Studies of the Dimensional Stability of Polymeric Films during Coating Processes

O. Miesbauer[1], S. Kiese, V. Prusko, I. Moufti Chalil, E. Heidenreich, C. Steiner, J. Fahlteich, K. Noller

[1] Fraunhofer-Institut für Verfahrenstechnik und Verpackung IVV, Giggenhauser Straße 35, D-85354 Freising. Phone: +49 8161 491-522. Fax: +49 8161 491-555. Email: oliver.miesbauer@ivv.fraunhofer.de

1. Introduction

The substitution of conventional energy sources by solar energy demands the availability of large areas of photovoltaic modules. Promising candidates for this target are cost efficient thin film solar cells whose photoactive materials are amorphous silicon, cadmium telluride, copper indium gallium diselenide (CIGS), polymers or low molecular weight organic substances containing conjugated π-electron systems [1, 2]. These materials can be deposited from gas phase or solution on top of polymeric substrates by roll-to-roll processes [1, 2]. This leads to light weight and flexible solar cells [1] which can be installed on flat roofs and curved building facades or integrated into consumer electronics and textiles.

A serious disadvantage of electronic devices based on organic or polymeric materials is their sensitivity to oxygen and water vapor from the environment. These substances oxidize the photoactive materials and metallic electrodes within the devices which can result in a decreased efficiency or in a complete breakdown [3]. However, also inorganic solar cells, especially CIGS, are degraded by water vapor [4].

Therefore, thin film solar cells have to be encapsulated in order to protect them against oxygen and water vapor and to ensure a lifetime of more than 25 years. This requires water vapor transmission rates of the encapsulation material to be lower than 10^{-4} g·m^{-2}·d^{-1} for organic [5] and CIGS [4] solar cells.

In order to maintain mechanical flexibility and light weight of solar cells, flexible barrier materials instead of conventional encapsulation by rigid glass are preferred [6]. While a tight metal foil is suited for the back sheet of the encapsulation, the front sheet has to be highly optical transparent. Polymer films fulfill the requirements of flexibility and transparency. However, their barrier performance against oxygen and water vapor is not sufficient by far [7]. Therefore, the barrier properties of polymer films have to be significantly improved by deposition of barrier layers [7].

One common concept for high barrier films consists in structures of inorganic (e.g. SiO_x, SiN_x, AlO_x) and (hybrid-) polymeric layers deposited in an alternating sequence [6, 7]. It is assumed that a (hybrid-) polymeric layer is smoothing the surface of the inorganic layer on top of which it is deposited. Therefore it decouples the defects being responsible for permeation of substances through the inorganic layers and, consequently, extends the permeation paths [6, 7, 8]. Other possible explanations for the effective barrier performance of such multilayer structures are the increase of the time lag of permeation [7] by alternating diffusivity and solubility values and the reduction of mechanical stress due to the (hybrid-) polymeric layers [6].

The high barrier films developed by the Fraunhofer POLO® alliance (Fig. 1) are based on a polyethylene terephthalate (PET) or polyethylene naphthalate (PEN) substrate film. They contain two layers of $ZnSn_2O_4$ (ZTO) or Al_2O_3 deposited by reactive dual magnetron sputtering [9]. These are separated by a hybrid polymeric (ORMOCER®) layer which is applied from liquid phase and cured by hot air [9, 10]. To protect the upper sputtered layer against mechanical damage, it is possibly coated by a further hybrid polymeric layer. For the barrier film structure PET Melinex® 400 CW / ZTO / ORMOCER® / ZTO / ORMOCER® a water vapor transmission rate of $(2.0 \pm 1.2) \cdot 10^{-4}$ g·m^{-2}·d^{-1} was measured with calcium test at 38°C, 90% r.h. [9].

A serious drawback of the current high barrier film structure of Fraunhofer POLO® is the too low environmental stability of the polyester substrate film. However, such a property is essential for the en-

capsulation of solar cells with an intended lifetime of at least 25 years [3] under environmental conditions. Therefore, the institutes Fraunhofer FEP, Fraunhofer IVV and Fraunhofer ISC currently work in the joint project *flex25* [11] on the transfer of this high barrier technology to substrate films which are stable against ultraviolet radiation (UV) and weathering.
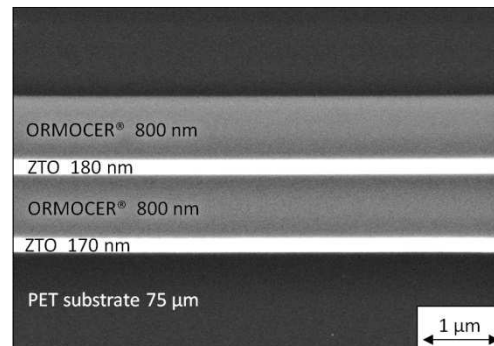

Fig. 1: Typical structure of the high barrier film of the Fraunhofer POLO® alliance

The discussion in the following chapter will show that fluoropolymer films are well suited substrates for such an application. However, in contrast to polyester films, they have insufficient dimensional stability during roll-to-roll processing and elevated temperature processing.

High temperatures of 120°C in combination with web tension especially occur during the production of the Fraunhofer POLO® barrier structure when the ORMOCER® lacquer is cured. Fluoropolymer substrates can be significantly strained during this process step as it was shown for an ethylene tetrafluoroethylene (ETFE) film in an earlier study [12]. As a consequence, already deposited barrier layers might be damaged.

In the present paper, potential candidates for the use as a substrate of an environmentally stable multi-layer barrier film are studied and compared regarding their mechanical behavior during the deposition of barrier layers.

2. Criteria for selecting polymer substrates for environmentally stable high barrier films

In this chapter properties of polymer films regarding their relevance for the encapsulation of inorganic and organic thin film solar cells are discussed.

2.1 Optical transparency

To maintain a high efficiency of the solar cell after encapsulation requires a sufficient optical transparency of the front sheet barrier film especially in that spectral region where the solar cell exhibits its maximum light absorptivity. In comparison to silicon solar cells, the spectral region of maximum light absorptivity is shifted to lower wavelengths in the case of organic photovoltaic devices [13]. On the other hand, ultraviolet radiation has to be absorbed by the encapsulation material since it also degrades photoactive materials and polymers within organic electronic devices [3].

In order to fulfill the optical requirements of solar cell encapsulation, high transparency is needed for the substrate film as well as for of the barrier layers while light reflection at their interfaces has to be reduced by adapting their refractive indices [14]. For PET and PEN the total light transmittance is equal to 90% and 87%, respectively [15]. Excellent light transmittance is exhibited by fluoropolymers [16, 17]: ETFE, for example, reaches a value of 90–98% in the visible region [17].

2.2 Stability against environmental influence

When solar cells are installed outside, the encapsulation is exposed to environmental influences like solar UV radiation, heat and humidity which can degrade the substrate film and the barrier layers [18]. In order to guarantee a long lifetime of the devices, the selected substrate film has to be sufficiently stable against these stress factors.

Polyesters like PET and PEN are known to degrade by hydrolysis when they are stored in humid atmosphere at high temperatures [18, 19]. When UV is absorbed by polyesters, photo-oxidation results in a significant decrease of their optical transparency and mechanical strength [20]. However, it is possible to maintain these properties for at least ten years by integration of stabilizers into the material which absorb the UV radiation [19, 20].

Fluoropolymers in general show good or excellent resistance against humidity as well as against thermal and UV induced degradation [16, 17]. Polyvinyl fluoride (PVF), for example, behaves stable when it is exposed to heat in combination with humidity [18].

2.3 Permeation properties

The barrier performance of a multilayer structure on a PET substrate film is largely caused by the barrier layers whereas the contribution of PET is only small [8]. When the PET substrate is substituted by a material with much lower permeability, this material can possibly have a significant contribution to the barrier performance.

Barrier properties of the substrate film, however, are considered to be of lower importance. Instead of this, the task of the substrate is to provide conditions which allow the deposition of durable barrier layers of high quality. These conditions are related to surface properties and dimensional stability of the substrate.

2.4 Surface properties

Intrinsic surface roughness as well as defects and particles on the surface of a polymeric substrate film are responsible for defects generated in an inorganic layer which is deposited on top of the surface [6, 7, 15, 20]. These defects will increase the permeability of the layer [7]. Therefore, the substrate surface should be preferably smooth and clean. An alternative is the planarization of the surface using a lacquer or a polymeric layer [6, 15, 20].

Furthermore, a sufficient adhesion between the substrate film and a deposited layer is necessary. This possibly requires the modification of the chemical properties of their surfaces e.g. by plasma pretreatment or the deposition of an adhesion promoting layer. Otherwise their delamination and consequently an increased permeability may occur after processing or applying external stress factors [21].

2.5 Dimensional stability

The change of the lateral dimensions of a substrate film leads to mechanical stress in the deposited inorganic layers. This stress may cause crack formation within these layers or delamination of the layers from the substrate [6, 15, 22, 23]. As a consequence, the permeability of the whole barrier film can be significantly increased.

Significant mechanical and thermal load and therewith changes in the lateral dimensions of the web may occur during roll-to-roll processing when it is exposed to high temperatures in combination with mechanical web tension [15, 20]. This is for example the case during the deposition of an ORMOCER[®] layer which is thermally cured at a temperature of 120°C. Furthermore, barrier films are exposed to

high temperature during lamination onto the thin film solar cell [24] or when organic electronic devices are built up directly on top of them [15, 20].

Several mechanisms of the dimensional change of the substrate film during these processes are known. When polymer films are heated, they show thermal expansion [15, 20]. Oriented films, however, begin to shrink after exceeding their glass transition temperature due to relaxation of strained molecular chains [15, 20]. Environmental humidity has an additional influence on the lateral dimensions of many polymer films, e.g. polyesters, when they absorb water vapor or desorb it at higher temperatures [20].

Furthermore, it has to be taken into account that the web tensile stress causes the polymer film to creep in machine direction, especially above the glass transition temperature. When temperature is increased, this behavior becomes more significant [25, 26] while Young's modulus of the film is reduced, in the case of PET from $4 \cdot 10^9$ Nm^{-2} at 23°C to $2 \cdot 10^9$ Nm^{-2} at 120°C [20].

Fluoropolymer films possess a relatively low tensile stiffness [16, 17]. ETFE, for example, exhibits significant creeping behavior at elevated temperature [27, 28] and Young's moduli of $5 \cdot 10^8$ Nm^{-2} at 23°C and $1 \cdot 10^8$ Nm^{-2} at 120°C [29] which are one order of magnitude lower than those of PET.

The term *dimensional stability* refers to the properties of a polymer film to exhibit no or low change in lateral dimensions when exposed to mechanical or thermal load or a combination of both.

3. Experimental details

Due to their excellent environmental stability and optical transparency, fluoropolymer films are considered as well suited substrates for environmental stable high barrier films used for the encapsulation of solar cells. Therefore, the behavior of the following fluoropolymer films during the deposition of the ORMOCER$^®$ layer was studied in detail: ethylene tetrafluoroethylene (ETFE), polyvinylidene fluoride (PVDF) and ethylene chlorotrifluoroethylene (ECTFE).

In order to be able to identify film properties like Young's modulus and shrinkage behavior being relevant for a good processability, these films were compared to well-known polyester films: PET Melinex$^®$ 401 CW, PET Melinex$^®$ ST504 and PEN Teonex$^®$ Q65WA. Among these polyester films, the PET Melinex$^®$ ST504 and PEN Teonex$^®$ Q65WA films are heat stabilized which significantly reduces their shrinkage behavior [20]. Compared to the PET films, the PEN film is advantageous due to its higher glass transition temperature and its higher Young's modulus [20]. Tab. 1 summarizes the films considered in the present study. From each film 0.4 m wide rolls have been processed in the roll-to-roll machines.

Tab. 1: Summary of the films considered in the present study

Polymer	Film type	Producer	Thickness / µm
PET	Melinex$^®$ 401 CW	DuPont Teijin Films	100
PET	Melinex$^®$ ST504	DuPont Teijin Films	125
PEN	Teonex$^®$ Q65 WA	DuPont Teijin Films	125
PVDF	Nowoflon PVDF	Nowofol$^®$	80
ETFE	Nowoflon ET 6235 Z	Nowofol$^®$	100
ECTFE	Nowoflon ECTFE	Nowofol$^®$	100

As a first step, the dimensional change of these films in machine and transverse direction was measured after their winding through the roll-to-roll machine for lacquering and laminating in pilot plant scale at the Fraunhofer IVV [30]. The objective of these trials was to simulate the mechanical and thermal load which is exerted on the films during the deposition of ORMOCER$^®$ lacquers. The temperature of the heat air dryer and the web tension were varied during this study (Tab. 2) in order to understand the influence of these parameters on the dimensional change of the films. While these trials were performed without the deposition of a lacquer, in additional trials the deposition of an

ORMOCER® layer with a thickness of about 1 µm onto the polymer substrate and its thermal curing at 120°C were studied.

Tab. 2: Experimental procedure and parameters

Step	Film treatment	Dryer or process temperature / °C	Web tension / Nm^{-2}
1a	Winding through lacquering machine	30	$2.5 \cdot 10^6$
1b		80	$1.5 \cdot 10^6$
1c		120	$1.5 \cdot 10^6$
1d		120	$2.5 \cdot 10^6$
1e	Deposition of ORMOCER®	120	$2.5 \cdot 10^6$
2	Deposition of ZTO	20 (cooled from backside)	$2 \cdot 10^6 - 1 \cdot 10^7$
3	Winding through lacquering machine	120	$2.5 \cdot 10^6$

After their treatment according to chosen variants from Tab. 2, three films PET Melinex® 401 CW, PEN Teonex® and ECTFE have been studied further. The second step was the reactive sputter deposition of a ZTO layer with a thickness of about 150 nm on top of the films. This process was performed at the roll-to-roll coating machine *novoFlex® 600* of Fraunhofer FEP (shown in [23]) with a web tension of $2 \cdot 10^6$ Nm^{-2} up to $1 \cdot 10^7$ Nm^{-2}.

As a third step, the films were treated in a further run through the roll-to-roll lacquering machine. In this case, a temperature of 120°C and a web tension of $2.5 \cdot 10^6$ Nm^{-2} were chosen since these values are known to result in high barrier performance of a cured ORMOCER® layer and in good winding quality of the film roll. This third step allows to determine whether the ZTO layer is damaged due to dimensional change of the film in the lacquering machine. Steps 1 and 3 of the experiments together correspond to the application of two ORMOCER® layers in the same barrier structure, one as a planarization layer on top of the substrate film (step 1) and the other as an intermediate layer between two ZTO layers (step 3).

In order to measure the dimensional change of the films during the steps, three rectangles with side lengths of about 0.3 m × 0.2 m and with their shorter sides parallel to machine direction were drawn on top of them for each variant. The distances between the parallel lines of the rectangles in machine and transverse direction were measured at several positions using a length measurement system *Electronic scale* with a resolution of 0.01 mm. From the distances s_i before and s_f after each step, the corresponding film strain $\varepsilon = \frac{s_f - s_i}{s_i}$ was calculated.

In addition, before and after treatment, the oxygen permeabilities of the films containing an ZTO layer were measured at 23°C, 50% r.h. using Mocon® OX-TRAN® 2/20 and 2/21 devices with lower measurement limits of $5 \cdot 10^{-2}$ $cm^3 \cdot m^{-2} \cdot d^{-1} \cdot bar^{-1}$ and $5 \cdot 10^{-3}$ $cm^3 \cdot m^{-2} \cdot d^{-1} \cdot bar^{-1}$, respectively. These measurements give an indication of the maximal strain of a film occurring during a process step when this strain leads to a damage of the layer and therefore to an increase of the permeability.

4. Results and discussion

The average values of the dimensional change of the films have been calculated from nine single values for machine direction (MD) and six single values for transverse direction (TD), respectively. In some cases, one outlier was excluded from the average value. The shown oxygen permeabilities are either single values or average values calculated from two or three single values. It is assumed, that presented single values at least show the correct order of magnitude for the permeability.

Fig. 2 shows the dimensional change of the films after their first winding through the lacquering machine (Step 1). At a dryer temperature of 30°C, only minimal changes were observed. However, elongation in machine direction and shrinkage in transverse direction are significantly increased for higher processing temperature. This result is consistent with the mentioned increased creeping behavior of polymer films above their glass transition temperature. The strain observed after cooling the film to room temperature is the residual strain of the creeping process [26]. The lateral contraction observed for the films is due to tensile strain in machine direction, possibly in combination with thermal shrinkage.
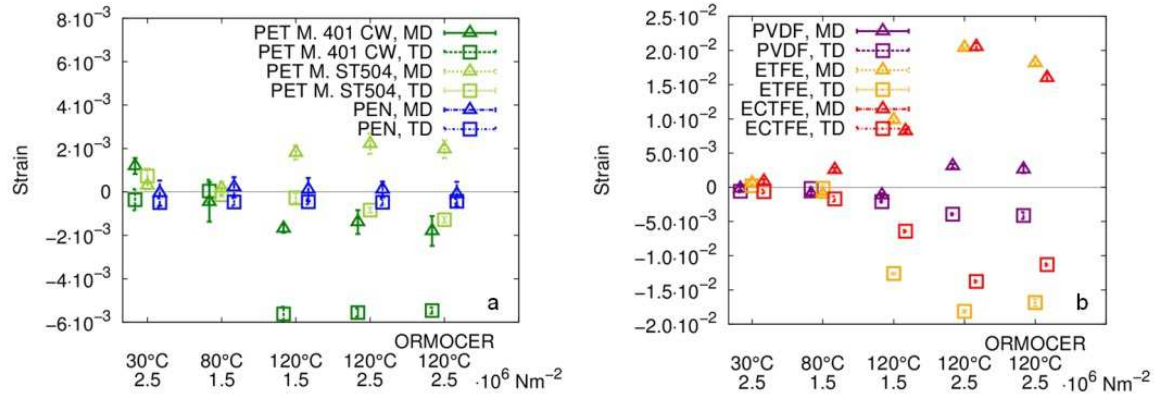

Fig. 2: Strain of polymer (a) and fluoropolymer (b) substrate films during their first winding through the roll-to-roll lacquering machine under the conditions shown in Tab. 2. The axis of abscissas shows the different processing conditions, i.e. temperature and web tensile stress.

Only the PEN Teonex[®] film shows a dimensional change of less than 0.1% after processing at temperatures up to 120°C. This is due to heat stabilization and is consistent with its Young's modulus of $3.5 \cdot 10^9$ Nm^{-2} being large also at 120°C [20]. The reduction of the thermal shrinkage in transverse direction due to heat stabilization can clearly be observed when PET Melinex[®] ST504 is compared to PET Melinex[®] 401 CW. In addition, the PET films would be expected to exhibit a higher strain than PEN Teonex[®] in machine direction since they possess lower Young's moduli of $2 \cdot 10^9$ Nm^{-2} at 120°C [20]. However, a significant strain is observed only for PET Melinex[®] ST504. In contrast, PET Melinex[®] 401 CW shrinks in machine direction at high temperature despite the exerted tensile stress. This fact confirms the result obtained in an earlier study [12] and is explained by the thermal shrinkage stress [31] exceeding the exerted tensile stress.

In comparison to the polyester films, a significantly larger increase of elongation in machine direction and of contraction in transverse direction is observed for ETFE and ECTFE when processing temperature was raised from 30°C to 120°C. Raising in addition the tensile stress from $1.5 \cdot 10^6$ to $2.5 \cdot 10^6$ Nm^{-2}, results in a further increase of the contraction and strain to values above 1% or even 2%, respectively. These results are clearly consistent with the Young's moduli of $1 \cdot 10^8$ Nm^{-2} at 120°C for ETFE and ECTFE [29] being significantly lower in comparison to those of polyester films.

In contrast, a dimensional change of below 0.5% under these conditions as obtained for PVDF. The shrinkage at 120°C and $1.5 \cdot 10^6$ Nm^{-2} allows to assume that also for PVDF a partial compensation of the tensile by the thermal shrinkage stress is present as it was observed for PET Melinex[®] 401 CW.

In contrast to these results obtained by winding the film through the lacquering machine, a slight reduction of the dimensional change of ETFE and ECTFE films during ORMOCER[®] deposition was observed. This is explained by the cooling effect which arises when the solvent of the lacquer is evaporated.

The dimensional change of films during ZTO deposition was found to be below 0.2% in each considered case. This is due to back-side cooling of the substrate film during the sputtering process [32]. A maximum surface temperature of 70°C was measured using temperature measurement stripes in earlier experiments.

If ECTFE was pre-stressed in a first winding through the lacquering machine at 30°C and $2.5 \cdot 10^6$ Nm^{-2}, a lower dimensional change of ECTFE / ZTO after the subsequent run at 120°C and $2.5 \cdot 10^6$ Nm^{-2} (Fig. 3) was measured, compared with the value obtained for ECTFE after its winding at 120°C and $2.5 \cdot 10^6$ Nm^{-2}. The reason for this is either the effect of the pre-stress of ECTFE in the first case or the strengthening of the film due to the ZTO layer.
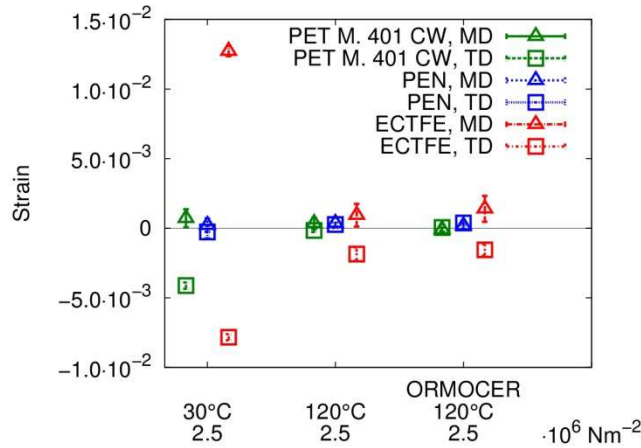

Fig. 3: Strain of PET Melinex® 401 CW / ZTO, Teonex® Q65 WA / ZTO and Nowoflon ECTFE / ZTO during their winding through the roll-to-roll lacquering machine (step 3), after the polymer substrates have been loaded or coated with ORMOCER® in step 1 under the conditions given as abscissas

Increasing the temperature of PET Melinex® 401 CW and ECTFE from 30°C to 120°C during step 1 results in a considerably reduced dimensional change of PET Melinex® 401 CW / ZTO and ECTFE / ZTO during the subsequent run (step 3) at 120°C. The same result is obtained when an ORMOCER® layer is deposited in step 1. Based on these results one can assume that the first thermal treatment in the roll-to-roll lacquering machine causes a limited heat-stabilization effect on the polymer film.

For the structure ECTFE / ZTO the oxygen permeability is about 10 times as high as the correspondent values for polyester / ZTO (Fig. 4). This is clearly a consequence of the higher oxygen permeation coefficient of the ECTFE film [7] and the higher surface roughness observed by atomic force microscopy for the ECTFE in comparison to the polyester substrates. However, by application of an ORMOCER® layer on top of ECTFE, the oxygen permeability is reduced by a factor of more than 40 due to the smoother surface and the lower permeation coefficient of ORMOCER®. This shows that an inorganic layer with low porosity and consequently low gas permeability can be deposited on top of a rough substrate when the substrate is smoothened before.

When the structure ECTFE / ZTO is treated in the lacquering machine at 120°C and $2.5 \cdot 10^6$ Nm^{-2} during step 3, its oxygen permeability is increased by a factor of more than 10. This result is assumed to be due to the formation of cracks within the ZTO layer. Therefore, it can be concluded that the maximal strain of the film in machine direction caused by thermal and mechanical load during this process is larger than the crack onset strain of the 150 nm thick ZTO layer. The crack onset strain is defined as that strain on which first crack formation is observed in an optical microscope. For ZTO single layers the crack onset strain corresponds also to the first observed increase of the water vapor permeation rate. A ZTO layer with a thickness of 150 nm on a PET substrate shows a crack onset strain of (0.8 ± 0.1) % [23].

The ratio between the oxygen permeabilities of Polymer (120°C, σ) / ZTO (120°C, σ) (see Fig. 4) and Polymer (30°C, σ) / ZTO rises in the same order as Young's modulus (at 120°C) of the polymers decreases: $3.5 \cdot 10^9$ Nm^{-2} for PEN [20]; $2 \cdot 10^9$ Nm^{-2} for PET [20]; $1 \cdot 10^8$ Nm^{-2} for ECTFE [29]. This confirms that the increase of the permeability in step 3 is caused by the formation of defects within ZTO which gets more significant with increasing strain, i.e. with decreasing Young's modulus.
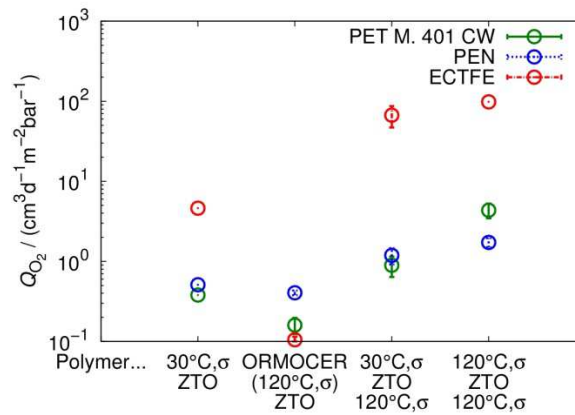
Fig. 4: Oxygen permeability at 23°C, 50% r.h., measured for three Polymer / ZTO and Polymer / ORMOCER[®] / ZTO films, possibly after winding the film or the polymer substrate through the lacquering machine at the given temperature and a tensile stress of $\sigma = 2.5 \cdot 10^6$ Nm^{-2}

When the substrate films PET Melinex[®] 401 and PEN Teonex[®] have not been pre-stressed by step 1, however, the oxygen permeabilities, measured after their winding with deposited ZTO layer through the machine in step 3, are similar. This shows again that the thermal shrinkage stress can compensate the tensile stress at least partially. During pre-stressing the thermal shrinkage behavior of PET Melinex[®] 401 is eliminated so that a compensation in a further winding is not possible any more.

The order of magnitude of the oxygen permeability measured for treated ECTFE / ZTO is independent of the fact whether ECTFE was pre-stressed or not. However, in the pre-stressed case, only a very small irreversible strain of ECTFE / ZTO is obtained after the winding through the machine, in contrast to the case without pre-stress. These different trends observed for the permeability and for the irreversible strain show that the actual maximum strain which arises during the process and which determines a possible damage of the ZTO layer might be significantly larger than the measured irreversible strain.

5. Conclusions

Due to their excellent optical transparency and environmental stability, fluoropolymers are highly preferred as substrates for the production of high barrier films which are required for the flexible encapsulation of thin film, and especially organic, photovoltaic cells. However, a serious drawback of fluoropolymer films is their weak dimensional stability under mechanical and thermal load.

In the present study, fluoropolymer and polyester films were compared regarding their behavior during the deposition of barrier lacquers. It was shown that a similar permeation of a sputtered ZTO layer on top of ECTFE as on a polyester film can be reached when the rough surface of ECTFE is smoothened by deposition of an ORMOCER[®] layer. Due to this possibility, a smooth surface is not an essential property of a substrate film to be suited for the intended application.

The oxygen permeability of an ECTFE film carrying a ZTO layer is increased by one order of magnitude when it is wound through the roll-to-roll lacquering machine under elevated temperature conditions. This result is explained by the formation of cracks within ZTO which are caused by the significant dimensional change of the film due to mechanical and thermal stress. For the production of high barrier films on top of a fluoropolymer substrate, however, it is essential to avoid such a behavior.

Therefore it is necessary to evaluate the behavior of additional fluoropolymer films during the deposition of barrier layers. In this context it will also be tested whether better results are obtained when the tensile stress or additionally the temperature of the heat air dryer are reduced.

During the present study a partial compensation of the tensile stress due to the thermal shrinkage stress was observed. A deeper insight into the interaction between mechanical creep at high temperatures and thermal shrinkage is necessary in order to use this fact systematically to obtain improved

film properties. These effects will be further studied by performing tensile tests at different temperatures and by the determination of the maximal strain arising during coating processes using different sputtered layers with well-known crack onset strains as an indicator.

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