{(1+\sqrt{2})}{2}L \approx 1.21L \quad (4)$$

where $2L$ is squared sample side (50 mm).

The samples optical transmission was measured on a MD 100 Carl-Zeiss-Jena type microdensitometer using white light.

The microscopic observations on the eroded and treated surfaces were undertaken on two types of microscopes: a scanning electronic microscope (SEM) and a laser scanning microscope (LSM). Besides the morphological observations, the laser scanning microscope can also be used for measuring the roughness without any physical contact with the examined surface.

3. Results and discussion

3.1. HF reaction kinetics

The HF acid reaction with glass is surface controlled occurring at the solution–solid interfaces.[23] Fig. 4 shows the variation of the dissolution rate and the weight loss per unit area (g/mm^2) with the attack duration. We observe that the weight loss increases continuously with time and that the reaction kinetics decreases. During the first 2 min of treatment, a high dissolution rate (3.1×10^{-4} g/mm^2 h) was recorded. It was followed by a sharp drop before tending toward weak variations. In the last 3 h (between 5 and 8 h) the mean dissolution rate decreases (0.58×10^{-4} g/mm^2 h). Tso and Pask[32] noticed in their work

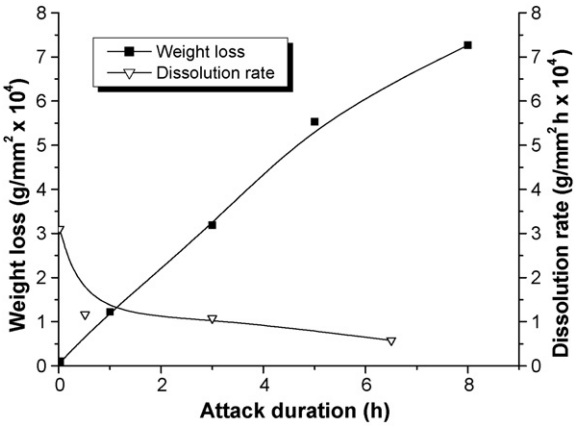

Fig. 4. Weight loss and dissolution rate variation versus HF (5%) acid attack duration.

on silica glass discs that the reaction rates are more important at the test beginning than at the end. They explained this fact by the presence of micro-cracks generated during the discs cutting operation. At the beginning, the reacting contact surface is important and as a result, the reaction rate is more pronounced. The reaction continues at a slower rate when the cracked layer is removed.

In order to verify this effect, we treated for comparison an as received glass (undamaged) in the same conditions during 1 h. The weight loss for the undamaged glass (0.09697 mg/mm^2) is actually less important than for the eroded glass (0.12266 mg/mm^2). The sandblasted specimens showed higher weight losses as a result of the larger surface area due to the presence of surface sandblasting flaws in accordance with what was suggested by Tso and Pask.[32]

A second probable cause for the slowing reaction kinetics is the diminishing concentration of the HF acid related to the formation of hexafluorosilicic acid according to reaction (1).

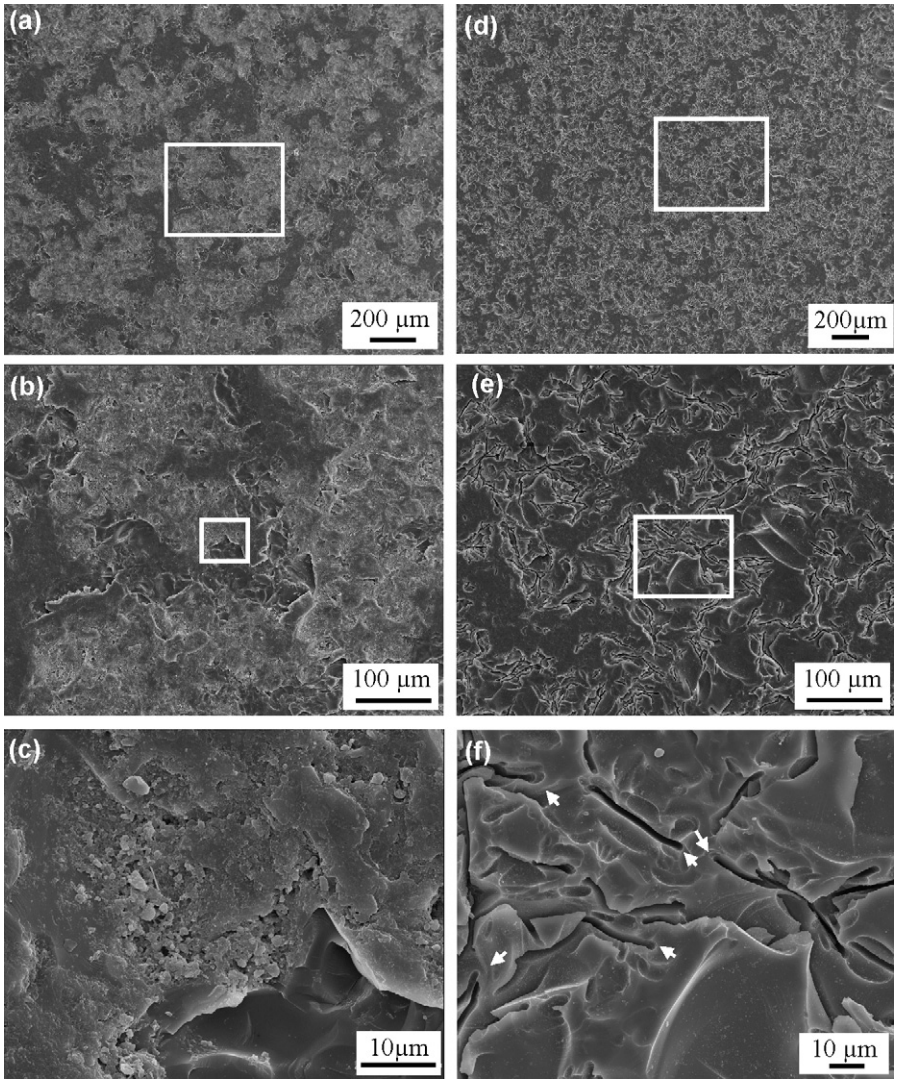

Fig. 5. Micrographs of sandblasted glass (a) and sandblasted glass treated during 1 min in HF acid (d). Micrographs of Fig. 5b and e represent a detailed view of the localized zones within the rectangles in Fig. 5a and d. Micrographs of Fig. 5c and f represent also a detailed view of the localized zones in Fig. 5b and e. The small arrows in Fig. 5f show examples of cracks blunting.

3.2. Sandblasting flaws morphological changes

The sandblasting flaws morphology evolution was followed by microscopic observations on SEM and LSM. The micrographs presented on Fig. 5 show an area in the central zone of the eroded glass before treatment (Fig. 5a) and after 1 min treatment in the prepared HF solution (Fig. 5b). We can notice that the untreated glass is characterized by homogeneously distributed damage flaws. These damage flaws appear as scaling zones resulting from successive and interacting sand grains impacts

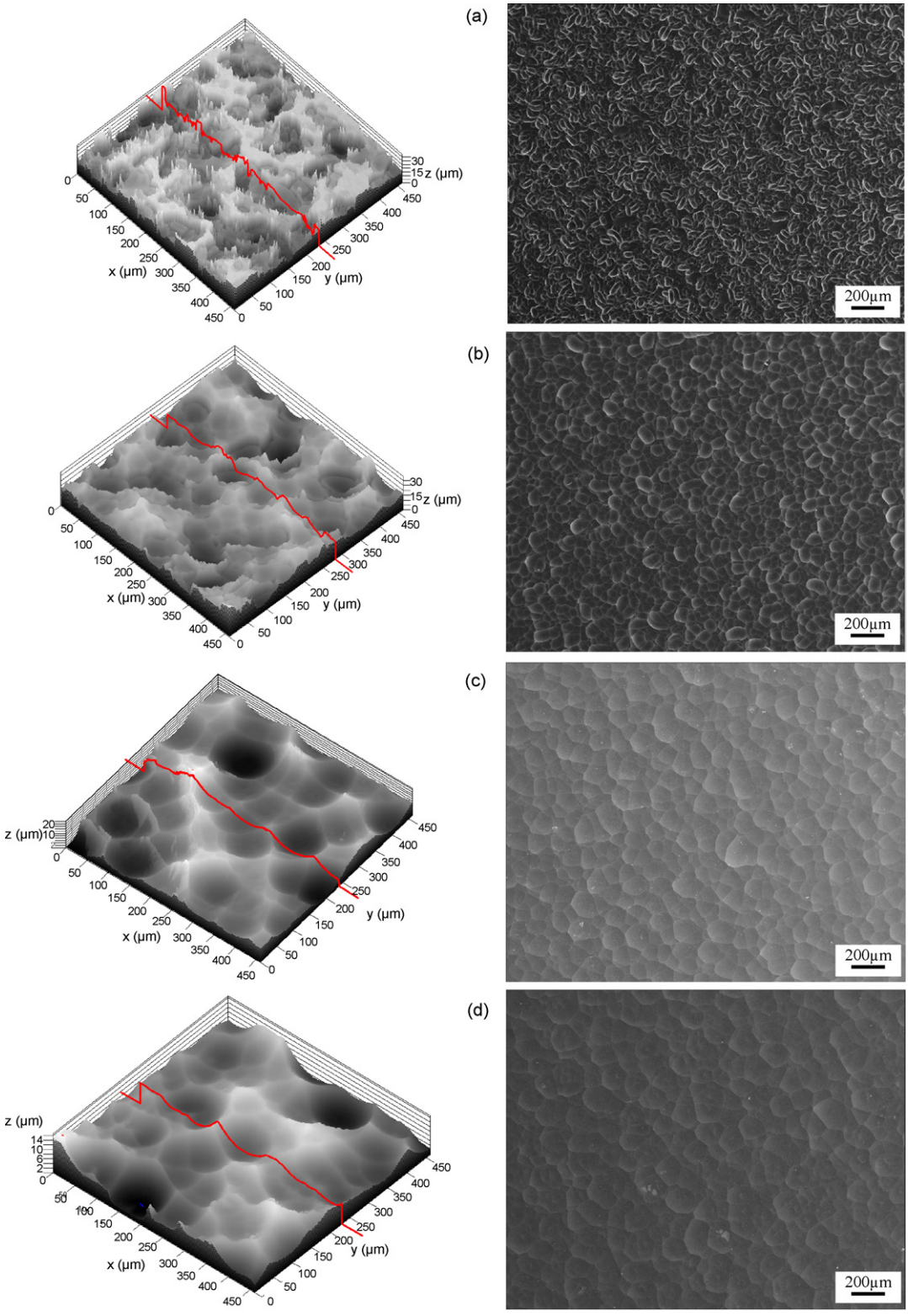

Fig. 6. Microscopic observations of sandblasted glasses and treated in HF acid during different durations: (a) $t = 2$ min, (b) $t = 1$ h, (c) $t = 5$ h, and (d) $t = 8$ h.

on the glass surface. Near these zones, we also observe tiny particles adhering on the eroded surface. Those could be glass fragments or sand dust. After 1 min treatment, many superficial cracks appear extending in depth and characterized by large openings of about 1 μm. These cracks were not visible before the chemical treatment. The HF acid attack is in fact well-known for making visible surface flaws that are otherwise initially undetectable.[18] These revealed micro-cracks present blunted crack fronts (Fig. 5c) that have an influence on the glass strength as we will see further.

After 2 min of treatment, we observe elongated and randomly oriented grooves (Fig. 6a). Those are the result of the chemical attack on the apparent randomly oriented micro-cracks (Fig. 5d). We also observe that the dissolution of the crests near these surface grooves lead to hollows clustering appearing as larger and nearly homogeneous craters. As the attack progresses these craters evolve toward quasi spherical shape caps (Fig. 6c and d). This shape is similar to what was observed by Tso and Pask[32] on the edges of cut glasses treated during a few hours.

The observed craters depth diminishes with the treatment duration whereas their apparent diameters slightly increase (Fig. 6). Therefore, the surface flatness of the treated glass improves. This has a positive effect on the roughness and the optical transmission as we shall see in the following section.

3.3. Roughness and optical transmission

Fig. 7 shows the variation of the optical transmission and the roughness (mean arithmetic Ra and total Rt) in relation to the HF acid attack duration. We can observe that the roughness values increase sharply at the beginning of the chemical attack. They reach a maximum (Ra = 5.456 μm and Rt = 38.609 μm) and decrease continuously beyond. At the beginning of the chemical attack, we observed a surface state degradation in a form of glass scales removal and surface cracks opening. This led to more surface irregularities, and therefore to much important roughness. After a maximal value the roughness decreases as a consequence of the diminishing disparity between crests and hollows as the reaction progresses. One hour treatment led to roughness values (Ra = 2.073 μm and Rt = 16.895 μm) close to those of untreated sandblasted glasses (2.135 μm and 18.475 μm).

The optical transmission of the as received glass (undamaged state) is about 91.5%. In general, at normal incidence, the transmitted light energy fraction for an as received glass does not exceed 92%. Each of the two glass sides will cause a loss of 4% by reflection.[33] After glass sandblasting with 200 g of sand, the generated flaws diffuse light and induce a decrease of the optical transmission down to 18.5%. During the first 2 min of chemical treatment, the transmission drops sharply to 12%. This period corresponds to the important roughness increase. Beyond this minimum, we observe a regular increase of the optical transmission. A treatment of 8 h made the transmission increase up to about 57%. This value is much higher than the optical transmission of the untreated sandblasted glass (18.5%).

In Fig. 8, we presented photographs to underline the glass transparency evolution with different treatment durations. As a reference (Fig. 8a), we observe that the characters LMNM are clearly visible through an undamaged glass. After a sandblasting with 200 g (Fig. 8b), the characters become totally blurred. They disappear entirely after the first 2 min of chemical treatment in HF acid (Fig. 8c). They increasingly reappear with the treatment duration in Fig. 8d, e and f for respectively 1, 3 and 8 h.

3.4. Strength

The strength variation of the treated sandblasted samples is presented in Fig. 9. The undamaged glass has a strength of about 117.5 ± 10.48 MPa. It is about 44.23 ± 0.91 MPa when the glass is sandblasted prior to any treatment. Dabbs and Lawn,[18] in their work on an indented soda-lime glass (0.25N) that was chemically treated in a HF/H_2SO_4 solution, observed a strength drop prior to the strengthening effect caused by the chemical attack starting after nearly 2 min. In our case, the strength starts almost immediately to increase from the beginning of the HF attack. After 15 s, we recorded a strength value of 57.73 ± 1.76 MPa. We can notice a sharp increase of about 300% after 1 h of treatment. This strength improvement is related to the cracks blunting as shown in Fig. 5f. The strength variation curve continues to increase slowly afterwards. The

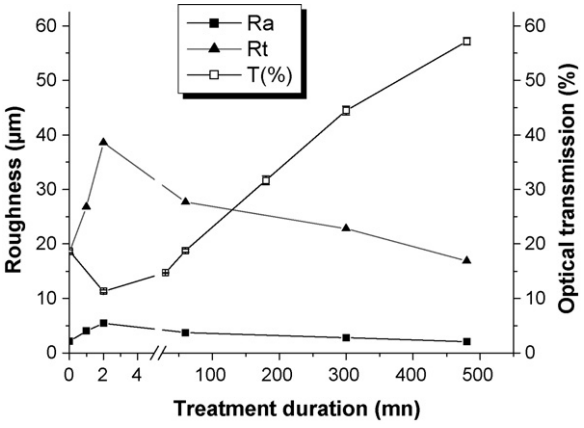

Fig. 7. Roughness and optical transmission variation with HF (5%) acid treatment duration.

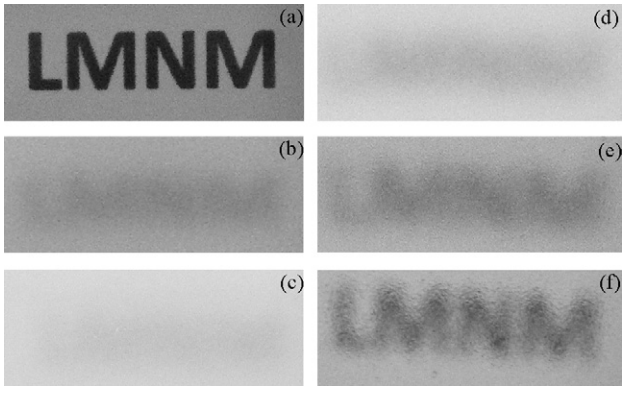

Fig. 8. Transparency of six glass samples: as received (a); sandblasted (b); sandblasted and chemically treated with HF respectively during 2 min (c), 1 h (d), 3 h (e) and 8 h (f).

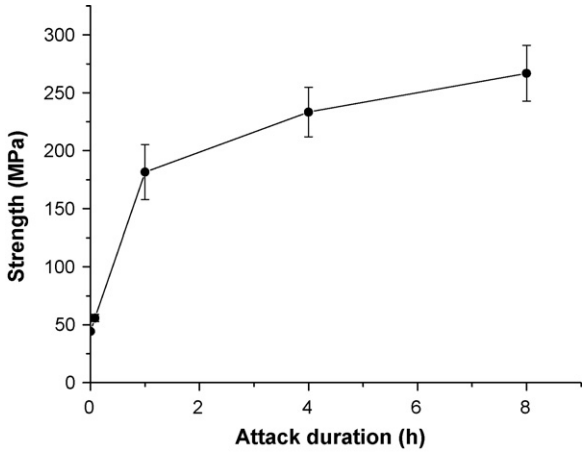

Fig. 9. Strength variation versus HF acid treatment duration.

strength improvement is limited by the bulk defects and the probable effect of the reaction products on the surface as it was suggested by Sglavo et al.[19] However, the strength values reached with the HF treatment are much higher than the initial strength of the as received state (117.5 ± 10.48 MPa).

3.5. Schematic diagram of the glass flaws evolution during chemical treatment

According to the observations made on the samples surface and the obtained results, we proposed the following diagram (Fig. 10). This diagram shows schematically the sequential evolution of the glass defects during the HF chemical treatment. The effect can be described by the two main sequential steps: An initial step of surface state degradation characterized by scaling removal, cracks opening and blunting, followed by a second step of surface flatness restitution.

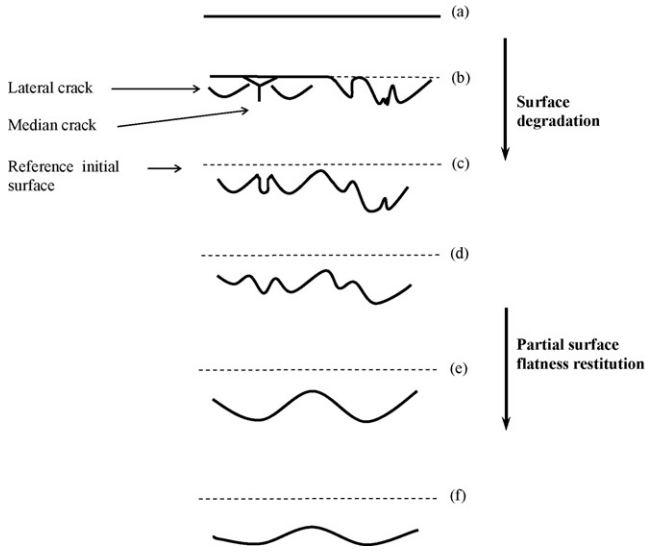

Fig. 10. Schematic representation of the sandblasted glass surface state evolution during the HF chemical treatment: (a) as received glass; (b) untreated sandblasted glass; (c), (d), (e) and (f) sequences of sandblasted and treated glasses during increasing durations $t1$, $t2$, $t3$ and $t4$.

4. Conclusion

On the basis of the obtained results, we can conclude with the following remarks: During the HF acid treatment of the sandblasted glass, we observed important morphological changes on the surface flaws generated by sandblasting. Accordingly, the strength and the optical transmission of the treated glasses were affected. At the beginning of the treatment, we assist to a surface deterioration characterized by scaling and surface cracks opening. This caused an increase of the surface roughness and a decrease of the optical transmission. As the treatment progresses the disparity between the crests and the surrounding grooves diminishes resulting into larger and less deep hollows. The improvement on the surface flatness leads to a reduction on the surface roughness and an improvement of the optical transmission. Surface cracks blunting is considered to be the main cause of the strengthening effect observed on the treated glasses from the beginning of the treatment.

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