

Correlation study of damp heat and pressure cooker testing on backsheets

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ABSTRACT: For photovoltaic (PV) modules, high-quality backsheets are essential. To verify these qualities, hot and humid tests of up to 1000 h are standard in the PV industry. Damp heat (DH) tests are useful for evaluating material quality and infant failures, especially for materials that are sensitive to hydrolysis. However, shorter test periods are needed. Hence, the objective of this study was to investigate and correlate the aging behavior of hydrolysis-sensitive polyester [poly(ethylene terephthalate) (PET)] based backsheets after exposure to DH and pressure cooker conditions. The material properties, such as thermal and mechanical parameters, correlated well between the pressure cooker at 120 °C and the DH test, with the qualification that differences in the failure mechanism were seen after 96 and 2000 h, respectively. Nevertheless, it should be acceptable to use pressure cooker tests (PCTs) for fast material testing. The PCT is proposed to significantly reduce the testing time for infant failures and material quality to around 4 days for PET-based backsheets. © 2016 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2016**, *133*, 44230.

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INTRODUCTION

Considerable research and progress has been made in the development of photovoltaic (PV) modules and their incorporated components to increase their reliability and service life. This benefits the market growth of renewable energy generated directly by the sun.^{1,2} To collect radiant flux cheaply by PV modules, relatively high module lifetimes are necessary. For PV modules, high product standards are required, and as a result, the module lifetimes of 25 years and more are guaranteed with a limited loss in functionality of the component. The weathering stability is the key factor in the reliability of PV modules. The main critical parts concerning degradation are polymeric materials used as protective frontsheets, sealants, encapsulation, and backsheets in a module.^{1,3–7}

There are different approaches to test the lifetime of a PV module. One option is testing the performance of the whole PV module. This is not only the most expensive option, but it also requires the most effort. Testing on the material and component levels, however, is less costly and has simplified sample preparation and provided more testing options. In the case of encapsulations, the advantages of testing film samples are merely applicability, preselection, or process improvements. The reason is that some interactions, such as reaction rates, interdependency, and aging mechanisms, of the components can hardly be

reproduced. However, for backsheets consisting of polyester, Knausz *et al.*⁸ already showed that differences in the material properties, whether the backsheet was aged as a single film or incorporated within a PV module, were negligible when the microclimate was comparable. Because of the different installation situations of the backsheet compared to the encapsulation, fewer degradation cases occurred in combination with other PV module components. This makes the single-film testing of poly(ethylene terephthalate) (PET)-based backsheets suitable. This is an advantage especially for fast material testing.

Backsheets in general have to provide mechanical stability and electrical insulation and should act as protection against weathering and as a selective barrier for atmospheric gases (water vapor, oxygen, acetic acid, etc.).⁵ Thus, the type and, therefore, the different degradation behaviors of a backsheet component highly affect the long-term stability of the whole PV module.^{1,3–5,8–11} Generally, multilayer laminates are used as backsheets. In many standard backsheets, PET is used as the core layer to offer mechanical stability and sufficient electrical insulation.^{1,8} The core layer is used in combination with protective outer layers (e.g., fluoropolymers, polyamides, highly stabilized PET, or other polymeric materials). The loss of functionality of the core layer can result in the delamination of the backsheet, decreased or insufficient electrical insulation properties, or

critical changes in the permeation properties and can subsequently lead to the breakdown of the whole module. Consequently, the aging behavior of the core layer is crucial for the lifetime of a backsheet and, therefore, for the whole PV module during its intended life of more than 25 years.^{8,12,13}

For the investigation of the aging behavior and its influence on the material properties in a reasonable period, accelerated aging tests have been conducted. In the case of polycondensates, such as PET or polyamide, as core layers in backsheets, hydrolysis is the dominating aging factor.^{5,12–15} The activation energy for thermal degradation is, on average, 197 kJ/mol.¹⁶ By comparison, the value is lower for hydrolysis, around 136 kJ/mol; this depends on the temperature and humidity level.¹⁷ Looney and Brennan¹² showed that the activation energy was even lower, around 111 kJ kg⁻¹ mol⁻¹, for PET hydrolysis. This indicated that hydrolysis was the dominant factor in all degradation cases, but there were contributions from other reactions, such as oxidative degradation and thermal esterification.¹²

Polymers with functional groups such as amides and esters (e.g., in polyesters) can degrade via hydrolysis under the presence of water and acids.¹⁸ Ester bonds are susceptible to hydrolysis, especially at elevated temperatures and moisture. The reaction leads to chain scission at the ester bond, which decreases the molecular weight.¹⁶ Additionally, the concentration of carboxyl end groups increases. However, the lower the value of carboxyl end groups in the origin material is, the higher the hydrolysis resistance will be as they support hydrolytic degradation.^{12,16} Antihydrolysis additives for polyesters can be based on carbodiimide (HN—C—NH) to chemically react with free moisture during processing and service and act as acid and water scavengers; this converts them into nonreactive urea structures.¹⁶ Phenylenebisoxazone is also an effective scavenger. It reacts with acid end groups and couples PET chains, thus increasing the polymer molecular weight.¹⁶

In general, hydrolysis leads to chain scission, which results in embrittlement, but also physical aging processes (e.g., postcrystallization), which can lead to a loss in mechanical properties.^{5,8,19} This can be simulated by hot and humid tests, such as damp heat (DH) storage tests (at 85 °C and 85% relative humidity). In the PV industry, DH tests are often applied for up to 1000 h to evaluate the quality of backsheets of PV modules. DH testing is included in IEEE 1262 as a qualification test for up to 1000 h and also in the IEC 61215 and 61730 standards.^{20,21} However, as Looney and Brennan¹² already stated, DH testing is not adequate for safely predicting the field lifetimes of PV modules, especially because of the fact that UV degradation is neglected. However, because of the shielding outer layers, such as fluoropolymers, the core layer is protected to a certain degree. Furthermore UV radiation on the backsheet is of minor importance compared to the stress of the frontsheet, and it depends on the location and type of mounting and the ground.^{22,23} Additionally, differences in the microclimate can cause different aging behaviors.

Even though the DH test exhibits drawbacks, as stated previously, it is a useful test for evaluating the material quality and infant material failures, especially for materials that are sensitive

Table I. Aging Parameters

Parameter	DH test	PCT			
		110	120	140	
Temperature (°C)	85	100	110	120	140
Relative humidity (%)	85	100	100	100	100
Pressure (bar)	Ambient	4	4	4	4
Duration (h)	500	12	12	12	12
	1000	24	24	24	24
	2000	48	48	48	
					96

to hydrolysis.^{7,12} This was also shown by Gambogi *et al.*,²⁴ Looney and Brennan,¹² and Gong *et al.*⁷

Usually DH testing of 1000 h or more is conducted in the PV industry. This equals approximately 3 months; this is too long for a fast material preselection. A decrease in the developmental time of backsheets can be achieved by increases in the temperature, humidity, and pressure. A saturated vapor pressurization test or pressure cooker test (PCT), which has been standardized (DIN EN 60749-33) and is commonly used in the semiconductor and printed circuit boards (PCB) industry, offers these specific environmental conditions.⁷ PCT testing is already used for backsheets to evaluate their weather resistance, in particular, their resistance to hydrolysis.^{12,25,26} However, it has not been clarified whether the aging mechanisms and effects on the material properties are equal to DH test exposure. This is particularly because the PCT exposure is high above the glass-transition temperature of PET (ca. 80 °C); this is in contrast to DH exposure.

Hence, the objective of this study was to investigate and correlate the aging behavior of PET-based PV backsheets after exposure under DH and PCT conditions. Moreover, a correlation between the results of the DH test and the further accelerated PCT was conducted.

EXPERIMENTAL

A standard composite backsheet film, consisting of poly(vinyl fluoride) (PVF) and PET was investigated. The PVF layer was approximately 30 μm thick, and the PET layer was approximately 200 μm thick. Samples were artificially aged under DH test conditions for up to 2000 h and under PCT conditions (at 100–140 °C, 100% relative humidity, and 4 bar) for up to 96 h (see Table I).

To analyze and verify differences in the thermal, mechanical, and chemical properties, the aging behavior was investigated by differential scanning calorimetry (DSC), tensile testing, and IR spectroscopy. As already demonstrated in the literature,^{14,18} the PVF exhibited no significant changes after artificial weathering. Additionally, the thickness of the PVF layer was about 1/7 that of the PET layer. These were the reasons why only the aging results of PET are discussed. Because of the targeted test duration of around 5 days (120 h), the relation of the two time abscissas was fixed to better compare the two test types (96 h of PCT testing = 2000 h of DH testing).

DSC

Thermal analysis was used to determine the aging effects on the morphology of the polymeric backsheets. The tests were performed with a PerkinElmer DSC 4000 instrument. Indium and zinc standards were used to calibrate the temperature and enthalpy. Samples of around 10 mg were cut and put into 50- μ L pans with perforated lids. Thermograms were recorded under a nitrogen atmosphere from 25 to 300 °C. A heating rate of 10 K/min was chosen to properly investigate the melting and crystallization processes.^{27,28} To identify reversible (physical aging effects) and irreversible (chemical aging effects) processes, two heating runs and one cooling run were conducted. The peak temperatures and enthalpies were evaluated for the purpose of determining the aging effects on the polymer morphology according to ISO 11357-3.²⁹ For every evaluation, at least two sample runs were evaluated.

Tensile Testing

Tensile tests on rectangular samples were carried out with a Z10 testing machine (Zwick-Roell, Ulm, D) according to EN ISO 527-3.³⁰ All samples had a width of 15 mm, a gauge length of 50 mm, and an overall sample length of 100 mm. The samples were cut after artificial aging and tested in the machine direction. The tests were conducted at a crosshead speed of 50 mm/min. Average values for elastic modulus and yield strength were deduced via force values and the crosshead speed from a total of at least seven specimens for each test series.

Fourier Transform IR Spectroscopy

To characterize the chemical structure and its changes after artificial aging, IR measurements were conducted. IR analysis was carried out in attenuated total reflection mode with a Spectrum GX Fourier transform IR spectrometer (PerkinElmer, Waltham, MA). The attenuated total reflection unit contained a zinc selenide crystal (Pike Technologies, Madison, WI). The transmittance spectra were recorded between 4000 and 650 cm^{-1} . Each spectrum was an average of 12 spectra measured at a resolution of 4 cm^{-1} . For better comparison, all attenuated total reflection spectra were normalized to 1408 cm^{-1} according to Urban,^{31,32} and this corresponded to the ring in-plane deformation.

RESULTS AND DISCUSSION

In the following section, we discuss the results of the aging tests. DSC is a versatile tool to determine physical aging processes such as postcrystallization. The melting enthalpy is directly proportional to the degree of crystallinity, which is an indicator of aging processes. However, chemical degradation, like chain scission, can be also detected by DSC measurements.^{18,27,28} An increase in the melting enthalpy and a shift to higher melting temperatures can be a result of postcrystallization. In contrast, chain scission due to hydrolysis results in a shift in the crystallization temperature and in an increase in the crystallization enthalpy.^{8,27}

Figure 1 exemplarily shows the thermograms (first heating, cooling, and second heating) of an unaged and DH 2000 h aged sample. With increasing temperature, the melting endotherms of the first heating decreased slightly. At about 192 °C, the curves resulted in a small endothermic peak, which was caused

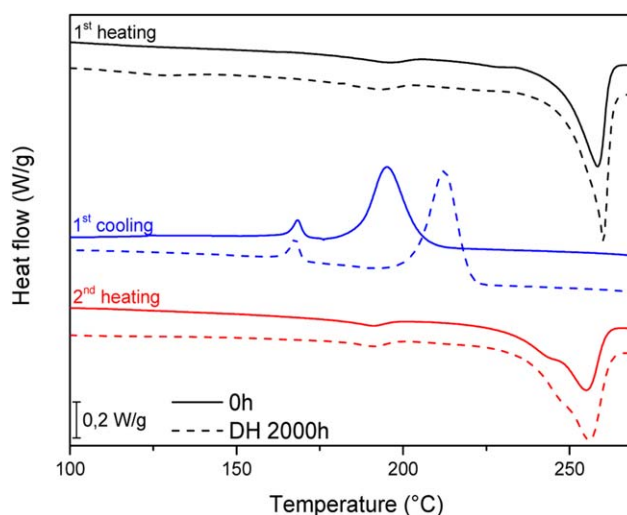


Figure 1. Thermograms of the unaged and aged samples. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

by the melting of the 30 μm thick PVF layer. The 200 μm thick PET layer melted around 258 °C. These values were in accordance with literature values.²⁷ No glass transitions or cold crystallization temperatures for PET were observed; this indicated a highly crystalline material.²⁷ In the crystallization curves, the first crystallization peak was attributed to PET (194 °C), and the second was attributed to PVF (168 °C). In the second heating run, PVF showed a melting peak at 191 °C, and PET showed a melting peak at 255 °C. As mentioned before, PVF exhibited no significant aging effects within the designated time. Distinctive changes in the initial melting and cooling behaviors of PET were observed after aging; significant increases in the melting enthalpy and crystallization temperature were observed during the first heating and cooling.

As PET is a polymer made via a condensation process under the elimination of water, it is sensitive to chain scission due to hydrolysis.¹⁸ In general, polymers with functional groups such as amides and esters (PET) can degrade via hydrolysis in the presence of water and acids.¹⁸ However, as it is used as a PV backsheets material, PET should be resistant to hydrolysis. This can be accomplished by an increase in the degree of crystallinity by heat treatment and by addition of suitable stabilizers (nucleating agents). A higher degree of crystallinity is also desirable when PET is subjected to elevated temperatures.¹⁶

In Figure 2, the PET melting enthalpies for all of the unaged and aged samples are depicted. In general, with rising test duration, the melting enthalpy increased. For the DH test samples, the value increased from 38.9 to 44.8 J/g after 2000 h. The increase in the melting enthalpy could have been caused by physical and chemical aging processes. One obvious physical effect was postcrystallization.^{18,33} Postcrystallization takes place in particular in imperfectly crystallized materials. In general, semicrystalline polymers, such as PET, exhibit no crystalline equilibrium because of the process conditions of the industrial production. During artificial aging, changes in the physical structure may happen; these lead to increases in the degree of

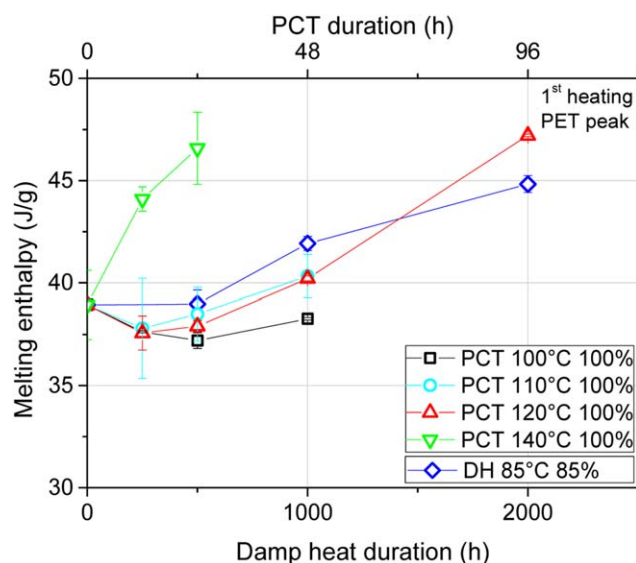


Figure 2. PET melting enthalpies. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

crystallinity and the lamellar thickness. Furthermore, a refinement of the crystalline structure occurs, where amorphous molecules near the crystallite surface are attached to the crystallite regions. This process is accelerated at temperatures above the glass-transition temperature (around 80 °C for PET) because of a higher mobility of the molecular chains. As a result of the increased degree of crystallinity, a higher melting enthalpy can be observed.^{18,33}

PCT at temperature levels of 100, 110, and 120 °C showed no significant changes in the melting enthalpy for up to 24 h. With longer exposure times (>24 h) the melting enthalpies of the samples aged at 110 and 120 °C increased to values almost similar to those from the DH test. After 96 h of PCT exposure at 120 °C, the enthalpy showed a slightly higher value (47.2 J/g) than the DH 2000-h sample (44.8 J/g). In general, the enthalpies were considered precise temperature values because of the fact that the whole weight of the multilayer film was used for the calculation of the enthalpy, and this could have led to a larger uncertainty. Also, differences in the layer thicknesses highly influence the enthalpy results, which could not be excluded.

The samples aged under PCT conditions at 140 °C exhibited the strongest melting enthalpy increase of 7.7 J/g to a value of 46.6 J/g after 24 h. Presumably, an aging temperature of 140 °C was too harsh for the material. Additionally, after 24 h, a strong embrittlement was already observed. The degradation rate depended on the humidity level, the temperature, and of course, the morphology of the polymer and the degree of crystallinity. The polymeric chains in the amorphous phase were shortened and, therefore, exhibited a higher mobility. Short chains located next to the lamellar surfaces could attach themselves to the crystallite region. This so-called chemocrystallization process occurred preferably under high molecular mobility, that is, above the glass-transition temperature; this was the case for the conducted aging tests.^{18,34} As a result, the density changed, like

Planes *et al.*³⁵ showed, and the melting enthalpy increased. This led to the assumption that with a 140 °C exposure, the chemical aging processes were more dominant than with exposures at lower temperatures (100–120 °C).

In contrast to the melting enthalpy, the melting peak temperature was almost unaffected by different aging conditions and durations. However, the crystallization temperature of all of aged samples increased after aging (see Figure 3). In addition, the higher the temperature level was, the higher the crystallization temperature was. A temperature of 100 °C for PCT was probably too low to cause the same change in the crystallization temperature as DH: the crystallization temperature increased to a value of 199 °C after 48 h of exposure. PCT at 110 °C showed good similarity up to 24 h compared to DH. After 48 h, the value stagnated around 201 °C, which was 10 °C below the crystallization temperature of the DH test after 1000 h.

The PCT samples aged at 120 °C showed almost similar behavior to that in the DH test, with deviations of a maximum of 4 °C of the mean value after 48 h of aging. Samples aged at 140 °C exhibited the strongest increase in the crystallization temperature after artificial aging, from a value of 194 to 215 °C after 24 h. As already described in the literature by Frick and Stern,²⁸ the crystallization temperature is affected by the molar weight and, therefore, by chemical changes in the polymer. The reason is that shorter chains, previously formed by chain scission, exhibit a higher flowability. This led to a higher mobility of the shorter chains and, with it, a better form energy of crystallites. This effect corroborated the previously stated assumption that the samples aged at 140 °C showed the dominating effects of chemical degradation (hydrolysis). Similar behavior was observed for the crystallization enthalpy: higher exposure temperatures and longer exposure times led to higher increases in the crystallization enthalpy. Nonetheless, an Arrhenius plot of the crystallization temperature revealed a linear behavior, that is, a constant activation energy, because hydrolysis was the main

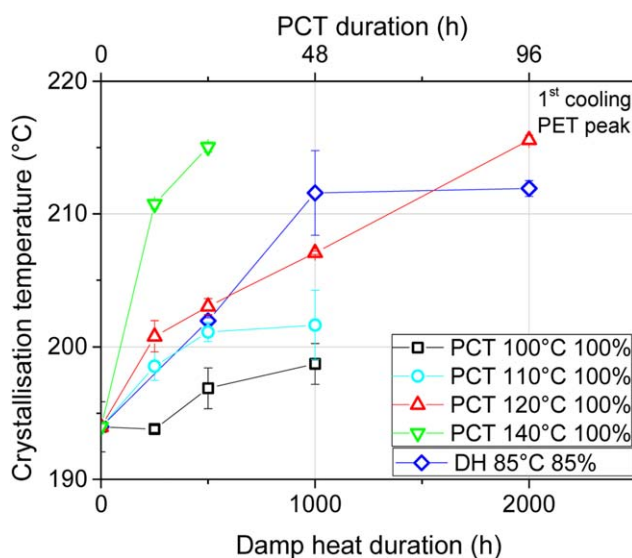


Figure 3. PET crystallization temperatures. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

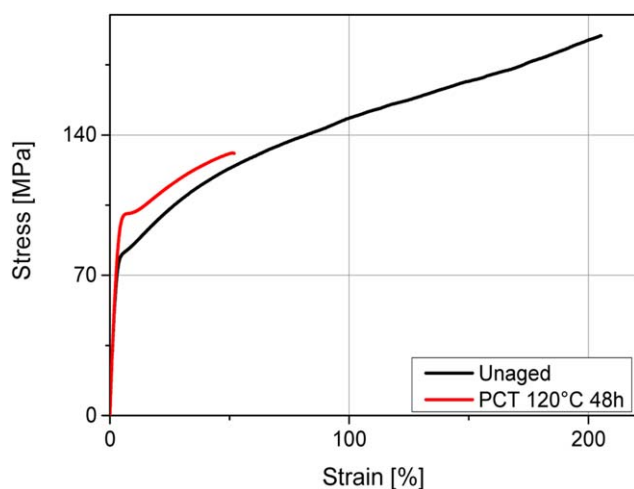


Figure 4. Stress–strain curves of the unaged sample and sample aged by PCT at 120 °C for 48 h. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

aging process; this was detected indirectly by the crystallization temperature.

For the purpose of analyzing the influence of aging on the mechanical behavior, tensile tests were conducted. This was done to obtain information about the physical and chemical aging processes. Physical aging processes, such as postcrystallization, are mainly visible before and in the yield region, whereas for chemical aging effects, such as chain scission due to hydrolysis, the region of the breaking point can be used as an indicator.^{19,33} The unaged film samples exhibited ductile material behavior with a pronounced yield point (see Figure 4). When the thickness ratio between the PET and PVF layer was 200–30 μm , the mechanical behavior was mainly defined by the thicker PET layer. Aging at elevated temperatures and humidity caused an embrittlement; this led to a significant increase in the yield stress and significant decreases in the stress and strain at break, respectively. An increase in the yield stress indicated physical aging, such as postcrystallization.^{13,14,19} The effect of a decrease in the strain at break is well reported in the literature and could be related to chemical aging with chain scission and, therefore, to degradation via hydrolysis; this is supported by increases in the crystallization temperature and enthalpy over the aging time.^{8,14,16}

In Figure 5, the yield stress over aging duration is plotted. As Oreski and Pinter⁵ already showed, the yield stress is a good indicator for physical aging processes because it is highly influenced by the degree of crystallinity. With increasing DH aging duration, the yield stress value increased. This was in good accordance with the DSC results, where an increase in the melting enthalpy due to postcrystallization was detected. After 2000 h of DH testing, the samples became brittle, and failure occurred before yield stress was reached. The PCT samples at 100, 110, and 120 °C showed similar behavior: with increasing test time, the yield stress increased from around 80 MPa to around 95 MPa after 48 h. Samples aged under 120 °C for 96 h became brittle and showed very low yield stress values around 6.9 MPa. The PCT samples after 12 h at 140 °C became too

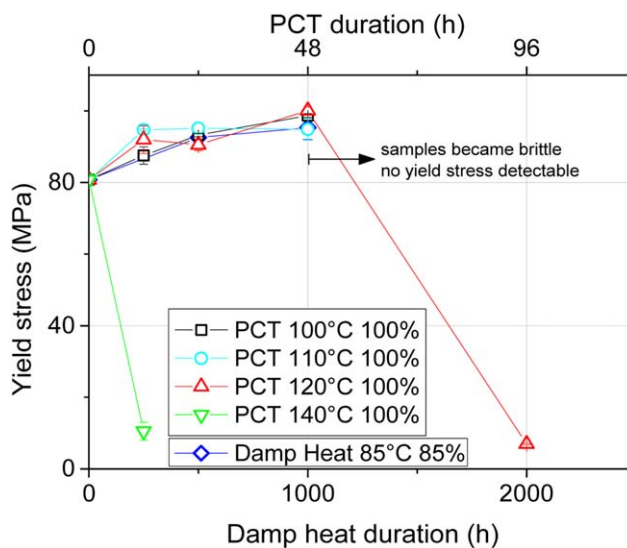


Figure 5. Yield stresses of all of the samples. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

brittle, and no yield stress was detected. After 48 h at 140° PCT, the samples became too brittle for mechanical testing. The yield stress results indicated dominating physical aging processes at lower temperature levels up to 48 and 1000 h. After longer exposure times (>48 h), the chemical aging processes seemed to be more dominating as the samples became very brittle. In general, the yield stress value is a good indicator for aging processes such as postcrystallization.¹⁴ However, it was not a suitable parameter to detect differences between PCT at 100, 110, and 120 °C.

A suitable and highly sensitive indicator for changes in molar mass is the strain at break value (see Figure 6). To use it as such, it is necessary to do the testing in the machine direction of the film, which is in contrast to the work of Kim *et al.*²⁶

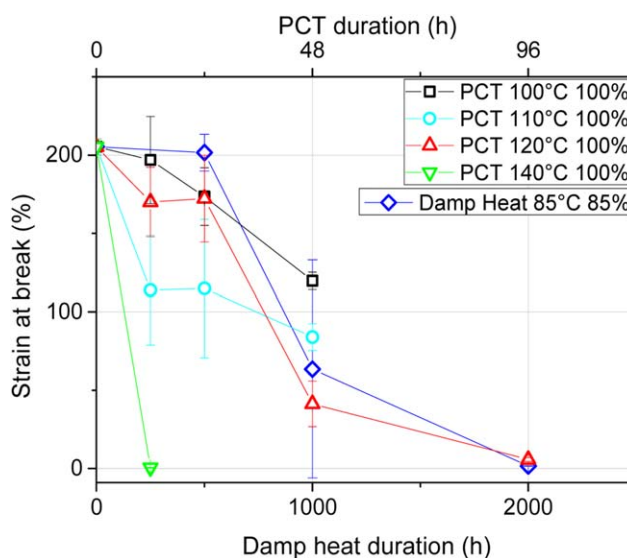


Figure 6. Strain at break values of the unaged and aged samples. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

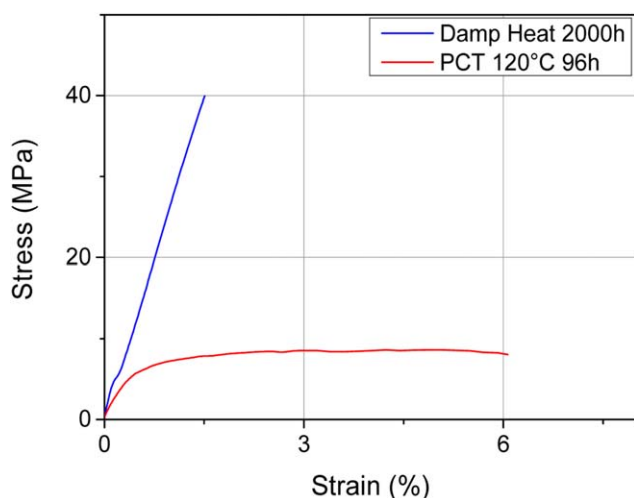


Figure 7. Stress–strain curves of the sample aged by DH for 2000 h and a sample aged by PCT at 120 °C for 96 h. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Polymers, especially films, show anisotropic behavior; this is generally due to their production process (drawing off after the extrusion process). When testing polymer films in the machine direction, a full orientation of the polymer chains in the load direction occurs after the yield stress is passed. The load gets absorbed via the main valence bonds of the molecular chains and no longer by intermolecular forces. Thus, the polymer chain length and the resulting entanglements determine the elongation at break.^{13,14,36,37} Consequently, when the molar mass is higher, longer chains and more entanglements can be formed. Therefore, chain scission via hydrolysis results in a decrease in the elongation at break. If testing is done in the transverse direction, the load is mainly absorbed by intermolecular forces and not by the main valence bonds of the molecular chain.^{13,14,36,37}

A relatively high standard deviation of the strain at break value for all samples was noticeable. The failure of a tensile sample is usually caused by a defect that induces a higher stress concentration (notching effect). A reason for the higher standard deviation could have been the fact that the sample preparation was done after artificial aging; this mechanically generated defects on the edges of the samples. However, different thickness distributions, enclosures, bubbles, and so on could also be initiators for defects.^{5,8}

The DH test for 500 h caused no significant changes in the strain at break value (202%); this indicated that mainly physical aging processes, such as postcrystallization, occurred. However, an exposure time of 1000 h led to a significant decrease in the strain at break to a value of around 60%; this could have been related to chemical aging with chain scission and, therefore, to hydrolysis.¹⁴ After 2000 h of DH testing, the samples were brittle and broke with almost no plastic deformation. PCTs at lower temperatures (100 and 110 °C) showed slightly different curve progressions compared to the results of DH testing with respect to the desired test time. The results of samples aged at 120 °C up to 96 h came very close to the DH results. Higher exposure

temperatures (PCT at 140 °C) led to a strong embrittlement of the samples. Chemical aging processes, such as hydrolysis and chemocrystallization, might have been more dominating in the case of higher exposure temperatures.

Despite the same order of magnitude of the elongation at break results of DH and PCT at 120 °C, a comparison of the original stress–strain curves of samples aged in PCTs at 120 °C after 96 h revealed differences (see Figure 7). The samples with DH for 2000 h became brittle and broke with almost no plastic deformation at 40.3 MPa and 1.6%. In contrast to that the stress of the PCT at 120 °C for 96 h samples levelled off to around 8.5 MPa until failure occurred around 5.8%. This and the fact that a yield stress around 6.9 MPa could be detected led to the assumption that the aging mechanisms of PCT were different at 120 °C after 96 h and in a DH test after 2000 h, respectively. Supposedly, after DH aging, the PET layer could still contribute to the mechanical load. In contrast to that, the PET layer might have been too degraded after PCT at 120 °C for 96 h to contribute to the mechanical load. It appeared that the mechanical behavior was mainly affected by the PVF layer. The assumptions were supported by a slightly higher crystallization temperature from the DSC results. Similar mechanical behavior was found for samples aged in PCTs at 140 °C for 12 h. Additionally, an Arrhenius calculation for the stress at strain level also showed a nonlinear behavior because of different aging mechanisms. Different aging mechanisms were reasonable as the DH test temperature at 85 °C was around the glass-transition of PET, whereas all PCT experiments were conducted above the glass-transition temperature and at a higher humidity level than DH. As the purpose of this acceleration was to develop a faster material quality test for PET-based backsheets than 2000 h of DH, the differences in the degradation mechanisms should be acceptable.

To characterize the chemical structure and its changes after artificial aging, IR measurements were conducted. As an internal standard for the normalization of the spectra, the absorbance peak at 1408 cm^{-1} (in-plane deformation of the aromatic ring) was used, as it was considered to be invariant with crystallinity, degree of orientation, and aging.^{38–40} Typical spectra for PET were obtained for all of the samples (see Figure 8). Strong absorption bands were detected in the region between 3000 and 2840 cm^{-1} ; this corresponded to C–H asymmetric and symmetric stretching vibrations. The peak at 1715 cm^{-1} was assigned to the carboxylic ester group (C=O). The absorption peaks in the region between 1600 and 1325 cm^{-1} were due to aromatic C–C stretching, whereas the band at 1240 cm^{-1} was caused by an overlap of the stretching of ester groups and the deformation of =CH groups. The absorption bands at 1096 and 1118 cm^{-1} corresponded to the stretching of O–C and the deformation of =CH. The bands at 1017, 970, and 872 cm^{-1} were also caused by the deformation of the =CH group. The deformation of the aromatic ring structure caused the strong absorption band at 722 cm^{-1} .^{13,14,39,41}

After the accelerated aging of the samples, only few changes could be detected. Significant differences were observed for samples with DH for 2000 h and PCT at 120 °C for 96 h

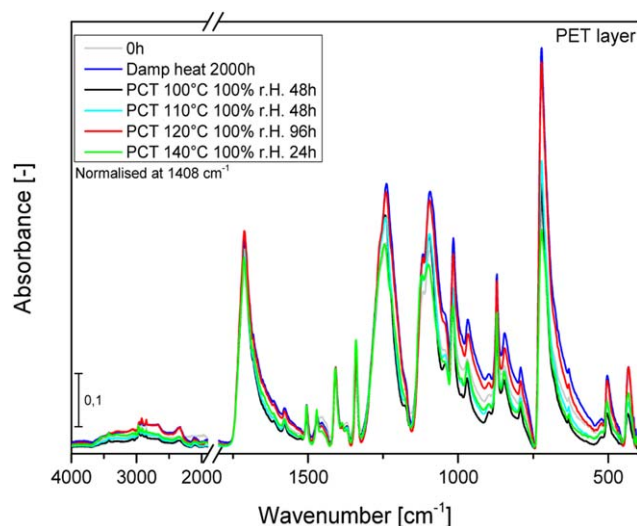


Figure 8. IR spectra of the unaged and aged samples (r.H. = relative humidity). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

compared to the virgin material. Increases in the intensity at 1712 cm^{-1} (C=O), 1683 cm^{-1} (terephthalic acid), 1240 cm^{-1} (ester, carboxyl), and 1096 cm^{-1} (ester, carboxyl) were indicators for hydrolysis, and the increase in the broad band between 3600 and 2500 cm^{-1} marked the deformation of the OH groups in a network of hydrogen bonds also due to hydrolysis.^{24,40} Even though there were some changes in the IR spectra, significant differences in the test temperatures were hard to distinguish.

CONCLUSIONS

As described previously, it was possible to determine aging-related changes by thermal analysis. The lower the exposure temperature in the PCT was, the more dominant were physical aging processes such as postcrystallization. The higher the exposure temperature was, the more dominant chemical aging processes such as hydrolysis and chemocrystallization were. The most similar test results to DH testing showed PCTs at $120\text{ }^{\circ}\text{C}$. The tensile test results showed significant changes related to aging, namely, an increase in the yield stress; this indicated physical aging processes, such as postcrystallization. Furthermore, this revealed a decrease in the strain at break values, which indicated chain scission due to hydrolysis. The mechanical findings were in good accordance with the results of the DSC measurements. Despite the good similarity of the results of PCT at $120\text{ }^{\circ}\text{C}$ and DH testing for 48 and 1000 h, respectively, the failure mechanisms at 96 h (PCT at $120\text{ }^{\circ}\text{C}$) and 2000 h (DH) were different. We assumed that the PCT at $120\text{ }^{\circ}\text{C}$ for 96 h caused more degradation than DH testing for 2000 h. Even though there were some changes in the IR spectra, differences in the test temperatures were hard to distinguish.

To summarize, the thermal and mechanical properties showed very close orders of magnitude between PCTs at $120\text{ }^{\circ}\text{C}$ and DH tests, with the constraint of presumable differences in the failure mechanism. Nevertheless, it should be acceptable to use the PCT at $120\text{ }^{\circ}\text{C}$ up to 96 h for fast quality testing to support

fast material development for PET-based backsheets instead of 2000 h of DH testing. The use of PCT ($120\text{ }^{\circ}\text{C}$ 96 h) led to an acceleration of the speed of testing by a factor of 21. PCT was proposed to significantly reduce the testing time to 4 days for the material quality and infant failures.

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