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Vincent Guiheneuf, Fabien Delaleux, Olivier Riou, Pierre-Olivier Logerais, Jean-Félix Durastanti. Investigation of Damp Heat aging on soda-lime glass for photovoltaic applications. 32nd European Photovoltaic Solar Energy Conference and Exhibition, Jun 2016, Munich, Germany. 10.4229/EUPVSEC20162016-5BO.11.6 . hal-01375930

**HAL Id: hal-01375930**

**<https://hal.science/hal-01375930>**

Submitted on 3 Oct 2016

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# INVESTIGATION OF DAMP HEAT AGING ON SODA - LIME GLASS FOR PHOTOVOLTAIC APPLICATIONS

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**ABSTRACT:** Damp heat test was performed on commercial soda-lime glass to characterize functional properties of glass in photovoltaic applications and to define aging mechanisms. In addition to the measurements of optical properties, FTIR, SEM-EDX and XPS analyses were carried out. The damp heat exposed samples presented an increased transmittance in UV, visible and near-IR ranges. The results were explained by hydration of glass network with heat. Molecular water adsorption involves a leaching process of network modifier cations and especially sodium ions. Hydrolysis also occurs in silica network with free molecular water reaction. This phenomenon enhances concentration of hydroxyl groups in glass highlighted by FTIR measurements and promotes formation of silanol groups. The tin side dependence on sodium leaching which acts as a passivating barrier for ionic transport is emphasized which would be worth taking into consideration for photovoltaic module design.

**Keywords:** soda-lime glass; photovoltaic; damp heat; aging; leaching

## 1 INTRODUCTION

A photovoltaic (PV) module consists of a stack of different kinds of materials: glass, polymer (e.g. ethylene vinyl acetate for encapsulation and polyvinyl fluoride for backsheet) and photovoltaic cell. Glass plays a major role in most of the photovoltaic systems such as substrate and cover. The glass front side of photovoltaic panels is exposed to many environmental stresses (UV radiation, heat, humidity, shading, abrasion by dust, snow load, etc.) depending on the localization and the weathering. Jordan et al. reported degradation rates in their publications. The averages of reported rates for Si and thin-film technologies are of 0.7%/year and of 1.5%/year respectively [1]. It is found that the main issue is caused by failure of cell interconnects, solder bonds or bypass diodes [2]. Other challenges concern the transmittance changes because of encapsulant discoloration, delamination at glass/encapsulant interface and anti-reflection coating degradation [3-4]. The aim of the present work is to investigate the influence of damp heat test on the properties of commercial float soda-lime glass. Efforts were made to characterize the functional properties of glass used in PV applications (i.e. transmittance) and to understand the degradation mechanisms of glass by physicochemical analysis.

## 2 EXPERIMENTAL

A commercial soda-lime glass was considered for this study (see table I) with a 2 mm thickness without texture nor coating. Aging accelerated tests were performed in an environmental chamber (MKF 240 model from Binder) under damp heat conditions, i.e.  $85^{\circ}\text{C} \pm 1^{\circ}\text{C}$  with 85% r.h.  $\pm 5\%$  for up to 1000 h. The glass samples were installed in a vertical position to similarly expose both their sides.

**Table I:** Composition of glass samples (at.%)

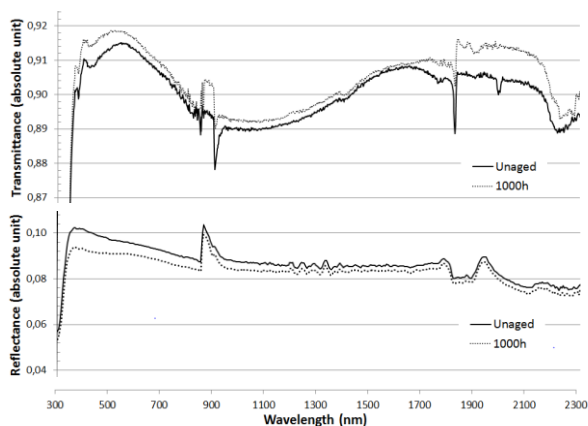
Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	SiO <sub>2</sub>	Others
0.7	9.0	3.5	11.9	74.4	0.5

The aging behavior was characterized by several methods which can be divided into optical and chemical techniques. Transmittance and reflectance measurements

were performed by using a spectrophotometer (Perkin Elmer Lambda 750 UV/Vis/NIR) equipped with an integrated sphere of 60 mm diameter. The absolute accuracy was assessed at 0.002 transmittance or reflectance units. Infrared spectra were obtained by averaging 32 scans taken with a Fourier transform infrared (FT-IR) spectrophotometer (Perkin Elmer Frontier) in Attenuated Total Reflectance (ATR) mode. The chemical measurements were carried out by using analytical scanning electron microscopy (ASEM, Jeol JSM 6301-F) with energy dispersive X-ray fluorescence (EDX). Due to the electrical insulating behavior of the glass, charge neutralization was made by platinum sputtering. SEM analysis provides chemical information within 1  $\mu\text{m}$  depth. The accuracy is less than 0.5%. Moreover, two glass samples (unexposed and after 1000 h under DH) were analyzed by X-ray Photoelectron Spectroscopy (XPS) using VersaProbe (PHI 5000) Scanning XPS microprobe with a monochromated Al K <sub>$\alpha$</sub>  anode ( $h\nu=1486.6$  eV, 200  $\mu\text{m}$  diameter). Some survey scans were performed on each sample in order to check the homogeneity of the surface composition. Next, one detailed scan was realized on each sample to obtain accurate quantitative analysis. The probed depth is less than 10 nm which allows to analyze the near-surface layer of glass.

## 3 RESULTS

Fig. 1 exhibits the transmittance and reflectance spectra of glass samples before and after 1000 h of damp heat treatment. A significant increase in the optical transmission after 1000 h of damp heat exposition in the 300–2300 nm wavelength range can be observed. This fact is interesting for photovoltaic conversion of Si cell, typically in the 400-1100 nm range. Nevertheless, the transmittance increase in the UV range could accelerate the polymer degradation. Hence, the efficiency of the PV device would be diminished. Concerning reflectance measurements, the behavior is opposed but stays in the same proportion. So, the absorption of glass samples remains unchanged after this accelerated aging.

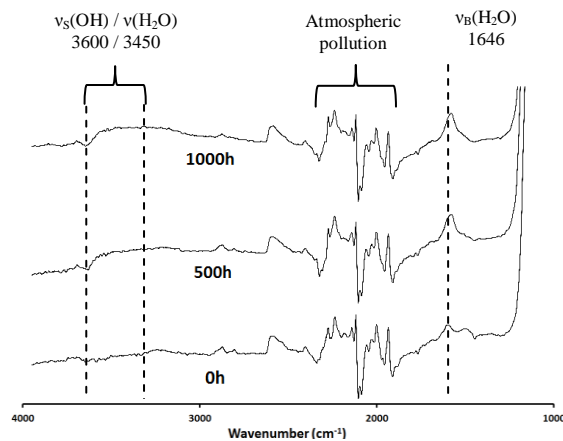


**Figure 1:** Transmittance and reflectance of glass sample before and after 1000 hours of damp heat treatment.

Fig. 2 shows the superposed FTIR spectra of each sample to visualize shifts of the characteristic peaks. By studying infrared reflectance spectra, structural changes can be noted during hydration of the three glass samples at different instants. The bending vibration of molecular water occurs at  $1611\text{ cm}^{-1}$  but it can be modified by hydrogen bond stretch [5]. The band at  $1646\text{ cm}^{-1}$  which is expected for molecular water ( $\nu_B(\text{H}_2\text{O})$ ), shows raised peak heights for samples after 500 and 1000 h under DH respectively. The peak shift towards lower wavenumbers can be explained by the weakening of the hydrogen bonds. The broad O-H absorption of silanol at  $3600\text{ cm}^{-1}$  is due to the specificity of hydroxyl groups that can be freed or hydrogen-bonded.

The chemical analyses of the glass bulk approximately within  $1\text{ }\mu\text{m}$  depth were performed by SEM-EDX to compare with those based on XPS analysis (see Table 2). Two kinds of results are displayed according to the Sn element concentration in near-surface glass composition for three samples: unaged, after 500 h and also after 1000 h under DH. On the tin side, SEM-EDX measurements reveal a leaching phenomenon of Na element during hydration. The  $\text{Na}_2\text{O}$  content decreases from 11.4 at.% to 9.9 at.% after 1000 h. The Na leaching does not appear linear because of the more pronounced drop in the Na content after the first 500 h contrary to the period between 500 h and 1000 h. The leaching did not occur for other intermediates ( $\text{Al}_2\text{O}_3$ ) or network modifiers (CaO, MgO) which can be explained by lower migration kinetics in glass. The mechanism is similar for

the no-tin side but in a more important proportion. The Na content decreases from 11.9 at.% for the unexposed sample to 9.2 at.% after 1000 h and here too, no significant change is observed for the other element. The tin side has a passivating barrier ability which reduces the sodium leaching and the glass network hydrolysis.



**Figure 2:** Infrared reflectance spectra of 3 glasses samples under damp heat conditions as a function of exposure time: 0, 500 or 1000 hours.

Table 3 shows the variation of intensity ratios of different chemical elements as a function of Si element for the non-aged or aged glass samples. The surface composition determined by XPS is above all very different from the bulk composition provided by SEM-EDX characterization. The Mg element is missing in significant proportions and so is the K element. It may be due to a high thickness dependent gradient of glass chemical composition. The strong presence of potassium could be explained by an assumed external and accidental pollution for example with KCl (see Fig.7). The Mg element is not detected although it was the third component in glass composition in the bulk analysis. One might expect its lack due to its good solubility in glass during float or annealing process. After this aging, there was a slight decrease in the surface Na concentration, of approximately 10 percent of the initial value. Concerning the  $I\text{ Ca}2p3 / I\text{ Si}2p$  and  $I\text{ K}2p / I\text{ Si}2p$  ratios, both are decreased in the same proportion. The  $I\text{ Sn}3d5 / I\text{ Si}2p$  ratio dropped off with regard to the initial ratio. The migration kinetics vary greatly according to the chemical

**Table II:** Variation of relative concentration (at.%) of components as a function of damp heat (DH) exposition duration obtained by SEM-EDX measurements

Side of float glass	DH exposition	$\text{Al}_2\text{O}_3$	CaO	MgO	$\text{Na}_2\text{O}$	$\text{SiO}_2$	SnO	Others: $\text{K}_2\text{O}, \text{Fe}_2\text{O}_3$
Tin side	Unaged	0.6	9.1	3.4	11.4	73.3	1.9	0.3
	500h	0.6	9.0	3.5	10.5	74.3	1.8	0.3
	1000h	0.7	9.0	3.6	9.9	74.7	1.9	0.3
No-tin side	Unaged	0.7	9.0	3.5	11.9	74.4	0.2	0.3
	500h	0.7	8.9	3.6	10.6	75.8	0.2	0.3
	1000h	0.6	9.2	3.7	9.2	76.8	0.2	0.3

elements because the oxides have a variable chemical reactivity with molecular water. In comparison with SEM-EDX analysis, one can suppose that Na element has stronger mobility than Ca or Mg elements.

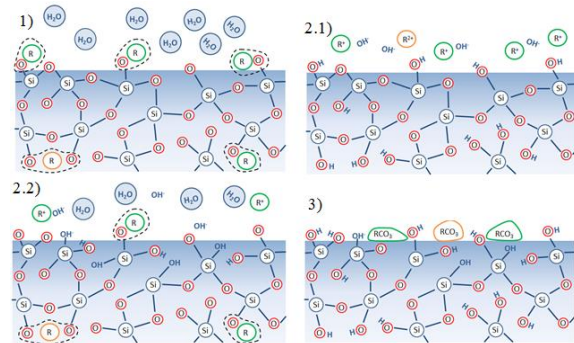
**Table III:** Variation of intensity ratios as a function of damp heat exposition by XPS measurements

Intensity ratio	Unexposed	After 1000h	Variation (relative unit)
I Al2p / I Si2p	0	0.01	-
I Ca2p3 / I Si2p	0.08	0.01	0.81
I K2p / I Si2p	0.10	0.02	0.81
I Na1s / I Si2p	0.20	0.18	0.11
I Sn3d5 / I Si2p	0.22	0.02	0.91

#### 4 DISCUSSION

The increase of the glass transmittance can be explained by the fact that molecular water penetrates into the glass. One might suggest that molecular water reacts with glass network as explained below and which is confirmed by FTIR, SEM-EDX and XPS analyses.

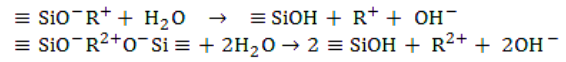
The study of glass aging during damp heat test also shows changes of surface and bulk chemistry representative of leaching process accelerated under a hot and humid atmosphere. The following model proposed by the authors and depicted in Fig. 3 is based on Schaut-Pantano's model [6]. The glass degradation mechanism can be described in three stages: 1) water gel film coating, 2) molecular water adsorption with reaction in glass and 3) corrosion and deposits of carbonates. Fig. 3 illustrates the following descriptions for each stage of glass degradation mechanism.



**Figure 3:** Aging mechanism of soda-lime glass under damp heat as described in Ref. [6]: 1) water gel film coating, 2.1) leaching of cations, 2.2) hydrolysis of glass network and 3) glass corrosion and deposits of carbonates.

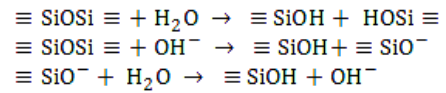
A water film coating is formed on glass during damp heat aging by condensation and adsorption of water vapor. In this water film, we can distinguish the physisorption for which water molecules bind on glass by means of Van der Waals forces and the chemisorption at the near water/glass interface for which chemical reactions take place with cations and silica to generate new specific chemical bonds. Then free molecular water diffuses through silica network. At this moment, two

types of chemical reactions can take place. The first one based on the reaction of molecular water with monovalent cations,  $R^+$ , like  $Na^+$  or  $K^+$  and divalent cations,  $R^{2+}$ , like  $Ca^{2+}$  or  $Mg^{2+}$  on non-bridging oxygen (NBO) sites. The cationic atoms are free and released apart from glass network to water film where they accumulate as soluble precipitates on a mixture of molecular water and hydroxide ions, which is the well-known mechanism of leaching:



The ionic migration aims to reduce the electrochemical potential between bulk and surface, so the pH will be raised in the liquid film during glass aging process.

But at the same time, molecular water also reacts with silica network on BO sites with three major chemical reactions to allow hydrolysis of silica network. They imply Si-O-Si or  $SiO^-$  groups with molecular water and hydroxide ions. The latter react within silica network providing a linkage of hydrogen bonds to form silanol groups:



When liquid film reaches pH 9, degradation of glass surface becomes more important with formation of precipitates of insoluble carbonates like  $Na_2CO_3$  and  $CaCO_3$ . The formation of carbonates involves a permanent staining which reduces the optical performance of top glass. However, transmittance measurements and visual inspection do not reveal permanent staining. The cationic leaching and hydrolysis of silica network provide the corrosion layer formation with lacunas left by leaching. The increasing porosity involves a most effective removal of soluble constituents, especially of network modifiers, which probably improves the transmittance properties. This fourth step has not been highlighted in the present study and it is certainly due to a restricted aging time.

#### 5 CONCLUSIONS

A set of technical characterizations was used to investigate the aging mechanisms of soda-lime glass under damp heat. The results suggest that the glass aging is induced by hydration of glass network during damp heat treatment. The network modifiers (Na, Ca, K) are more or less leached at the glass surface and corrosion involved by hydrolysis of Si-O-Si network occurs. A transmittance increase for optical properties is highlighted. This can have a positive impact on photovoltaic conversion. Nevertheless, the increase of transmittance in the UV range may enhance degradation of the screened materials (encapsulant, cell, back-sheet) and should be investigated in a future work.

It has also been shown that tin side of glass plate modifies the damp heat impact on functional properties. The tin side has a passivating barrier ability which reduces the sodium leaching and the glass network hydrolysis. It would be interesting to design a

photovoltaic module with tin side at glass/polymer interface. It induces a decrease of the cationic migration which could permit to avoid the devastating PID phenomenon [7].

## 6 ACKNOWLEDGEMENTS

Financial funding of the project was provided by the French government through the Ministry of Higher Education and Research (MESR). The authors would like to thank Mr. David Alamarguy and Mrs. Anne Migan-Dubois (GeePs Laboratory, France), for having provided us with the XPS analysis. This analysis was supported by the grants from Region Ile-de-France, CNRS and Agence Nationale de la Recherche. The authors are grateful to Messrs Ivan Cotrebil and Ivan Guillot (ICMPE Laboratory, France), for performing Vickers indentation cracks using a microhardness tester. Their thanks also go to Mr. Patrick Ausset (LISA Laboratory, France) for the SEM-EDX analysis and the interesting discussions on natural glass aging and to Mr. Laurent Ibos (CERTES EA 3481, France) for his help in FTIR measurements.

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