

Effect of crosslinking on EVA-based encapsulant properties during photovoltaic module fabrication process

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Abstract - The key objective of this work is to create a comprehensive comparison between experimental analysis methods for detecting crosslinking reaction in EVA encapsulant material during PV module fabrication process. Several analytical methods, spanning from classical gel content extraction measurements over different thermo-analytic and mechanical approaches to spectroscopic analysis as Thermally Stimulated Current (TSC), have been investigated as to their ability to reveal the crosslinking state of ethylene vinyl acetate (EVA). The basic results show that TSC complements perfectly other thermal analysis methods like as Differential Scanning Calorimetry (DSC), Dynamic Mechanical Thermal Analysis (DMTA) to determine fundamental properties of EVA such as molecular mobility characteristics and also to monitor the crosslinking reaction. Exothermic peak as revealed by DSC analysis can be used to estimate the crosslinking rate of EVA during PV module encapsulation process as fast quality control test.

Résumé - L'objectif principal de ce travail est de permettre une meilleure compréhension entre les différentes techniques analyses expérimentales pour détecter la réaction de réticulation au niveau de l'encapsulant Ethylène-Vinyle Acétate (EVA), durant le process de fabrication d'un module PV. Plusieurs méthodes d'analyse, allant du test classique d'extraction du taux de gel aux différentes techniques d'analyses thermomécaniques ainsi que les techniques d'analyse de spectroscopie comme le courant stimulé thermiquement (TSC), ont été étudiés pour permettre de révéler le phénomène de la réaction de réticulation de l'encapsulant l'EVA. Les résultats de base montrent que la TSC complète parfaitement d'autres méthodes d'analyses thermiques comme la calorimétrie différentielle à balayage (DSC), l'analyse mécanique dynamique (DMTA) pour déterminer les propriétés fondamentales de l'EVA. Ces techniques permettent de déterminer les caractéristiques relatives à la mobilité moléculaire et aussi pour mieux mettre en évidence la réaction de réticulation. Le pic exothermique comme révélé par l'analyse par DSC, peut être utilisé pour estimer le taux de réticulation de l'EVA au cours du process d'encapsulation du module PV comme un test rapide de contrôle de qualité.

Keywords: EVA - Photovoltaic module - Crosslinking process – Thermal properties - Mechanical properties.

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1. INTRODUCTION

The polymer encapsulant used in PV modules serves to provide the functions of structural support, electrical isolation, physical isolation/protection and thermal conduction for the solar cell circuit [1]. Normally the encapsulant materials are made of a high insulating polymer to maintain good circuit isolation.

The relative stability is crucial to the long term durability of the encapsulant material for PV modules deployed in the field, where along service lifetime and reliable power output are needed [2]. Also, it is important to provide a method for determining that the EVA encapsulant has sufficient crosslinking density to be capable of surviving outdoors for 25 years and of passing the IEC module qualification tests.

This method may be used to assess the quality of the EVA used in fabrication (where the peroxide agent facilitating the cure has a finite shelf-life) and may be used to establish (or verify) the time temperature conditions used during lamination.

DSC analysis technique can provide a method for measuring the crosslinking density in EVA encapsulant, or verifying adequate completion of the curing process after it has been processed in a PV module.

Knowledge of exact thermal stability of EVA based encapsulant and how they vary during thermal processing is important to understand the effect of crosslinking.

The crystallization and the crosslinking properties of EVA experienced during encapsulation process are so examined by DSC analysis. An understanding of the crystallization of EVA is important as the degree of crystallinity affects mechanical and processing properties of the material. In this paper the thermal stability of EVA will be defined and their dependence on temperature will be presented.

2. EXPERIMENTAL

2.1 Material and lamination-crosslinking process

EVA Sample, prepared with commercial EVA standards based on 9918P, is referred to as original EVA [3]. EVA samples were inserted between two pieces of Teflon and placed on a piece of glass of the same size as used for the actual module in order to produce the correct thermal conditions during the crosslinking cycle is executed using the Spi-Laminator at temperature of 160°C for 15 min [4].

EVA sample submitted to the cure step cycle is referred to as crosslinked EVA. The gel content (degree of crosslinking) produced during the cycle cure is about 70% [5].

2.2 Analysis techniques

2.2.1 Thermally Stimulated Current Technique (TSC)

The TSC experiments provide information about the mobile phase in polymeric materials. TSC experiments were run in depolarization mode (TSDC). The TSDC procedure was to apply a voltage $E_p = 200$ V across the EVA specimen and ramp the temperature from -50 °C to 85 °C at heating rate of $7^\circ\text{C}/\text{minute}$.

Under polarization temperature $T_p = 60^\circ\text{C}$ and at temperatures sufficient for dipole mobility, the dipoles will align without the imposed field and produce a polarization current as illustrated in figure 1.

2.2.2 Differential Scanning Calorimetric (DSC)

For DSC, a measuring instrument 204 F1 Phoenix®, Netzsch, Germany, was used. Standard crucibles with a manually perforated lid were deployed. A circular sample was punched out from the EVA foil. A heat-cool-heat temperature programming protocol was used in DSC experiments to evaluate the behavior of the EVA samples from -80°C to 200°C . The heating and cooling rates were $10^{\circ}\text{C}/\text{min}$.

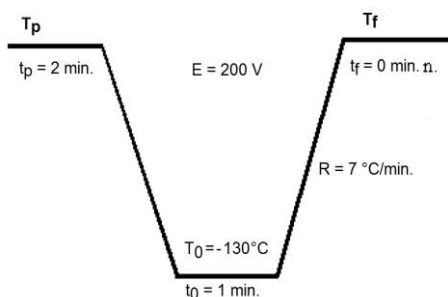


Fig. 1: Thermal program for the TSDC experiments

2.2.3 Dynamic Mechanical Thermal Analysis (DMTA)

Thermomechanical properties of the EVA samples were measured using a Rheometric DMTA IV. Specimens were cut from the samples and mounted in the rectangular tension fixture. The typical sample dimensions were 10 mm long, 3.3-3.5 mm wide and 0.4-0.5 mm thick. The frequency was 1 Hz and the static force was maintained at 20% higher than the dynamic force

2.2.4 Thermogravimetric Analyzer (TGA)

Thermogravimetric Analysis (TGA) measures changes in weight of a sample with increasing temperature. This, in principle, simple measurement is an important and powerful tool in solid state chemistry and materials science. The method for example can be used to determine thermal stability of materials and also the reaction kinetics. TGA measurements can be conducted in inert and oxidative atmosphere on a TA Instruments TGA 2950 Thermogravimetric Analyzer, which has a sensitivity of 0.1 mg.

2.2.5 Tensile properties

The tensile testing is the most versatile of the applied characterization methods in describing the aging behavior of polymer materials. Tensile tests were carried out with a screw driven universal test machine (Zwick Z010 Allround-Line, Zwick, Ulm, D) at 23°C according to EN ISO 527- 3. Figure 2 shows the specimen test dimensions. Rectangular specimens of 100 mm in length and 15 mm in width were cut using a device with fixed razor blades and rotating sample. The test speed was 50 mm/min. From a total of at least five specimens for each test series, average numbers for elastic modulus (E_t), stress-at-break (σ_b) and strain-at-break (ε_b) were deduced [6].

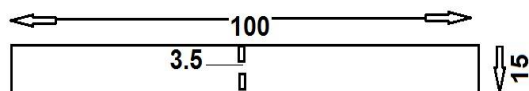


Fig. 2: Sample dimensions for tensile test

2.2.6 Electrical resistivity measurements

For evaluating the electrical properties of EVA an experimental set-up was installed according to the IEC recommendations system. EVA samples are contained between the upper and lower metal plate electrodes within a confinement ring soldered to the lower plate. The electrodes are connected to heating resistances to allow measurements to be taken at several temperatures up to 150°C which are stabilized with a temperature regulator type 2965 (Tettex). The electrodes pressure is kept constant and equal to 2, 5.104 Pa. The applied voltage is adjusted to a value of 500 V_{AC} and frequency of 50 Hz to favor optimal sensibility of measurements. Insulating resistance (R) was measured with a Megohmmeter type MOM11. The resistivity (ρ) is deduced from the measurements [7].

3. RESULTS AND DISCUSSIONS

3.1 Thermal relaxations by TSC

Figure 3 provides the TSC results in TSDC mode for EVA sample polarized at 60°C before and after crosslinking process. The TSDC behavior of EVA, exhibits two peaks represented mobility in two distinct phase separated amorphous domains. The increase in peak magnitude for the low temperature relaxation likely reflects an increase in the number of mobile dipoles in that domain. At polarization temperature of 60°C, for each EVA material, there is a low temperature relaxation process and a high temperature process. Original EVA has a low temperature peak at about -37°C, and a high temperature peak at about 30°C. The high temperature peak appears to have a shoulder above 50°C. The crosslinked EVA has a somewhat broad low temperature peak at about -38°C, and a high temperature peak at about 26°C. The complex behavior of this peak suggests that there are two processes occurring in this temperature range; first there is the relaxation characteristic of the glass transition (T_g) and second, there is the crystal melting as indicated in the DSC results as discussed below.

We observe a decrease in current intensity for the crosslinked EVA particularly for the high glass transition temperature. There is a large reduction in the depolarization current that is especially apparent for the high temperature peak.

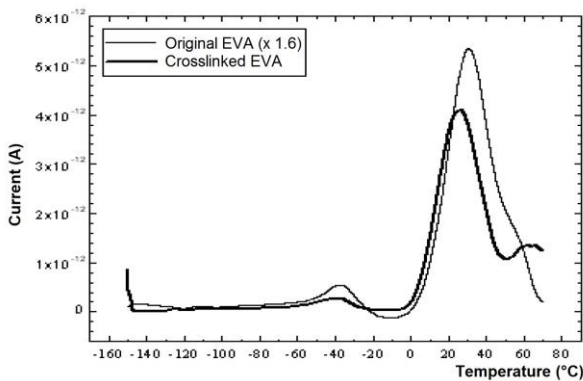


Fig. 3: TSDC curves of the original and crosslinked EVA at polarization temperature of 60°C

We observe a decrease in current intensity for the crosslinked EVA particularly for the high glass transition temperature. There is a large reduction in the depolarization current that is especially apparent for the high temperature peak.

However, it is clear that in the high temperature transition, there is a distinct difference between the original and crosslinked EVA. This strongly suggests that the EVA is crosslinked selectively in the high temperature phase, and this is consistent with the large reduction in depolarization current for the high temperature T_g in the TSC data.

3.2 Thermal transition by DSC

A heat-cool-heat temperature programming protocol was used in DSC experiments to evaluate the behavior of the cured EVA sample. Figure 4a- shows the DSC results for the original and crosslinked EVA sample in the first heating program. The distinctive feature of these results is that there are two endothermic processes [8]. There is a low temperature event with peaks at temperature 50.9°C and 47.3°C for original and crosslinked EVA respectively. After cooling and during the second heating, Fig 4b- shows that the low endothermic peaks are shifted to high temperatures and are located at 60.6°C and 56.3°C for original and crosslinked EVA respectively. Also, the DSC peak does not correspond to anything that was observed in the TSC experiments.

The second relevant feature is the exothermal reaction peak of the crosslinking reaction, which occurs at 162.4°C and 158.9°C for original and crosslinked EVA respectively. This peak, which relates to the reaction heat generated in the radical crosslinking reaction, varies strongly with the (residual) amount of crosslinking agent in the EVA samples as will be more described in paragraph 3.7.

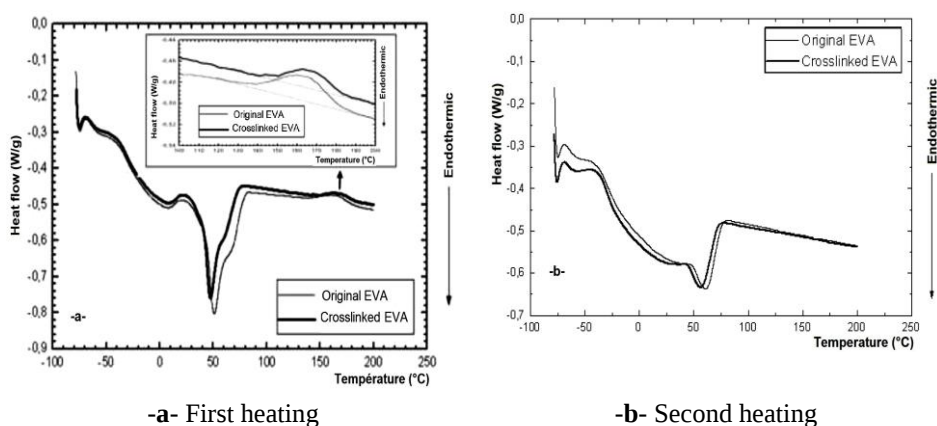


Fig. 4: DSC curves of the original and crosslinked EVA

The DSC curve also provides the crystallization behavior of EVA during lamination process. Usually the encapsulation process of solar cell module is carried out under pressure at elevated temperatures with the range of 140°C to 160°C. The EVA encapsulant is exposed to these conditions for several minutes. The EVA materials undergoes both melting and re-crystallization. The re-crystallization of the polymer in the interlayer can lead to loss of optical quality. It is important to minimize the recrystallization of EVA during solar cell encapsulation process. In order to examine the

effect of crosslinking process on crystallinity of EVA, the degree of crystallinity was calculated via the total enthalpy method [9] according to the {Eq. (1)}:

$$\chi_c (\%) = \frac{\Delta H_m}{\Delta H_{100}} \times 100 \% \quad (1)$$

where χ_c is the degree of crystallinity, ΔH_m is the specific enthalpy of melting of the sample studied and ΔH_{100} is the specific enthalpy of melting for 100% crystalline Polyethylene (288 J/g).

Since the previous thermal history of a polymer affects the calculated degree of crystallinity, EVA crystallinity was evaluated after being subjected to a second heating designed to remove the effect of prior thermal history [10, 11]. The degree of crystallinity of EVA before and after crosslinking remains unchanged and is calculated to be 5%. Therefore, the crystallinity of EVA depends only on the actual cooling rate of the lamination process, but not on the heating rate during endothermic melting. The recrystallization degree represents by endothermic melting enthalpy depicts the overall crystallinity in EVA.

3.3 Measurement of the crosslinking rate in EVA before and after encapsulation process

The most important method for measuring the crosslinking degree of an EVA is solvent extraction method. In addition to this, DSC curve may also provide information about the curing degree of the polymer encapsulant. The ratio of exothermic enthalpy of crosslinked EVA at different laminating temperatures to as received EVA can be used to estimate the gel content of EVA. The crosslinking rate (C_r) or gel content is estimated by the {Eq. (2)} from the exothermic peak as follow:

$$C_r (\%) = \frac{\Delta H_e}{\Delta H_c} \times 100 \% \quad (2)$$

where, ΔH_c , original EVA enthalpy (W/g); ΔH_e , crosslinked EVA enthalpy (W/g).

The enthalpy value is deduced from the area of exothermic peak of DSC curve during the first heat in the range of temperatures from 120°C to 200°C, where the crosslinking reaction is nearly completed after about 8.5 minutes, visible in a sloping-off of the curve. The original and crosslinked EVA enthalpy is to be 4.1176 J/g and 2.764 J/g respectively. Therefore, the crosslinking rate obtained by DSC method is 67 %. In the second measurement, no indication of incomplete crosslinking is found, as the heat flow shows a straight line and no remaining exothermic peak as revealed by figure 4b-.

The gel content of EVA obtained by solvent extraction is within 4% upper than that by DSC curve. The DSC method detects the enthalpy changes of crosslinking reaction, even when a slight amount of reaction occurs. However, solvent extraction method measures the amount of undissolved EVA during solvent extraction treatment to calculate the curing degree of crosslinked EVA.

Since the slight crosslinked EVA may dissolved in solvent, the DSC method will provide more accurate data of crosslinking reaction extent. Moreover, there is another advantage of using DSC method over solvent extraction treatment, time consuming and not so environmental friendly procedures undertaken by solvent extraction method.

However, the error of gel content measured by DSC curve may increase when the exothermic enthalpy of crosslinking reaction is small.

3.4 Dynamic thermo- mechanical properties by DMTA

DMTA analysis is used to evaluate the mechanical relaxations in EVA samples. The DMTA curves represented by the storage modulus (E') and loss tangent ($\tan \delta$) of the EVA during encapsulation process are illustrated in figures 5 and 6 respectively. DMTA analyses revealed that the storage modulus increases dramatically from 50 MPa to 2 GPa.

In the same process, the loss tangent curve exhibits two relaxation peaks in good agreement with the TSC thermogram. Crosslinked EVA exhibits lower loss tangent than original EVA due to the effect of crosslinking.

EVA exhibits at least two distinct drops in modulus. The low temperature drop is accompanied by a peak in the $\tan \delta$ as revealed in figure 5. This low temperature mechanical loss process is taken to correspond to the low temperature relaxation peak observed in the TSDC [12].

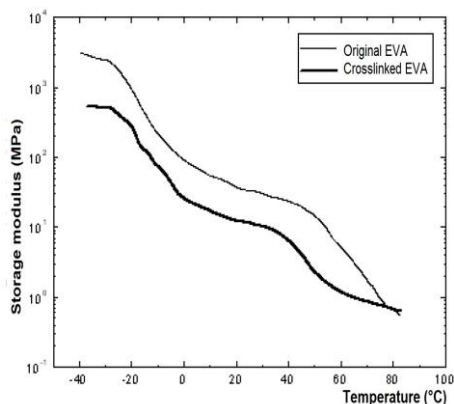


Fig. 5: Variation of storage modulus of EVA before and after crosslinking process

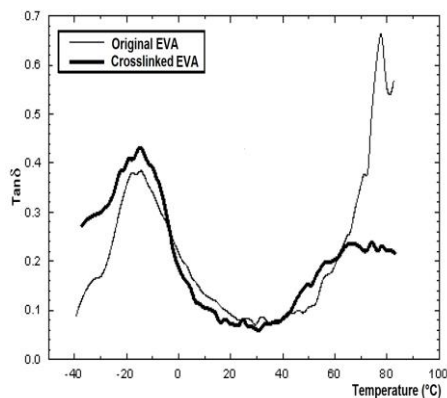


Fig. 6: Variation of loss tangent factor of EVA before and after crosslinking process

3.5 Static mechanical properties by tensile tests

The ultimate mechanical properties of EVA before and after crosslinking process are shown in **Table 1** and **2** respectively. These tables summarize the most important static mechanical properties like elastic modulus, strain-at-break and stress-at-break.

Basically, and in agreement with requirement profile for elastomeric encapsulant, all materials show a highly ductile behavior, high flexibility and pronounced yield point.

Evaluation of the average elastic modulus, i.e. the slope to the stress- strain curve in the elastic deformation region at low strains, yielded values of 10 MPa and 6 MPa for original and crosslinked EVA samples respectively.

It is noted a moderate decrease in elastic modulus due to the crosslinking reaction which is in good agreement with DMTA results.

Table 1: Tensile test results of original EVA

	E_t (MPa)	σ_b (MPa)	ε_b (%)
Probe 1	9.90	5.87	1511.21
Probe 2	10.02	5.56	1435.08
Probe 3	9.64	5.55	1364.15
Probe 4	9.72	4.76	1281.54
Probe 5	10.53	5.87	1382.74
Mean value	9.96	5.52	1394.94
Deviation	0.35	0.45	85.28

Table 2: Tensile test results of crosslinked EVA

	E_t (MPa)	σ_b (MPa)	ε_b (%)
Probe 1	5.95	3.34	1010.33
Probe 2	5.95	4.45	1127.74
Probe 3	5.28	3.33	1090.85
Probe 4	7.15	5.55	1189.52
Probe 5	7.19	5.15	1116.24
Mean value	6.30	4.36	1106.94
Deviation	0.84	1.02	65.06

Figure 7 shows exemplary stress-strain curves of EVA before and after crosslinking process for the test probe 3, illustrating the general stress-strain behavior of the EVA materials.

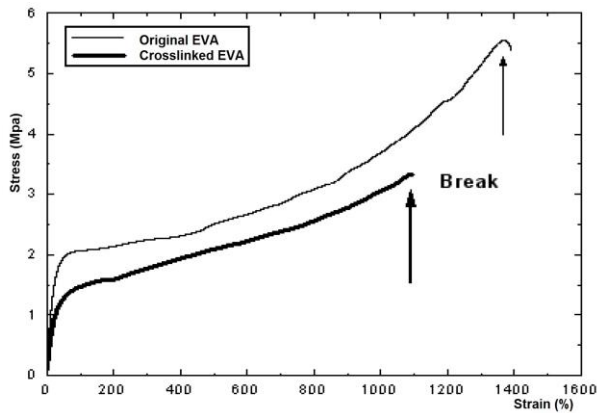


Fig. 7: Stress-strain curves of EVA before and after crosslinking process

Otherwise, significant changes could be observed in the post-yield region. While all samples exhibited an essentially bi-linear stress-strain relation-ship, the crosslinked EVA samples are less significantly stiffer in this region and the strain-at-break values are lower [13].

Regarding tensile testing results, the original and crosslinked EVA material exhibit a high ductile behavior with the average strain at break (stress at break) values of about 1366% and 1110% (5.52 MPa and 3.32 MPa) respectively. This can be attributed to the

three-dimensional widely meshed polymer network, which restricts re-orientation and slipping of the polymer chains, thus significantly constraining the plastic deformation. Also, for the investigated EVA samples a clear influence of crosslinking process parameters as time, temperature and pressure is observed. Strain-at-break and stress-at-break values are very sensitive to changes in molecular mass of the polymer and therefore sensitive to chemical crosslinking reaction [14].

3.6 Thermal stability by TGA in inert and oxidative atmosphere

Figure 8 shows the weight change of EVA material as function of the temperature by TGA analysis in inert atmosphere (Argon gas) during PV module encapsulation process. It is not easy to correctly model the EVA degradation process due to the fact that the reaction is complicated and involves multiple steps.

There is no general way of defining the onset of degradation of a polymer material, but we can define the temperature in a scanning TGA experiment as the start of weight loss. In a TGA weight loss curve the degradation onset of the original and crosslinked EVA is 85°C and 51°C respectively.

At this temperature, the weight loss of the original and crosslinked EVA is 0.125% and 0.1806% respectively. Weight loss below 100°C is initial water content both in EVA. It demonstrated that crosslinked EVA is very stable and no substantial weight loss will occur at processing or operational cell temperature.

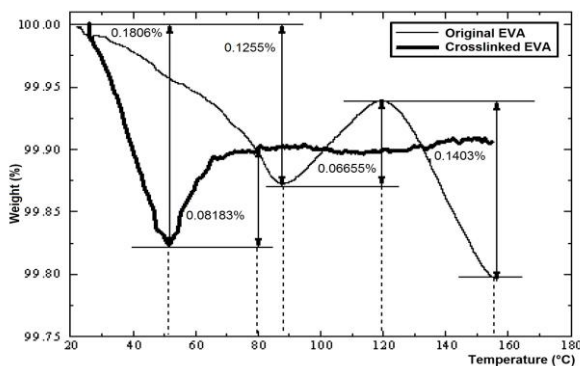


Fig. 8: TGA curves of EVA before and after crosslinking process

Also, figure 9 shows the TGA curves analysis in isothermal mode at 160°C, which was in the typical range of the crosslinking reaction process, and in oxidative atmosphere (oxygen gas). It appears clearly that holding EVA samples at temperature of 160°C for 5 hours; the mass loss weight is 1.5% and 0.05% for original and crosslinked EVA respectively.

This is the evidence that crosslinked EVA exhibits very high stability in oxidative atmosphere and under high temperature due to the crosslinking reaction and also the presence of anti-oxidant additive [15, 16].

3.7 Effect of crosslinking on the resistivity of EVA

As shown in figure 10, the resistivity decreases with increasing temperature i.e. the EVA has a negative resistivity temperature coefficient. The experimental measurement results show that crosslinked EVA exhibits higher resistivity than the original EVA.

This provides the high electrical insulation which is needed to guarantee circuit isolation from environmental damages.

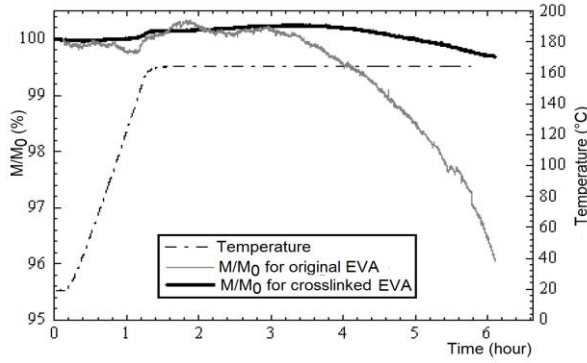


Fig. 9: TGA curves of EVA in isothermal mode before and after crosslinking process

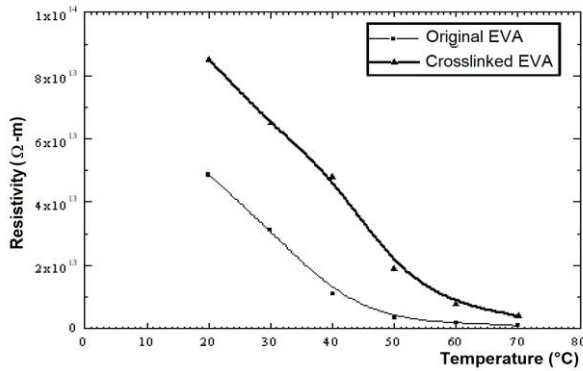


Fig. 10: Variation of the resistivity of EVA as function of temperature

The second approach is the evaluation of the activation energy of EVA during encapsulation process by using the Arrhenius method. Experimentally [17], the temperature dependence of resistivity is given by {Eq. (3)}:

$$\rho = \rho_{\infty} \times \exp \left(- E_a / R \times T \right) \quad (3)$$

Where, ρ and ρ_{∞} are the resistivity at temperature T and T_{∞} respectively, E_a (J/mol) is the activation energy and R is the Gas constant (8.314 J/mol.K).

A plot of $\text{Log } \rho$ vs. $1000/T$ will yield a straight line as seen in figure 11, where the activation energy W can be obtained from the curve slope ($-E_a / R$) according to {Eq. (4)}:

$$\text{Log } \rho = \frac{-E_a}{R} \frac{1}{T} + \ln \rho_{\infty} \quad (4)$$

The activation energy of original and crosslinked EVA is calculated to be 69.4 kJ/mol and 53.0 kJ/mol respectively by Arrhenius method, which is in good agreement with literature results [18].

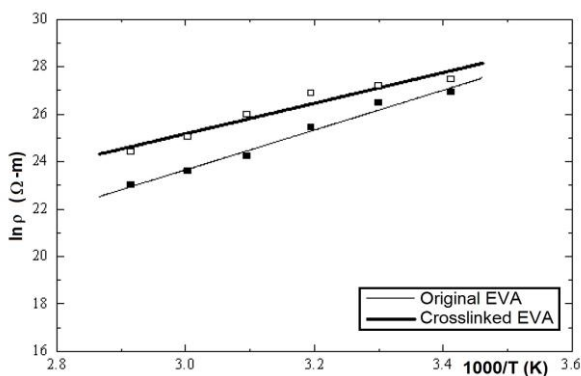


Fig. 11: Arrhenius curves of EVA before and after crosslinking process

4. CONCLUSION

Analysis of EVA material has indicated that there are two glass transitions that can be detected by TSDC. DSC analysis has further indicated the presence of endothermic and exothermic peaks, this is consistent the presence of two glass transitions. DMTA results indicate that a rather large mechanical loss process occurs at low temperature, and a second mechanical loss process occurs at high temperature. Also, the results of DMTA and tensile tests results are in good agreement, where the stability of crosslinked EVA is proved by TGA results in oxidative atmosphere (presence of oxygen). Since the thermal history affects, in general considerations, the properties of EVA, it is expected that the DSC curve reveals the structural change and may offer information of EVA re-crystallization during cooling step in lamination-crosslinking process. The exothermic peak around 160°C is due to the heat release from crosslinking reaction in EVA and the exothermic area relates to the percentage of uncrosslinked EVA. Therefore, the gel fraction of crosslinked EVA can be estimated by the area of exothermic region.

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