

# Influence of Acid Frosting on the Microstructure Characteristics and Mechanical Strength of Eroded Soda-Lime-Silica Glasses by Sandstorm

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## Article history

Received: 27-02-2019

Revised: 12-04-2019

Accepted: 25-04-2019

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**Abstract:** Most glass products such as cars windshields, solar panels protecting glass and windowpanes are exposed to unavoidable sandblasting effects especially in areas prone to sandstorms. This sand impingement results into micro cracks and surface flaws which altered the structure and strength of the exposed glasses thereby lowering their service life. In this regard, attempt was made in this research to investigate the influence of acid frosting technique as a means of surface modification on the microstructure characteristics and mechanical strength of eroded soda-lime-silica glass. Several glass samples measuring 80x60 mm were initially prepared and exposed to sand impingement from a sandblasting machine with an air flow speed of 15m/s and an impact angle of 90°. The sandblasted glass samples ( $S_{\text{control}}$ ) were later immersed in Hydrofluoric Acid (HF) for acid frosting procedure at dipping period of 5, 10, 15 and 20 minutes respectively. Microstructure characteristics were examined using scanning electron microscopy while flexural strength test was conducted to assess the mechanical strength of the sample glasses. The results showed that mechanical strength of the etched-sandblasted samples (S+E series) improve significantly as the dipping period increases compared with sandblasted glasses. In term of microstructure, homogenously distributed micro-cracks and numerous flaws were observed on the eroded glass samples while for the S+E series, the stress riser effect of these micro-cracks are well eliminated which accounts for the strength improvement.

**Keywords:** Glass, Sandblasting, HF Etching, Microstructure, Micro-Cracks

## Introduction

Glass is a general term which is commonly given to any solid that exhibits transition temperature near its melting point and that lacks long range structural order (Uhlmann, 1980). Soda-lime-silica glass otherwise known as commercial glass is the most commonly used glasses which constitute over 70% of world's glass products due to their ease of production, low cost and broad area of applications such as buildings, automobiles, domestic vessels, headlamps, solar panels protective glass, drinks and beverages containers. In theory, glass is a very strong but brittle material with a theoretical strength value of about 7GPa but in service applications the actual strength value is far lower, failing around 100 MPa (Garza-Mendez *et al.*, 2007). This wide margin observed between the estimated theoretical strength of glass and its

actual strength in service has been attributed to various surface flaws, defects and micro-cracks inherent within the glass matrix (Macrell, 2001; Green *et al.*, 2003).

Most commercial glass products such as cars windshields, headlamps, windowpanes, window panels and solar panels protective glass are often exposed to unavoidable sandblasting effects especially in area prone to sandstorms phenomena (desert regions). Continuous exposure to sand impingements induces surface flaws and micro-cracks which causes deterioration of the glass surface. This deterioration of the glass surface thus alters the Microstructural integrity, reduces the overall mechanical strength property and light transmittance of the glass (Bousbaa *et al.*, 2003; Bouaouadja *et al.*, 2000).

Several works have been studied and reported on the erosion of soda-lime glasses by sandblasting effects in order to explain and model sandstorms impingement on

glass materials (Bouaouadja *et al.*, 2000; Owoeye *et al.*, 2015; Lawn *et al.*, 1997; Wada, 1991) while investigations have been reported on various methods of strengthening soda-lime glasses (Shutov *et al.*, 2002; Master, 1989; Castacon and Gonokhovskii, 2003; Sglavo *et al.*, 1993). However, not many works have been reported on strengthening of sandblasted soda-lime glasses with the aim of eliminating the deteriorating effects resulting from sand impingement (Kolli *et al.*, 2009; Bousbaa *et al.*, 2003). In this regard, this research aims at investigating the influence of acid frosting on the microstructure characteristics and mechanical strength of eroded soda-lime-silica glasses.

## Materials and Methods

### Materials

The starting materials used for this work are flat soda-lime glass with 4 mm nominal thickness, sand particles and hydrofluoric acid (HF 48%). The soda-lime-silica glass was commercially obtained in its as-received state while its mean chemical composition, physical and mechanical behavior were based on industrial standard recommended for a typical soda-lime-silica glasses. Sands with size range of 0.1–0.3 mm and particle shapes ranging from spherical to angular peculiar to most eroding sand particles were gotten from beach area typical of sand generated during sandstorms in Nigeria. Hydrofluoric acid solution was obtained in its pure state from Beta Glass Industries in Nigeria.

### Sandblasting Process

Several glass samples measuring 80×60 mm were initially cut from the as-received flat soda-lime-silica glass and were prepared for sandblasting process. The sandblasting process was carried out using a horizontal sand impingement apparatus in accordance with Yabuki *et al.* (1999) as shown in Fig. 1. Glass samples were fixed respectively at the sample holder section of the sand blower apparatus and were exposed to sand

influx (20 g/min) from the machine operated at air flow speed of 15m/s and impact angle of 90°. The glass samples were exposed to the eroding sand particles until the whole surface was covered. A highly blurred appearance was observed due to surface roughness and micro-cracks resulting from sandstorm effects.

### Chemical Treatment (Acid Frosting)

Samples were selected from all the sandblasted glass samples and were immersed in a solution containing 200 ml of hydrofluoric acid (HF 48%) at varying immersion period of 5, 10, 15 and 20 minutes respectively. This acid frosting reaction was carried out in a fume cupboard under room temperature (as shown in Fig. 2). After completion of each immersion period, the glass samples were taken with a pair of laboratory tong and rinsed thoroughly with distilled water. The thoroughly rinsed etched-sandblasted glass samples were later dried at ambient temperature prior to characterization.

### Characterization

Tests such as microstructure examination and flexural strength were carried out in this work to evaluate the microstructure characteristics and mechanical strength of the glass samples respectively. The microstructure examination was investigated using gold coated samples by high performance Phenom Prox Scanning Electron Microscopy (SEM) in order to assess the morphology features. The flexural strength test was carried in relation with ASTM D790 standard at room temperature (25°C) by a three-point bending strength test using Instron universal testing machine (Instron 3369, 50 KN load capacity) operated at a strain rate of 5 mm/min, and a support radius of 2 mm. Load was applied on the sample to obtain a stress rate increase of 0.2 N/mm sec<sup>-1</sup> until fracture and force obtained at the point of fracture was noted. The tests were conducted in triplicate to establish the reliability of the results.

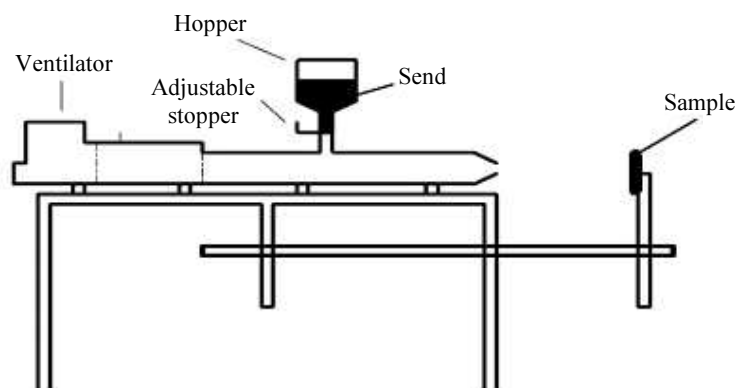


Fig. 1: Sand impingement set up according to Yabuki *et al.* (1999)



**Fig. 2:** Acid etching process in fume cupboard

## Discussion

### *Microstructure Characteristics*

Representative microstructures of the sandblasted glass sample ( $S_{\text{control}}$ ) and etched-sandblasted glass samples (S+E series) are presented in Fig. 3-5 respectively. Representative Secondary Electron (SE) images were used since there is no much difference in the morphology features across the S+E series. Figure 3 which represents the morphology of sandblasted glass samples, it was observed that the glass showed a highly damage flawed surface structure which are homogeneously distributed within the glass matrix, micro-cracks and few deteriorated portions were also observed in the glass matrix. These damage flawed and deteriorated portions observed in the glass matrix might be attributed to impact of impingements from eroding sand interacting with the glass surface according to Kolli *et al.* (2009). Particles were also seen adhering to the glass surface which might be due to sand dusts or glass fragments as stated by Kolli *et al.* (2009).

Figure 4 presents sandblasted glass samples etched for 10 minutes ( $S+E_{10}$ ), it can be observed that numerous grain boundaries are seen in the microstructure which can be attributed to the ability of the etchant to encapsulate the surface flaws, blunting and reduction in the length of micro-cracks created by sandblasting. This encapsulation of the surface flaws created numerous surface compressive stresses which are uniformly

distributed through the glass matrix. This surface compressive stress has been identified to improve the strength properties of a glass matrix and which has also been reported by Sanya *et al.* (2018).

Figure 5 which represent the morphology of the glass samples etched for 15 minutes, it can be observed that the microstructure showed similar features as shown in Fig. 4 when the sandblasted samples was etched for 10 minutes. However unlike Fig. 4, the induced surface stresses are more which are homogeneously distributed and the grain boundaries are closer. These features account for the higher strength properties observed at that point than the rest of the samples.

### *Mechanical Strength*

The results of the flexural strength of the untreated glass and the treated glass samples are shown in Fig. 6. It can be observed that the strength of the normal glass (untreated) sample drastically decreased by 57.5% from 16.520 MPa to 7.019 MPa (for  $S_{\text{control}}$ ). This drastic reduction in strength can be attributed to presence of damaging flaws, micro-cracks and deteriorated surface observed on the sandblasted glass samples resulting from the eroding impact of sandstorms interacting with the glass surface (Bousbaa *et al.*, 2003; Madjoubi *et al.*, 1999; Bouaouadja *et al.*, 2000; Owoeye *et al.*, 2015). However, it will be observed for S + E series that there is significant increase in the strength of the glass samples when the sandblasted samples were treated in HF acid (acid frosting) for 5, 10, 15 and 20 minutes respectively.

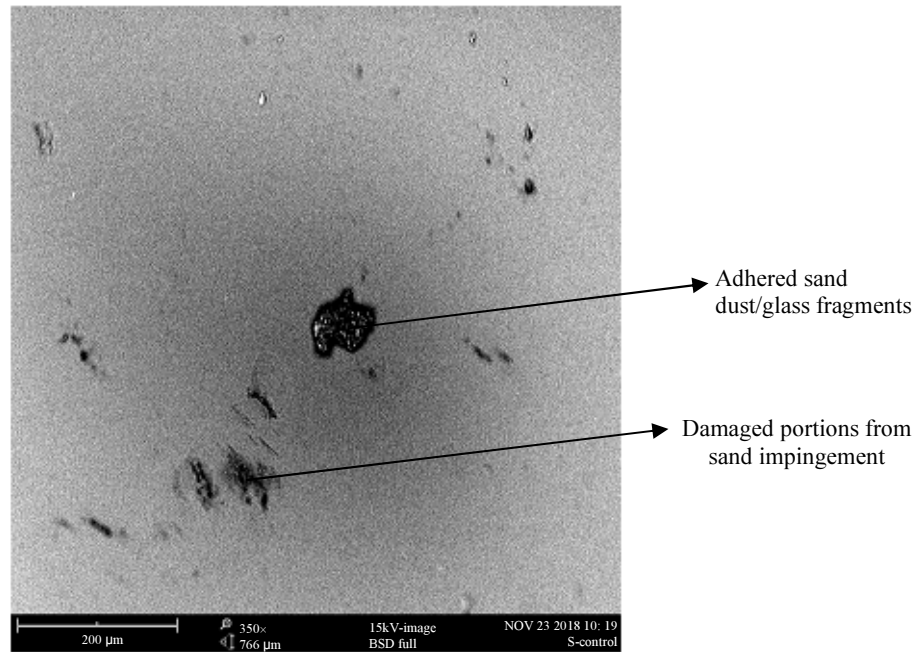


Fig. 3: Representative SE image of the sandblasted glass samples ( $S_{\text{control}}$ )

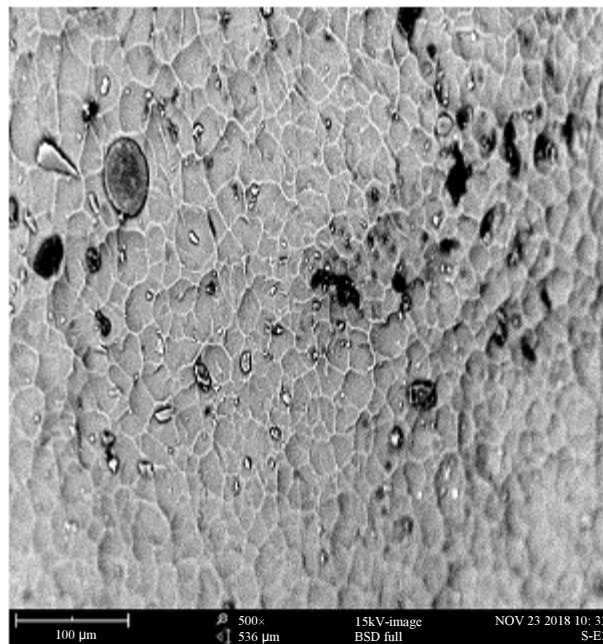
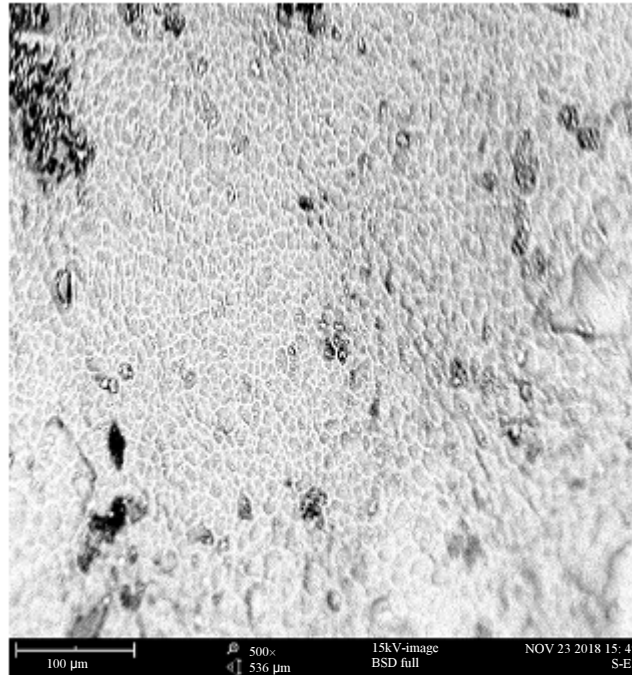


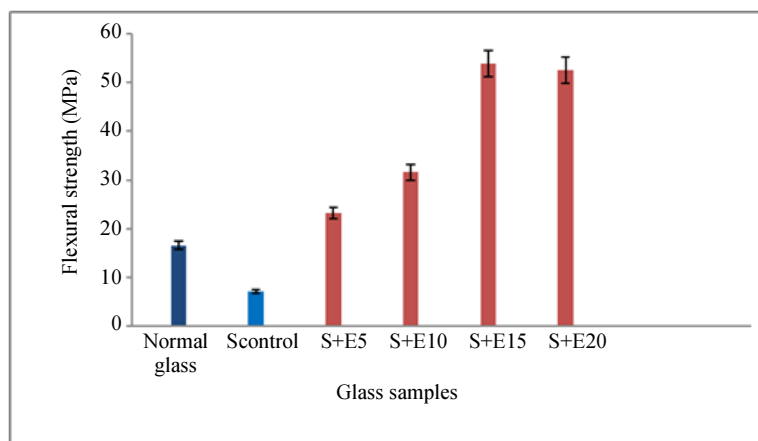
Fig. 4: Representative SE image of sandblasted glass etched for 10 minutes ( $S+E_{10}$ )

This significant increase in strength can be attributed to the ability of the etching fluid (HF acid) to encapsulate surface flaws, sealing micro-cracks and reducing the length of the micro-cracks initiated by the sand impingements on the glass from sandstorm process. The strength improvement can also be attributed to the surface compressive stresses observed on the glass matrix resulting from acid frosting process (Kolli *et al.*, 2009). These surface

compressive stresses create barriers to further propagation of micro-cracks and flaws extension in the glass matrix. However, for sand blasted glass samples etched for 20 minutes ( $S+E_{20}$ ), a slight drop in strength of about 2.4% was observed which might indicate that at longer period of acid frosting the acid can in turn be attacking the glass matrix and resulting into re-opening of micro-cracks or the slight decrease could be as a result of measurement errors as reported by Kolli *et al.* (2009).



**Fig. 5:** Representative SE image of sandblasted glass etched for 15 minutes (S+E<sub>15</sub>)



**Fig. 6:** Flexural strength of the untreated and treated glass samples

## Conclusion

The influence of acid frosting on the microstructure characteristics and mechanical strength of eroded soda-lime-silica glasses by sandstorm was investigated. The results show that:

- Sandstorm has a negative effects on the properties of soda-lime glasses especially when used in area prone to such phenomenon
- The strength of the glass samples reduced drastically by 57.5% when exposed to sandstorms simulated by sandblasting operation
- There is significant improvement in the strength of the S+E glass samples and which increased as the immersion period increased
- In terms of morphology, surface flaws, micro-cracks and deteriorated portions characterized the microstructure features of the glass samples exposed to sand impingement from sandblasting while for S+E glass samples these surface flaws and micro-cracks were eliminated to a minimum level due to the surface compressive stresses initiated by the etching fluids creating a barrier to their progression within the glass matrix

## Acknowledgement

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

## Author's Contributions

All the Authors make considerable contributions to conception and design of this work and also give final approval of the version submitted and revised version.

## Ethics

The Authors declare no conflict of interest and that this work has not been published previously anywhere.

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