



Proton irradiation induced changes in glass and polyethylene terephthalate substrates for photovoltaic solar cells

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ABSTRACT

An alkaline earth boro-aluminosilicate glass (Eagle XG), a soda-lime glass, and a light-weight polyethylene-terephthalate (PET) foil, used as typical substrates for photovoltaics, were treated by an energetic proton beam (3 MeV, dose 10^6 – 10^7 Gy) corresponding to approx. 30 years of operation at low Earth orbit. Properties of the irradiated substrates were characterized by atomic force microscopy, optical absorption, optical diffuse reflectance, Raman spectroscopy, X-ray photoelectron spectroscopy, Fourier-transform infrared (FTIR) spectroscopy, and terahertz (THz) spectroscopy. Minimal changes of optical and morphological properties are detected on the bare Eagle XG glass, whereas the bare PET foil exhibits pronounced increase in optical absorption, generation of photoluminescence, as well as mechanical bending. On the other hand, the identical substrates coated with Indium-tin-oxide (ITO), which is a typical material for transparent electrodes in photovoltaics, exhibit significantly higher resistance to the modifications by protons while ITO structural and electronic properties remain unchanged. The experimental results are discussed considering a potential application of these materials for missions in space.

1. Introduction

Emerging solution processable photovoltaics (organic photovoltaics – OPV and perovskite photovoltaics) as well as other organic electronics are of high interest for space applications due to possibility to be produced by roll-to-roll (R2R) process on plastic substrates. The easiness of R2R production of solar cells and modules on plastic substrates guarantees light weight of the modules and cheap manufacturing process, which are the biggest advantages of emerging photovoltaic (PV) technologies compared to conventional photovoltaics. In addition, such devices are flexible, hence, easy to handle and more resistant to mechanical stress in demanding applications such as in space missions. Furthermore, due to simplicity of the manufacturing process, that is based on printing and coating, such solar cells can be produced on space stations. Despite these attractive features, lifetime and stability of OPV and perovskite devices for the use in space still need to be evaluated. When leaving the Earth's atmosphere, the devices are exposed to very harsh environment that includes ionizing irradiation from various sources. For satellites orbiting the Earth the major concern is exposure

to electrons and protons trapped in the Earth radiation belts [1]. An argument about the possibility of using OPV and perovskite PV in space, should be supported by the knowledge on the effect of the ionizing irradiation on the devices in general and on their components in particular.

The irradiation induced effects on photovoltaic devices have been studied many times since the 60s [2–5]. These investigations primarily discuss the results obtained on completed inorganic devices that are typically characterized by current-voltage measurements accompanied by distinct spectroscopy-based tests. Only limited results have so far been published on organic-based devices [6] or on the microscopic characterization of the devices after irradiation [7].

This work aims to contribute to filling this gap. It presents a study revealing the effect of the proton irradiation on the materials commonly used as substrates or superstrates for OPV (two types of glass and polymer foil) and comparison of the properties of untreated and irradiated materials. Thereby, the study lays foundation for further characterizations of complete OPV devices. To provide a complex insight into the material properties, the substrates were characterized by

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atomic force microscopy (AFM), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), optical spectral absorption, optical diffuse reflectance, Fourier-transform infrared (FTIR) spectroscopy, and terahertz (THz) spectroscopy. Similar tests and characterization were also performed on the same materials that are coated with indium tin oxide (ITO), which is typically used as a transparent electrode. In this case, additionally to the above methods, the samples were characterized by Kelvin probe force microscopy (KPFM) to monitor changes in ITO work function (WF_{ITO}) which is critical for optimal OPV device operation [8]. The experimental data are discussed in terms of material degradation considering their applications in space.

2. Experimental details

Alkaline earth boro-aluminosilicate glass (Eagle XG) purchased from Corning, soda-lime glass procured from Schott AG, and polyethylene terephthalate (PET) foil manufactured by DuPont were investigated. The tested ITO coated PET foil (OC50) were purchased from Eastman.

Proton irradiation was performed by the 3 MV Tandatron accelerator (“Horia Hulubei” National Institute of Physics and Nuclear Engineering, Magurele, Romania). The fluency applied to glasses was 10^{15} protons/cm² which corresponds to a dose that a device at low Earth orbit would face within approx. 30 years of operation [9]. The fluency applied to PET foil was lower (10^{14} protons/cm²) because of the previous experience of PET being severely damaged under proton beam exposure. As the ITO layer is usually not directly exposed to the environment (it is typically an interlayer between other materials) we also tested a set of the substrate materials in the configuration where the protons are incident on the substrate material before reaching the ITO layer (denoted as “backside irradiated” in the text). [Supplementary Fig. S1](#) illustrates the simulations carried out for 10,000 incident particles in order to estimate: (i) the penetration depth of protons in the sample; (ii) the scattering pattern of protons in each case. The estimated number of vacancies produced in each material under proton irradiation is given in [supplementary Fig. S2](#). Knowing the proton path length (d) and the proton beam cross section (A), the volume (V) of the material in which the interaction took place can be calculated:

$$V = A \cdot d \quad (1)$$

The amount of sample mass affected by the irradiation is:

$$m = V \cdot \rho \quad (2)$$

where ρ is the material density.

The equivalent dose (H) received by the sample is:

$$H = Q \cdot E / m \quad (3)$$

where Q represents the beam charge in μC and E is the proton beam energy in MeV. The irradiation conditions calculated for each sample using the above equations are given in [Table 1](#).

AFM topography was performed on an ICON system (Bruker) in the PeakForce regime (typical values: peak force 6 nN, gain 8) using a coated tip of nominal resonance frequency 75 kHz (BudgetSensors Multi75E-G). NTEGRA Prima AFM (NT-MDT) was used for KPFM characterization (typical values: dz 0 nm, AC voltage 2 V). For recalculation of the KPFM results to absolute WF values we used a

Table 1
Irradiation conditions for the substrates.

Sample	Density (g/cm ³)	Penetration depth (μm)	Fluence (p/cm ²)	Dose (Gy)
Eagle XG	2.4	82.7	10^{15}	2.41×10^7
Soda-lime	2.33	83.35	10^{15}	2.48×10^7
PET	1.38	116.3	10^{14}	2.99×10^6
ITO	7.12	47.1		

thermally evaporated gold layer of known WF 5.0 eV as measured by XPS [10]. The KPFM potential is not constant within the scanned area ($3.0 \times 0.2 \mu\text{m}^2$). The potential variations are described by root-mean-square (RMS) roughness of the potential, which is within 4 meV for all the presented figures. This value is thus used as error bars.

Raman spectroscopy was carried out with an InVia Raman spectroscope (by Renishaw). Excitation laser wavelength was 442 nm for glass substrates. For PET foils a 785 nm laser was used, as under 442 nm excitation the Raman spectrum was totally obscured by photoluminescence. The spectra were processed by a baseline subtraction and normalization.

The optical absorption spectra and the spectral diffuse reflectance were measured by QE65000 spectrometer (Ocean Optics) in the spectral range 200–1100 nm using an integration time of 15 ms, average 1, boxcar 0. Light from the source (DH1000, Ocean Optics) was led to the sample by an optical fiber (P400-1-SR) and then it enters the spectrometer. The changes due to irradiation were compared to the non-irradiated sample considering the sample thickness. The differential absorbance was computed using the following formula:

$$\Delta k = \frac{1}{d} \left(\frac{A_{\text{irradiated}}}{A_{\text{non-irradiated}}} \right) \quad (4)$$

where d is the sample thickness, and the absorbance for irradiated and non-irradiated samples are determined from

$$A = -\log \frac{S_{\lambda} - D_{\lambda}}{R_{\lambda} - D_{\lambda}} \quad (5)$$

where S_{λ} is the sample intensity, D_{λ} is the dark signal and R_{λ} is the reference signal.

The spectral diffuse reflectance was measured by the same illumination source and spectrometer with the use of an integration sphere ISP-50-8-R-GT, considering the non-irradiated samples as reference.

The results are presented in the form of the ratio $S_{\text{irradiated}}/S_{\text{non-irradiated}}$. THz spectroscopy was performed by a TPS3000 spectrometer (TeraView) in the spectral range 0.06–2 THz using a spectral resolution of 0.75 cm^{-1} .

The surface chemical composition of the samples was investigated by X-ray photoelectron spectroscopy (XPS) using AXIS Supra spectrometer (Kratos Analytical Ltd., UK) equipped with a hemispherical analyzer and a monochromatic Al K α X-ray source (1486.6 eV). The XPS spectra were acquired at a constant 90° take-off angle from the analyzed area about $0.7 \times 0.3 \text{ mm}^2$. The survey spectra were collected with the pass energy of 80 eV, whereas the high-resolution spectrum scans were recorded with the pass energy of 20 eV. The measured spectra were processed in CasaXPS software with Shirley background correction procedure and Gaussian line shapes of variable widths. The obtained XPS spectra were calibrated on 285 eV in accordance with literature [11].

FTIR spectra in transmission mode were measured using a N₂-purged Thermo Nicolet 870 spectrometer equipped with a KBr beam splitter and a MCT detector cooled by liquid nitrogen. The optical absorbance was calculated as $A = -\log(R/R_0)$, where R is the spectrum of the analyzed material and R_0 is the reference (background) spectrum of ambient air. In all cases, the spectra represent an average of 128 scans recorded with a resolution of 4 cm^{-1} .

3. Results and discussion

3.1. Atomic force microscopy

Bare substrates are affected differently by the proton irradiation. Eagle XG glass shows only minimal change in AFM topography which is also confirmed by a negligible decrease in surface RMS roughness by approx. 0.1 nm ([Fig. 1a,d](#)). Originally flat and smooth (RMS roughness 0.35 nm) soda-lime glass ([Fig. 1b](#)) surface gets covered by linear

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