Evaluation of creep in thermoplastic encapsulant materials deployed outdoors

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ABSTRACT

There has been recent interest in the use of thermoplastic encapsulant materials in photovoltaic modules to replace chemically cross-linked materials, for example ethylene-vinyl acetate. The related motivations include the desire to reduce lamination time or temperature, to use less moisture-permeable materials, and to use materials with better corrosion characteristics or improved electrical resistance. However, the use of any thermoplastic material in a high-temperature environment raises safety and performance concerns, as the standardized tests do not currently include exposure of the modules to temperatures in excess of 85°C, even though fielded modules may experience temperatures above 100°C. Eight pairs of crystalline silicon modules and eight pairs of glass/encapsulation/glass thin-film mock modules were constructed using different encapsulant materials, of which only two were designed to chemically cross-link. One module set with insulation on the back side was exposed outdoors in Arizona in the summer, and an identical set was exposed in environmental chambers. High-precision creep measurements (±20μm) and performance measurements indicated that, despite many of these polymeric materials being in the melt state during outdoor deployment, there was very little creep because of the high viscosity of the materials, the temperature heterogeneity across the modules, and the formation of chemical cross-links in many of the encapsulants as they aged. In the case of the crystalline silicon modules, the physical restraint of the backsheet reduced the creep further.

Introduction

With the recent interest in thermoplastic photovoltaic (PV) encapsulants and adhesives, there have been increasing concerns in the standards community regarding the potential for viscoelastic creep of these materials. The current qualification and safety tests (IEC 61215, IEC 61646, IEC 61730, UL 1703) [1–4] stress modules up to a maximum temperature of only 85°C in the 'damp heat', 'thermal cycling' and 'humidity freeze' tests. Additionally, small areas of a module may reach much higher temperatures during the 'hot spot' test [5], but the localized nature of this test does not examine the conditions inherent in the hottest operating environments and mounting conditions of modules. In very hot environments, modules are known to reach temperatures in excess of 100°C [6,7]. One could envision a material with a melting point near 85°C undergoing a highly thermally activated drop in viscosity, resulting in significant creep at 100°C. To evaluate this potential scenario, modules with eight different encapsulant types were assembled using two different module constructions and exposed outdoors in Mesa, Arizona, for the summer of 2011. A replicate module set was also exposed to high temperatures in indoor environmental chambers. The results of those tests are presented in this paper, along with a discussion of the implications for qualification testing, safety standards and manufacturing practices.

> **"In very hot environments, modules are known to reach temperatures in excess of 100°C."**

Experimental

The encapsulant materials used (or under investigation for use) in PV modules were obtained from industrial manufacturers. Under an agreement with these manufacturers, we may disclose the general class of the polymeric resin and the physical properties we have measured (see Table 1). For the poly(dimethylsiloxane) (PDMS) encapsulation, a different formulation was used for the thin-film mock modules than for the crystalline silicon modules, but both are similar sparsely cross-linked gels. The PDMS values in Table 1 apply to the thin-film mock modules. Notably, a non-curing poly(ethylene-vinyl acetate) (NC-EVA) was formulated identically to a standard EVA formulation but without the inclusion of a peroxide to promote curing during lamination.

The set of silicon modules was made using forty-two 156mm upgraded metallurgical silicon cells with a final average aperture-area efficiency of 14.6%. The PDMS modules used sixty 156mm multicrystalline cells and were 13.9% efficient. The thin-film mock modules were constructed using two pieces of 3.18mm-thick, 61cm × 122cm glass. The rear surface of the back plate was painted black to simulate the optical absorption of a thin-film module. The inside of the front glass had a thin transparent conductive oxide (TCO) layer, which was removed in a 12.7mm region of the perimeter using laser ablation. The TCO was electrically connected to a ribbon passing through a hole cut in the back plate. This allowed the wet high-pot test to be carried out, to assess if creep would pose a safety or performance concern. Thin-film mock modules were mounted by adhesively attaching fibreglass

| Encapsulant material type		DSC determined transitions			DMA determined transitions at 0.1 rad/s	
		T_g (°C)	T_m (°C)	T_f (°C)	T_g (°C)	T_m (°C)
Commercial PV EVA resin	EVA	-31	55	45	-30	47
Commercial PV EVA resin with all components but the peroxide	NC-EVA	-31	65	45	-28	69
Polyvinyl butyral	PVB	15	N/A	N/A	17	N/A
Aliphatic thermoplastic polyurethane	TPU	2	N/A	N/A	3	N/A
Part-catalyzed, addition cure polydimethyl silioxane gel	PDMS	-160	-40	-80		
Thermoplastic polyolefin #1	TPO-1	-43	93	81	-35	105
Thermoplastic polyolefin #3	TPO-3	-44	61	55	-41	79
Thermoplastic polyolefin #4	TPO-4	-34	106	99	-21	115

Table 1. Phase transitions determined by differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA). DMA glass transitions (T_g) were determined at the peak of the phase angle, and the DMA melting transitions (T_m) were determined when the phase angle was 45 degrees, except for the cross-linked PDMS and EVA, for which an inflection point in the modulus was used.

channels to the back, allowing the front piece of glass to move freely.

For the thin-film mock modules, the creep (displacement of the front glass relative to the back glass) was measured using a high-precision depth gauge with ±1μm increments. This gauge was mounted to a flat plate to ensure that it was positioned perpendicular to the side of the module and in the plane of the glass. By using this set-up, creep measurement reproducibility was better than ±20μm.

Modules were deployed in Mesa, Arizona, from May to September 2011 on a rack at a 33-degree latitude tilt and azimuth of 255 degrees so that they would face the sun more directly at the hottest part of the day. Additionally, a single NC-EVA thin-film mock module was exposed in Golden, Colorado, at a 40-degree tilt and 180-degree azimuth [8]. For both the thin-film and silicon module types, insulation was placed on the back side to simulate a rooftop installation (resulting in maximum measured temperatures between 102 and 104°C in Mesa, Arizona). The temperature of the modules was monitored by placing thermocouples underneath the insulation on the back side of each module. One thermocouple was placed in the centre of each module, and the other about 7.5cm diagonally inward from one corner of the module.

Following deployment in the field, the formation of polymer chain cross-links on the Arizona-fielded, NC-EVA, thin-film mock module was evaluated using size-exclusion chromatography (SEC) in conjunction with multi-angle laser light scattering (MALLS, Waters Corporation GPCV 2000 instrument) and viscometric detection (using a capillary viscometer detector CV from Waters). Samples were cut using a ceramic saw blade, which allowed them to be taken
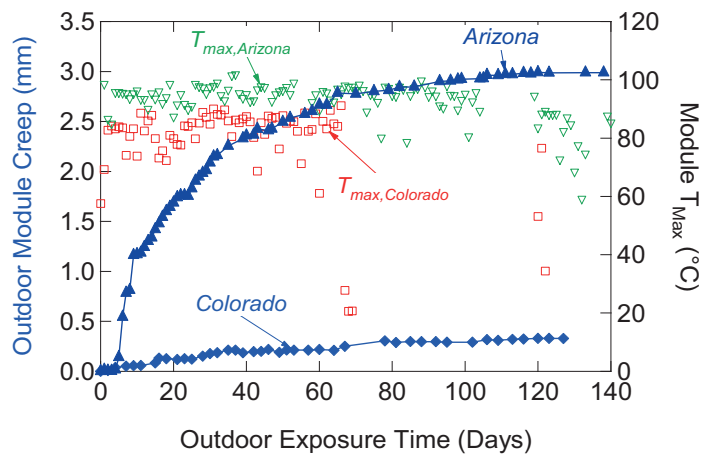
Figure 1. Measurement of thin-film mock module creep during outdoor exposure: module displacement and daily maximum module temperature for the NC-EVA modules deployed in Arizona and Colorado.
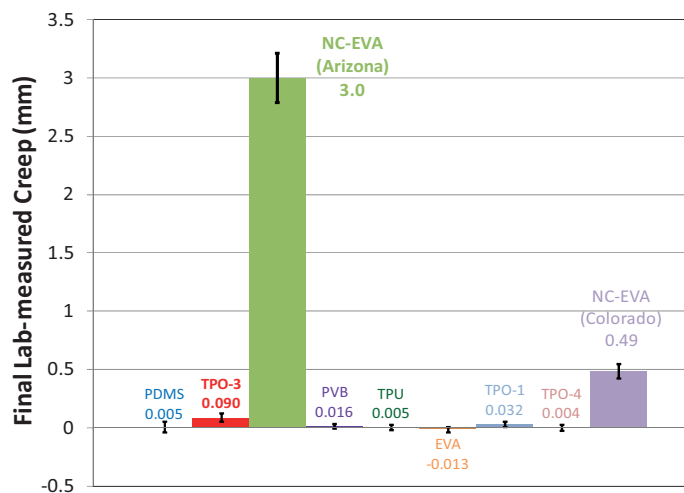
Figure 2. Detailed indoor measurement of thin-film mock module creep after field deployment in Arizona and Colorado.

at different distances from the edge. Polymer was removed from the glass by extraction in a solution of tetrahydrofuran (THF) for 72 hours; however, this left behind approximately 60% of the EVA as an insoluble fraction. The residual glass-EVA specimens were soaked in trichlorobenzene (TCB) overnight at 150°C to solubilize the remaining EVA. Samples of 'virgin' (unexposed) NC-EVA film were dissolved in THF and TCB at room temperature and 150°C, respectively, to serve as controls. Solutions were made with approximately 1mg/ml concentrations.

Results

Outdoor testing

Of the outdoor exposed modules, only the thin-film mock modules constructed using NC-EVA experienced significant creepage, moving 3.0±0.2mm over the course of the summer (Fig. 1). Despite this, the NC-EVA modules still passed the wet high-pot test. A more detailed indoor measurement after the exposure indicated that the TPO-3 module crept 0.090±0.036mm and the TPO-1 module crept 0.032±0.024mm (Fig. 2). Even though most of these thermoplastic encapsulants reached either the melt or rubbery state during exposure, no significant movement was observed.

> **"Of the outdoor exposed modules, only the thin-film mock modules constructed using NC-EVA experienced significant creepage."**

In Fig. 1 note the absence of creep beyond day 110 in Arizona and the general reduced creep rate when the maximum temperature is lower during the first 60 days or so; moreover, the Colorado-deployed module barely crept and rarely reached temperatures above 90°C. These observations indicate that creep is possible for uncured EVA when the maximum module temperature approaches around 90°C. Because the modules were mounted individually (i.e. so as to allow air to flow between them), as opposed to a closely packed installation, the temperature was between 10 and 15°C cooler at the edges of the modules during the hottest part of the day. The temperature differential appeared in both the thin-film mock and the crystalline Si modules, but the presence of an Al frame in the crystalline silicon modules is expected to make it more difficult, though not impossible, to realize an installation configuration that would significantly reduce this temperature
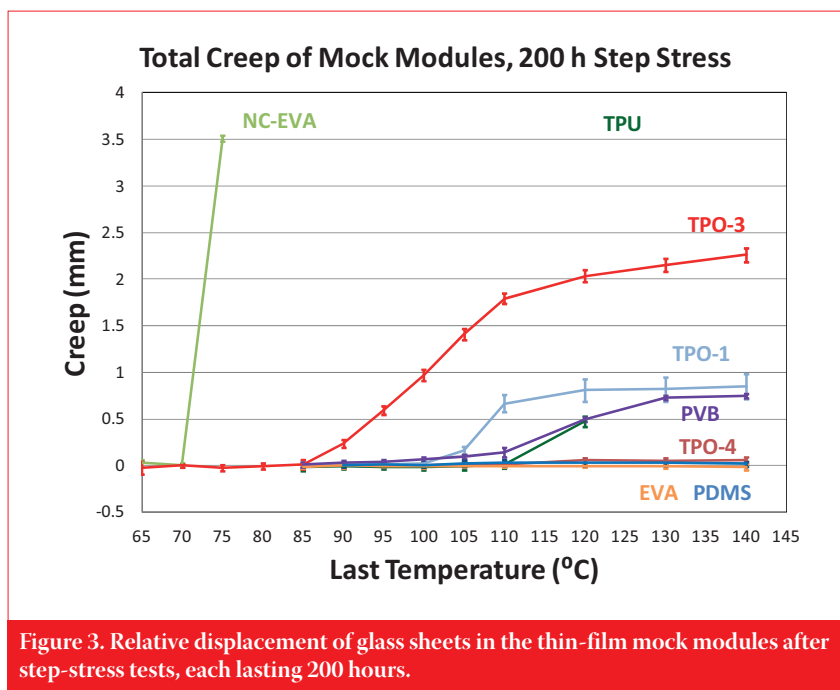

Figure 3. Relative displacement of glass sheets in the thin-film mock modules after step-stress tests, each lasting 200 hours.

heterogeneity. It is this temperature differential that helps to reduce the creep considerably, despite the fact that most of the module is well above its melting point of around 65 to 69°C.

Over the first 30 days, creep increased more quickly in the Arizona module, but slowed down in mid-summer even though the temperature did not fall appreciably. Typical EVA formulations are known to cross-link as they age in the field [9]. This reduction in creep rate suggests that, even without the peroxide additive, NC-EVA is cross-linking at these high temperatures.

Indoor testing

The modules were also examined indoors in a step-stress experiment. Modules were mounted vertically, with the crystalline Si modules simply resting on their frames, and the thin-film mock modules mounted vertically using adhesive on their back sides, allowing the front glass to move (Fig. 3). More creep is seen in indoor testing than in outdoor experiments at lower temperatures, where the cool perimeter of the outdoor modules limits motion. The NC-EVA began creeping detectably at 75°C in the thin-film mock module construction, and at 80°C in the crystalline Si construction. Similarly, the TPO-3 and TPO-1 thin-film mock modules began to creep detectably at 90 and 105°C respectively. Furthermore, the slope of the creep vs. temperature curve for TPO-3 does not continue to increase rapidly beyond 95°C, suggesting that it is chemically cross-linking at temperatures above 90°C. At even higher temperatures, above 120°C, the amount of creep in each cycle begins to plateau for TPO-3. Similar behaviour is also observed for TPO-1, TPO-4 and polyvinyl butyral (PVB). The only thermoplastic material that did not

appear to cross-link as it aged was the thermoplastic polyurethane (TPU). In this case, after exposure at 130°C, the module plates were displaced by more than 1cm where they contacted the chamber floor. In addition, starting at 105°C a large number of bubbles began to appear within the module, and became present throughout the module after exposure to 110°C.

> **"The only thermoplastic material that did not appear to cross-link as it aged was the thermoplastic polyurethane (TPU)."**

Molecular weight changes

The cross-linking of the NC-EVA in the fielded module was verified using SEC in conjunction with MALLS and viscometry detection. Three samples were removed from the modules: one was cut from the edge of the module, one from about 2cm in from the edge, and one from about 4cm in. Fig. 4 shows that the THF-insoluble, TCB-solubilized sample taken from the edge of the NC-EVA module deployed in Arizona has a lower molecular weight distribution than the unaged TCB-solubilized control sample, indicating that chain scission has dominated over polymer cross-linking. In contrast, the samples taken at distances of 2cm and 4cm in from the edge have a higher molecular weight than the control, indicating that cross-linking has dominated over chain scission. However, all the aged samples show changes in intrinsic viscosity relative to the control, which is a sign of some degradation, causing changes in the degree of branching of the polymer chains.

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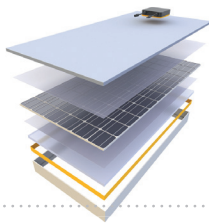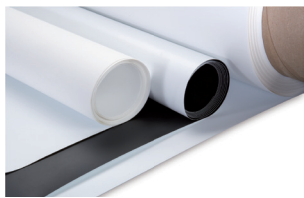
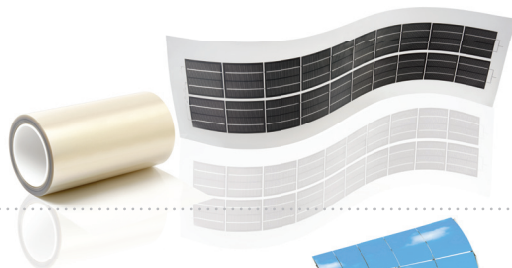**3M™ Scotchshield™ Films**
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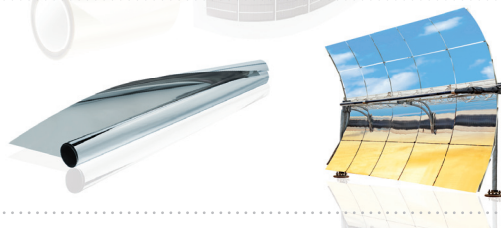
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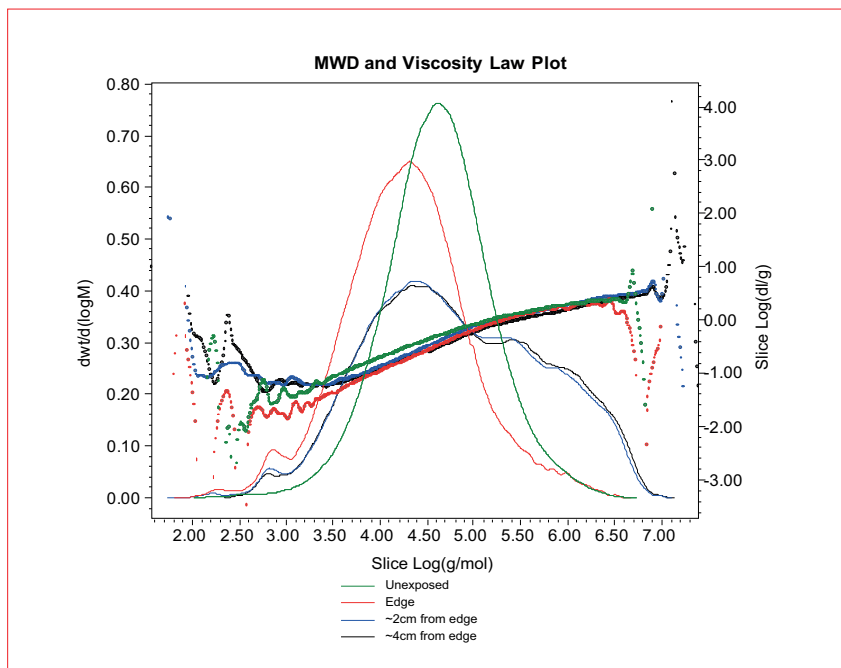for numerous applications in photovoltaic modules.

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MWD and Viscosity Law Plot

Unexposed
Edge
~2cm from edge
~4cm from edge

Figure 4. SEC-MALLS and viscometry results from the NC-EVA thin-film mock modules after exposure in Arizona. Samples were dissolved in trichlorobenzene (TCB) by extraction at 150°C overnight. The thin curves show the weight fraction of slices (left axis); the thick curves show the intrinsic viscosity of slices (right axis).

temperature of 50°C for the corner and 60°C for the centre thermocouples [12,13]. At 50°C, the diffusivity of water in EVA is $1.6 \times 10^{-6} \text{cm}^2/\text{s}$. For the deployment time of 140 days, the characteristic penetration depth (x) was around 4.1cm ($x = \sqrt{Dt}$) [11]. In contrast, for a distance of 2cm the characteristic time is 29 days.

Marias et al. [14] compared the permeation characteristics of H_2O and O_2 in 33 wt% VA EVA at 25°C and found it to be selectively permeated by water 350× as fast as by oxygen, at the same vapour pressures, principally because of differences in solubility. If we use 10% RH in Arizona at 25°C for the water content, the partial pressure of water is about 0.24cmHg, as compared to 21% oxygen with 16cmHg, which means that water permeates EVA about 5.2× as fast as oxygen does. This makes it more likely that oxygen is the limiting reagent enabling the shift from cross-linking dominated reactions to chain-scission dominated reactions. However, more research is necessary to verify the kinetic pathways to confirm which component is limiting the reaction.

The reduction in creep rate of field-exposed modules and the formation of cross-links as shown by SEC-MALLS indicate that in anaerobic and anhydrous conditions, EVA will cross-link when exposed to heat and UV light, even without the addition of peroxide-based cross-linkers to the formulation. Because the thin-film mock modules were constructed so that light passed through the polymer, it is not known if these effects are driven by heat, UV light or a combination of the two. However, it does indicate that typical EVA formulations would be expected to cross-link with time, reducing the potential for thermally induced creep.

This result is supported by numerous observations of fielded modules in which discoloration of EVA is observed in the centre area of a cell, and yet greater oxygen incorporation is seen around the cell perimeter [10]. Similarly, it has been observed in aged samples that the gel content in the more highly yellowed EVA at the centre of the cell is higher than in the EVA at the perimeter [9]. Thus, yellowing and cross-linking are correlated to lower oxygen incorporation, and oxidative bleaching and lower cross-link densities are correlated to higher oxygen incorporation.

For the THF soluble fraction, very little difference in the molecular weight distribution was seen between the EVA samples extracted from the fielded modules and from the unstressed EVA (Table 2). The THF solution was only able to dissolve about 40% of the polymer. It therefore appears that any degradation causing chain scission or cross-linking renders EVA significantly less soluble in THF.

Discussion

Oxidation and cross-linking

There are many different chemical pathways possible for producing cross-linking in polymer chains. For NC-EVA, the fact that the formation of cross-links varies with position in the sample indicates that some chemical species must be entering or leaving the module package, thus affecting the kinetics. Starting from the edge and proceeding inwards, there are gradients in temperature, water content and oxygen content. The temperature gradient from the edge to the centre is at most around 20°C at the hottest part of the day (as indicated by infrared imaging). This would not be expected to create by itself large differences in the reaction kinetics over a 2cm distance. The diffusivity of water in EVA has an activation energy of about 38kJ/mol (0.40eV) [11], which was used to estimate an Arrhenius activation energy-weighted effective module

Creep and its consequences

Because the three TPOs and PVB did not have exponential-like increases in creep with temperature, it is concluded that exposure to high temperatures caused cross-linking reactions to dominate over chain scission. Therefore, as in the case

Sample		Mn [g/mol]	Mw [g/mol]	Mz [g/mol]	PDI	MP [g/mol]
THF soluble fraction	Unexposed	20,400	97,800	448,000	4.8	42,800
	Edge	19,900	89,100	379,000	4.5	40,300
	~2cm from edge	19,900	84,600	326,000	4.2	37,900
	~4cm from edge	21,300	104,000	437,000	4.9	40,900
TCB soluble fraction	Unexposed	15,600	97,800	512,000	6.3	44,400
	Edge	5,540	66,800	632,000	12	21,100
	~2cm from edge	8,720	394,000	2,220,000	45	25,000
	~4cm from edge	10,600	382,800	2,050,000	36	22,000

**Table 2. SEC-MALLS results.**

of EVA, it is probable that they too would cross-link with time in field exposure. In contrast, the TPU thin-film mock module began to creep at 100°C, and a large number of bubbles formed in the interior of the module. At higher temperatures the effective viscosity dropped dramatically and the front plate contacted the bottom of the chamber, causing creep that was too high to be measured in these chambers (see the dashed arrow in Fig. 3 for TPU). The TPU seemed to degrade dramatically at elevated temperatures. On the other hand, all the polymers with only carbon–carbon bonds in the backbone (EVA, TPOs and PVB) appear to cross-link when exposed to high temperatures. This will help to mitigate the potential for creep in these thermoplastic encapsulant materials.

The onset of creep correlates reasonably well with the melting points determined by DMA using the phase angle of 45 degrees (Table 1). The three TPOs showed much greater creep than PVB, despite the glass transition of the latter at 16°C because of its greater viscosity [8]. However, DMA measurements indicate the phase angle of PVB reaches 45 degrees, when measured at 0.01 rad/s and at a temperature of 115°C, which correlates better with the onset of its creep.

> **"None of the crystalline Si modules exhibited measurable creep when deployed outdoors."**

None of the crystalline Si modules exhibited measurable creep when deployed outdoors. Only the TPO-3 module demonstrated a statistically significant performance loss, which was due to a cracked cell, presumably weakened or cracked during lamination because of TPO-3's higher viscosity relative to EVA [8]. Despite reaching what could be considered nearly the highest temperatures possible for a fielded module, none of the crystalline Si modules experienced a detectable safety or performance failure as a result of using a thermoplastic encapsulant. However, the modules were mounted with the cell strings arranged vertically. If the NC-EVA module had been mounted with the cell strings horizontal, it is more likely that some creep would have occurred in the cells in the centre of the module. Subsequent accelerated stress tests (humidity freeze) will be performed on these modules to investigate if longer term deployment results in deformation and/or performance degradation.

In indoor studies, the onset of creep for the NC-EVA silicon module occurred at 75°C. In this case, despite the fact that the sides of adjacent rows of cells were touching, there was no discernible performance loss with step-stress tests up
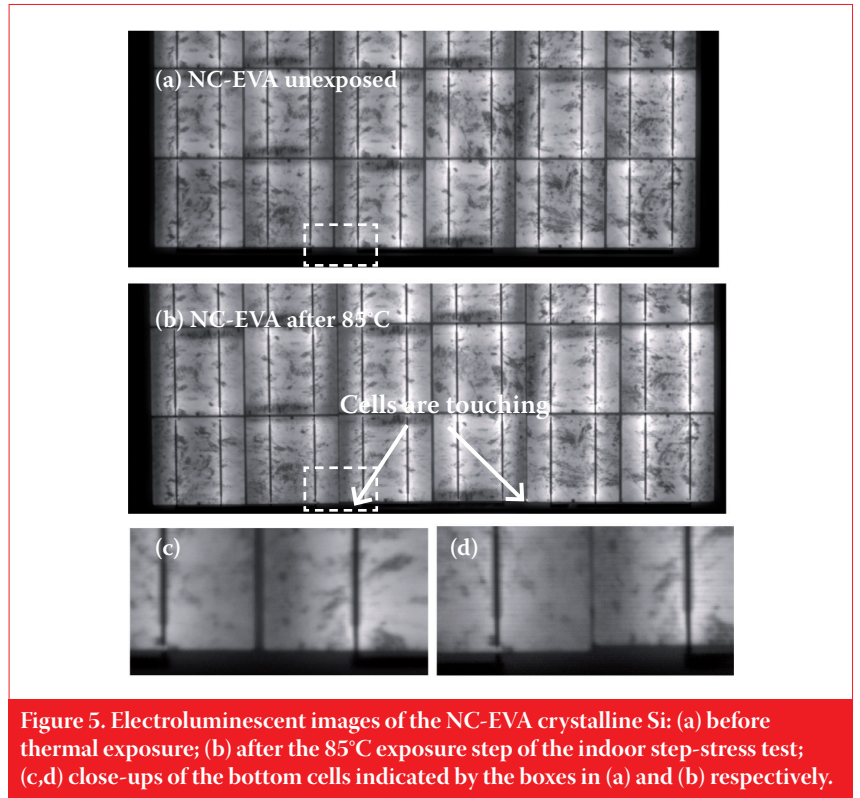
Figure 5. Electroluminescent images of the NC-EVA crystalline Si: (a) before thermal exposure; (b) after the 85°C exposure step of the indoor step-stress test; (c,d) close-ups of the bottom cells indicated by the boxes in (a) and (b) respectively.

to 100°C (Fig. 5). Although this significant cell movement did not directly create a performance issue, it is likely that extended exposure to thermal cycling after this amount of creep would increase the mechanical stress on the tabbing and solder bonds, leading to higher failure rates in the long term.

Conclusions

The results presented here indicate that the potential for creep of the examined thermoplastic materials is negligible for the majority of PV installations. When a typical polymer-backed crystalline Si module was exposed outdoors in Arizona during the summer, with insulation on the back to simulate a rooftop mounting configuration, the module did not creep, even when made with EVA containing no cure chemistry. Thus, it was not possible to detect any short-term durability or safety issues likely to result from completely uncured EVA.

> **"To assess the potential for creep, manufacturers should consider not only the location of phase transitions and the viscosity at temperatures between 85 and 100°C, but also the effects of degradation on those material properties."**

Only modules with an unrestrained front glass made of glass were shown to have any propensity to creep outdoors. This is due in part to the non-uniformity of temperature, resulting in small areas that significantly resist creep. Evidence that NC-EVA, TPO-1 and TPO-3 thermally cross-link, despite the absence of peroxide, was also presented. This unintended cross-linking actually serves to mitigate the potential for creep in some materials.

The use of 85°C in IEC standards necessitates cross-linking of EVA to achieve gel contents in excess of around 60%. However, even if the EVA of a framed, polymer-backed Si PV module had a very low gel content, it would have to be deployed in an extreme environment during the hottest time of the year, in close contact to a mounting structure that restricts heat transfer, for there to be a significant chance of creeping before it eventually thermally cross-linked on its own [9]. Therefore, to assess the potential for creep, manufacturers should consider not only the location of phase transitions and the viscosity at temperatures between 85 and 100°C, but also the effects of degradation (chain scission or cross-linking) on those material properties.

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