

Development of methods for testing the dimensional stability of splicing tapes

Degree project for Bachelor of Science in Chemical Engineering

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Abstract

SP Technical Research Institute of Sweden has in resent years observed trends of change in composition of splicing tape. This is a kind of tape consisting of a carrier made of polyethylene with reinforcement thread made of polyester added. These kinds of tape have been appearing more in recent years in the market. The general change in composition is drastic and one issue is how the ageing behaviour of the tape is influenced by the changes.

Manufacturers and companies turn to SP to get approval of certification on their products and SP-Method 1380 and SP-Method 5138 developed in the Department of Chemistry, Materials and Surfaces are used to test and evaluate the tapes in the process of certification. Since time is an issue, it is convenient to find a method that takes shorter time and that shows results that are relevant enough and that reflects the long time ageing effects of the splicing tape setup in a building.

The dimensional stability of splicing tapes, and especially the appearance of channel formations between the building film and splicing tape during accelerated ageing, has been studied in this thesis. Different types of splicing tapes were tested according to SP-method 5138. The aim was to study the reason why and when in the process of aging the channel formation is occurring in respect of the physical ageing. Construction films with different thickness were used in the study.

Dimension stability evaluations were done to each of the components of the test subjects that were tested according to SP-method 5138. This was done through accelerated aging in different timescales and temperature regions in this project. Theory behind physical aging of polymers was described because it applies to the methods used to evaluate the materials studied in this thesis.

A Fourier Transform Infrared Spectroscopy (FTIR) analysis was done on the materials to identify the polymeric materials in splicing tape and building film used in this thesis. Differential Scanning Calorimetry (DSC) was used for a thermal analysis of the materials to provide information about their thermal characterization. The aim was to find correlations with the effect of ageing process in elevated temperatures.

The results of the tests made in this thesis show that the rate of change in dimensions tends to slow down after two days, and the formation of channels occur already during the temperature increase in the beginning of the exposure used in SP method-5138.

Information gathered from this thesis can be used to try shortening the time of evaluating tapes according to SP-method 5138. A suggestion is to change the exposure time to two days instead of 10 days.

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1. Introduction

1.1 Background

SP Technical Research Institute of Sweden has in recent years observed trends of changes in composition of splicing tapes that are made for construction of buildings. The general composition of splicing tapes has often consisted of a carrier made of polyethylene (PE) combined with adhesive on one side. There are types that consist of a thin carrier with adhesive and polyester reinforcement thread added. The second mentioned type of splicing tape has been appearing more in recent years in the market. The composition gives them good properties at the moment of installation, they are easy to handle and work with and the adhesiveness is good. The general change in composition is drastic and one issue is how these changes affect the ageing behaviour of the tape during testing performed by SP.

Splicing tapes main purpose is to joint building films in constructions that keeps and protect the insulation material of buildings from air and moisture. For buildings that are made to be airtight, the aspect of having good materials around the area of where the building film joints are is important (Sandberg, P.I. & Sikander, E. 2004). Due to its effect on the overall airtightness in a building the performance of splicing tapes affects to the energy costs due to heat losses (Sandberg, P.I. & Sikander, E. 2004).

Manufactures and companies turn to SP to get approval of certification on their products and SP-Method 1380 and SP-Method-5138 developed in the Department of chemistry, Materials and Surfaces is used to test and evaluate the tapes in the process of certification.

- SP-Method 1380: Covers testing and performance requirements for jointing materials used as air and vapour barriers. The method relates to jointing stripes, jointing compound or tape. In respect of polymer materials used in building application a requirement is that fully acceptable performance must be maintained at least 50 years.
- SP-Method 5138: Is a method that evaluates the dimension stability of splicing tape. The maximum change in length required is 0,5%, furthermore no appearance of channel formation or tendencies that could lead to air and vapour leakage between the tape and underlying material. In uncertain cases the samples are to be evaluated according to EN-12730 were the airtightness is evaluated.

The accelerated ageing process of SP-Method 1380 take 24 weeks to complete, this part of the method is designed to simulate 50 years of aging in real life. Time is an issue both for SP and for the customers, it is therefore convenient to find a method that takes

shorter time and that shows results that are relevant enough and that reflects the long time ageing effects of the splicing tape setup in a building.

1.1.1 Clarification of the issue

The issue that has been studied in this thesis is the appearance of channels between the building film and splicing tape observed as a result after accelerated ageing tests performed according to SP-method 1380 and 5138 at SP. With focus on physical ageing effects and the exposure time in SP- method 5138, these are the questions intended to be answered:

- What is the underlying reason for this effect?
- At what temperature and when during the process of ageing does the channel building occur?
- Can the channel formation be correlated to thermal characteristics of the materials via Differential Scanning Calorimetry (DSC)?
- Is there any way of cancelling out the effect of the construction film on the samples?
- How do the temperatures of the accelerated ageing correlate to the temperature where the splicing tapes are in the building structures?
- Is there any way of shortening the time it takes to evaluate the splicing tape in the process of approval of certification based on the result of the tests performed in this thesis?

1.2 Purpose

Data and conclusions from this thesis is intended to serve as part of a process of upgrading existing SP-methods or development of a new method that takes shorter time to evaluate splicing tape in respect of dimension stability. The report handles the physical ageing of the splicing tape and the methods used in this study are based on parts of SP-methods 1380 and 5138.

1.3 Boundaries

Due to time limitations tests with ageing time of 24 weeks according to SP-method 1380 will not be performed. The thesis handles 3 different brands of splicing tapes and two kinds of building films with different thickness bought for this project. Due to general confidentiality products that are tested will be mentioned and related to as codes.

2. Theory

2.1 Ageing of Polymers

Ageing is a term used to the describe changes over time, in the area polymer science it is used to describe changes of the property of a material over a period of time. Strength and toughness, density, change in composition and reactivity are some properties that can be studied when ageing a polymer material. Depending on what the reasons are for changes in property the ageing of polymers can be categorized in two major sections Physical and Chemical (White 2006). Ageing of polymers independent on the chemical interactions of the surroundings is a study of physical ageing (White 2006). One way of observing the physical changes in polymer is to measure the variation in dimension (Klason et al. 2001, 56). Physical ageing occurs when a polymer material is in a non-equilibrium state. The arrangement of the molecules in the material makes them move towards an equilibrium state (White 2006, 1397). This process is called molecular relaxation and leads to change in volume of the material over a period of time (White 2006), this effect can be observed as shrinking of a material (Klason et al. 2001).

In the process of fabricating plastic materials, thermoplastics are often used because of its ability to obtain it's properties after being melted and cooled down. They are commonly used in extrusion, injection or blow moulding processes of (Klason et al. 2001). In such processes the temperature is elevated to obtain a hot melt that can be shaped in whatever form the product is intended to be. When shaped during a production process energy is added to the material which makes the molecules flow and form a non-equilibrium thermodynamic state. The molecules in this state strives towards going back to a thermodynamic equilibrium state through relaxation. When the material undergoes a fast cooling during fabrication process, the rate of molecules can't keep pace with the thermal equilibrium process, the rate of molecular relaxation goes to slow in the process of the change required to keep a thermodynamic state of equilibrium (White 2006). The molecules in the material that does not reach a state of thermodynamic equilibrium during the time of production will be kept in firm positions, built in tensions and an ongoing strive towards an equilibrium continues in form of changing dimensions in an extended period of time (White 2006).

However, a rate increasing parameter is temperature as long as it is kept below melting temperature. The effect on the process of reaching equilibrium becomes accelerated when the temperature is elevated, it can be observed through studying the rate of change in dimension and it is considered to be an accelerated ageing process (White 2006). The rate of change is dependent on the ageing temperature (T_a) and increasing the temperature will accelerate the process. A T_a that is far below the glass transition temperature (T_g) will keep the material far from the equilibrium state thereby keeping the thermodynamic driving forces large, but the kinetics in the process is dependent on the difference between T_a and T_g and will limit the rate of change (White 2006).

Furthermore, in semi crystalline polymers the ageing takes place both in the amorphous part and in the crystalline part shown in Figure 1. The physical ageing in the crystalline part undergoes a recrystallization process and in the amorphous part of the polymers the

ageing is similar to the process that takes place during a glass transition, a consolidation of molecules in space (Klason et al. 2001).



Figure 1. Illustration showing crystalline and amorphous regions.

2.1.2 Accelerated Ageing

Physical ageing process of a polymer can be accelerated by increasing the temperature keeping it below melting temperature (White 2006). The effect on the process of reaching equilibrium is that the rate of change in dimension is higher and the equilibrium state is changed. This way of ageing polymers is applied in the various test methods (Klason et al. 2001).

2.2 DSC

Differential Scanning Calorimetry (DSC) is a thermal analysis method used to measure the temperature and heat flow that is related to the transitions in a material as a function of time and temperature (Crompton 2013). The method provides both quantitative and qualitative information that has to do with physical and chemical changes correlating with the exothermic, endothermic processes and heat capacity. The energy absorbed and released by the sample is measured under a period of time under which the temperature of the sample is increased, decreased or held constant (isothermal). Applicate temperature programs in a DSC analysis use to involve all the variations of temperature changes (Crompton 2013). DSC compares the rate heat input with the rate of temperature rise in the samples. The method is based on the assumption that the samples are small enough to obtain thermal equilibrium almost instantly (Brown 1988).

The purpose with this method is to characterize the thermal behaviour through plotting the difference in enthalpy that occurs in the sample and compare the changes in some inert reference material (Crompton 2013). The recorded heat flow differential (W/g) on the Y-axis can be plotted against temperature (°C) and time on the X-axis as described in Figure 2 (Crompton 2013).



Figure 2. An illustration on where and how in a DSC curve different transitions in the material are and can look like in ideal analysing conditions.

Analysing the changes of heat flow during transitions between different states in the material of the sample the information provided by the curve plotted during an analysis can be used to determine e.g. the melting point (T_m), degree of crystallinity and T_g (Crompton 2013).

2.2.1 Degree of crystallinity and melting point.

During the melting process in a semi crystalline plastic material it is the crystalline part that undergoes a transition from solid state to liquid. As the heat flow in to (endothermic) and out of (exothermic) the material during transition between states, the degree of crystallinity can be calculated from the information obtained during the melting transition (Crompton 2013). The difference in energy that takes the crystalline part to undergo the transition, melting (endothermic) or crystallization (exothermic) is obtained by integrating. The area between the point temperature of initiation of transition (T1) to temperature where the conversion ends (T2) under (melting) or above (crystallization) a baseline between the points can be used to calculate by integration as shown in the Figure 3 (Crompton 2013).



Figure 3.DSC analysis of the degree of crystallinity by calculating the area under the baseline.

By dividing the measured (by integration) enthalpy of fusion (Δ H) during the transition of the sample with the theoretical enthalpy of fusion for a 100% crystalline sample (Δ H_{100%}) it is possible to calculate the degree of crystallinity in the plastic based on equation 1 and 2.

$$\Delta H = \int_{T_1}^{T_2} Cp \ dT \tag{1}$$

$$\frac{\Delta H_{sample}}{\Delta H_{100\% cristallinity}} = crystallinity of sample \%$$
(2)

Melting point is taken from the peak point of the melting/crystallization process as shown in the Figure 3.

2.2.2 Glass transition.

The glass transition state is the temperature region of which the amorphous part of a polymer undergoes a transition. Elevating the temperature through T_g in an amorphous material makes it go from a hard glassy state to a soft rubber like state (Crompton 2013). During the transition a relaxation of internal stress, many physical properties changes fast and it can be seen as a change in heat capacity (Crompton 2013). Analysing a DSC curve, the transfer will be seen as a drop from a baseline and recovery of another baseline, the change is taken from the difference between the baseline at

extrapolated midpoint between the T_g initiation and T_g ending half step heat capacity change ($\Delta Cp_{1/2}$) as shown in the Figure 4. This is the point on where the $T_{g 1/2}$ is taken and is considered as the T_g of the material (Crompton 2013).



Figure 4. *DSC analysis, determining* T_g

2.3 Structure of splicing tape

The general composition is as told in the introduction made up of a carrier consisting of low density polyethylene (LDPE), acrylic adhesive and in some cases reinforcement thread made of Polyethylene terephthalate (PET) as shown by the Figure 5 and 6.



Figure 5. Close profile on the cross section of splicing tape. Release liner is the part that gets pealed of at the moment of installation.



Figure 6. Illustration of the pattern the reinforcement thread is in. The adhesive on the sticky side covers reinforcement thread.

2.3.1 PE

Polyethylene is a semi-crystalline thermoplastic that is generally divided in two categories; low density polyethylene (LDPE) and high density polyethylene (HDPE) (Klason et al. 2001). Generally, the LDPE has long branched side chains that gives it more volume and limits the degree of crystallization in comparison to HDPE, that has more liner chains with fewer and shorter side chain branches which gives it a higher degree of crystallinity and a higher density thereby the classification high density PE presented in Table 1 (Klason et al. 2001).

Table 1

HDPE	T _g ≈	-120°C
	T _m ≈	115-140°C
LDPE	T _g ≈	120°C
	Tm≈	105-125°C



Figure 7. The chemical structure of the repeating unit in PE.

Due to its low T_g it has good low temperature characteristics, it is soft and expandable and suits well in for different kind of industrial extrusion moulding processes like blow moulding and injection moulding (Klason et al. 2001).

2.3.2 Polyesters

Polyethylene terephthalate (PET) and polybutylene terephthalate (PBT) are similar in characteristics, but the stiffness of PET is higher as compared to PBT (Klason et al. 2001).

Table 2

PBT	Tg≈	40°C
	T _m ≈	220-225°C
PET	Tg≈	70°C
	T _m ≈	265°C



Figure 8. The chemical structure of the repeating unit in PET.



Figure 9. The chemical structure of the repeating unit in PBT.

Both are thermoplastics and well suited for moulding processes, PBT crystalizes in lower temperatures and are easier to form in lower temperature than PET (Klason et al. 2001).

3. Method

3.1 Materials and standard procedure

Splicing tapes used in this thesis were named "A", "B", and "C" and the two types of construction films "a" and "b" as explained in the Table 3 and 4.

When the products were combined to make a joint they will be referred to as the Table 5 describes.

Table 3

Splicing tape type:	Reinforcement thread (Yes/No)
Α	Yes
В	Yes
С	No

Table 4

Construction film type:	Thickness(mm)
a	0.11
b	0.20

Table 5

Tape Film	Α	В	С
a	Aa	Ba	Ca
b	Ab	Bb	Cb

Threads from tape A were referred to as TR_A and from B as TR_B Carriers from tape A were referred to as Car_A, from B as Car_B and from C as Car_C. Tapes with reinforcement thread removed were marked with symbol "*", for example when tape A with the reinforcement thread removed were tested in combination with film b, it was referred to as Ab*.

Equipment and chemicals used for the tests and analysis:

- Five forced ventilated Oven (Memmert) units with racks that can hold an elevated temperature for the samples ±2°C.
- Calcium carbonate powder.
- Conditioning space with climate $23\pm2^{\circ}$ C and $50\pm5\%$ relative humidity(RH)
- 1000 and 500 mm ruler with 0,5mm accuracy.
- PE Film
- Stainless steel Plate.
- Web camera (Logitech HD 750p)
- 99,2 % Ethanol

- DSC: Melter toledo DSC 1 star system, gas controller; GC 100
- Scale: Melter Toledo AT20
- Sample Holder: Aluminium Light 20ul, Ref.holder: 21,95 mg
- Punch
- Sample holder press
- Trough: Inner diameter (ID) :195 mm, height: 20mm
- Glass disk
- Forceps
- Scalpel
- FTIR device: Nicolet 6700 FTIR
- Acetone

As standard procedure the prepared sample were put to a conditioning space with standard climate $23\pm2^{\circ}$ C and $50\pm5\%$ relative humidity (RH) in minimum 24 ± 1 hours before insertion to the oven. Samples were conditioned when taken out, measured and documented after 24 ± 1 hours. Visual examinations were done on all the samples after ageing.

Tests performed according to SP-method 5138 the ageing time was 10 days. The ovens were setup making a temperature ramp from room temperature up to $65\pm2^{\circ}$ C during a time range of 24 hours. Following 8 days the temperature was constant, followed by a ramp down to room temperature 24 hours before outtake.

The range between the measure points were taken and documented before insertion. The difference in range between the measuring points after ageing was calculated in percent relative to the length before ageing. Data was taken from the average value of the range between the measures points for respective material (tape and film). Observed channelling between tape and film was documented.

3.2 Dimension stability test of splicing tape

Three dimension stability tests where only the splicing tapes were evaluated, were performed and referred to as T1 (test one), T2 (test two) and T3 (test three).

3.2.1 Test 1 splicing tape (T1_tape)

Samples of the tapes A, B and C were cut to obtain a 335mm long piece. A line was drawn close to each of the two edges of the short side of the tape with a measure point on each line according to Figure 10.



Figure 10. Illustration of how tape sample was prepared in T1.

The release liners were pealed of and calcium carbonate powder (talc) was applied to the adhesive side of the tape. The adhesive tend to obtain a more fluent form in elevated temperatures and the use of talc prevented them to get stuck to the surface, making it easier to handle the samples.

Ovens were setup and running with the temperatures shown in Table 7 meant for specified samples minimum 24 ± 1 hours before insertion of the samples.

The range between the measure points was measured and documented with ± 0.5 mm accuracy were as the time and temperature were marked on to the samples before insertion into the oven. Samples were made according to Table 6. Each series was representing the amount of time the samples were to be ageing: 1, 2, 5 and 10 days.

1 Day		2 Days	5 Days		10 Day		
Temperature		Temperature		Temperature		Temperature	
[°C]	Tape	[°C]	Таре	[°C]	Таре	[°C]	Таре
45	A1	45	A1	45	A1	45	A1
65	A2	65	A2	65	A2	65	A2
90	A3	90	A3	90	A3	90	A3
45	B1	45	B1	45	B1	45	B1
65	B2	65	B2	65	B2	65	B2
90	B3	90	B3	90	B3	90	B3
45	C1	45	C1	45	C1	45	C1
65	C2	65	C2	65	C2	65	C2
90	C3	90	C3	90	C3	90	C3

Table 7

Oven	Temperature
	[°C]
1	45±2
2	65±2
3	90±2

A difference in length before insertion and after ageing was calculated and plotted in a graph with the difference in range in the x-axis and the ageing time in the y-axis.

3.2.2 Splicing tape test T2 and T3

Two series were prepared for T2: one and two days. In T3 the same amount of series were prepared as in T1. The reinforcement thread was pealed of from samples A and B before insertion to the oven in T2 and T3. Two sheets of PE film were placed beneath the samples.

Two measure points were drawn on each line close to the edges of the short side of the tape as shown in Figure 11.



Figure 11. Illustration of how tape sample was prepared in T2.

The average value of the range between the two measure points were used to calculate the difference before and after ageing. It was plotted in a graph with the difference in range in the y-axis and the ageing time in the x-axis.

3.3 Dimension stability test of construction film

Samples of film a and b were to be cut $300 \times 60 \pm 1$ mm with the 300mm long side 90° angled relative to the production direction of the building film. Lines and three measure points were drawn on the film as shown in Figure 12. The samples were then marked

with type (a and b), temperature code (1, 2 or 3). Each sample setup was triplicated and marked with x, y and z. The samples were then arranged according to series shown in Table 8. The series was repeated and marked with the representing time the samples were to be aged during 1, 2, 5 and 10 days.



Figure 12. Illustration of how tape sample was prepared in T3.

Table 8

a1	a2	a3	b1	b2	b3
х	x	х	х	х	х
у	у	у	у	у	у
z	z	z	z	z	z

Table 9

Oven	Temperature		
	setup [°C]		
1	45±2		
2	65±2		
3	90±2		

Each series contain 18 samples and were repeated four times to make it a total of 72 samples to prepare. The samples were aged on a stainless steel plate.

Ovens were setup and run with the temperatures according to Table 9, minimum 24 ± 1 hours before insertion of the samples. After conditioning, the samples were measured and documented. An average value of the length between the 3 measure points were used to plot a graph with the difference in length in y-axis and the ageing time in the x-axis.

3.4 SP-method 5138

The main part of this test was performed according to SP-method 5138. Two pieces of construction film were cut $75 \pm 1 \text{ cm x } 20 \pm 1 \text{ cm}$ and joint together. According to SP-method 1380 version 3 with tape $70 \pm 1 \text{ cm}$ applied on the overlap of the construction film as shown in the Figure 13.



Figure 13. A cross section on how a sample was prepared making a joint according to SP-method 1380 and 5138.

The joints were made 90° relative to the production direction of the construction film. Perpendicular to the joint two lines were drawn near the short side edges of the tape and four measure points were marked on the lines as shown on the Figure 14.



Figure 14. *From upside on how a sample was prepared making a joint according to SP-method 5138.*

3.4.1 Building film according to SP-method 5138

Samples of film a and b were cut $75 \pm 1 \text{ cm } \times 20 \pm 1 \text{ cm } 90^\circ$ relative to the direction of production. A line was drawn close to each of the two edges of the short side of the sample and four measure point were drawn on each line according to Figure 15.



Figure 15. *An illustration on how a sample of film was prepared according to SPmethod 5138.*

3.4.2 Splicing tape on building film without joint

Samples of film a and b were cut $75 \pm 1 \text{ cm } \times 20 \pm 1 \text{ cm } 90^\circ$ relative to the direction of production direction. Tape A, B and C $70 \pm 1 \text{ cm}$ long was applied on the surface of the film as shown in Figure 14. A line was drawn close to each of the two edges of the short side of the tape and four measure points were drawn on each line according to Figure 16.



Figure 16. A sample were the tape was applied on the film not covering a joint.

3.5 DSC

The DSC analyses were performed on a Mettler DSC 1 instrument (heat-flux DSC, Mettler Toledo) equipped with a gas controller and a sample robot. The temperature and heat-flow accuracy of the DSC are frequently checked using pure indium and zinc as reference materials. Small pieces of sample were punched out from each sample and cut to circular plates in order to obtain the specified mass (3,5-8 mg). The temperature program was based on ISO standard 11357-3 and included heating the samples to a temperature higher than T_m (in order to remove thermal history), then cooling the samples to a temperature lower than the crystallization temperature and a second heating where the T_m and crystallinity was measured. The crystallinity data was

calculated using an value of heat of fusion $\,(\Delta h_{100\%}\,)$ of 293 J/g for PE and 140 J/g for PET.

Tests were also done with cycles under the T_m in order to see if the results done according to standard still were the same

3.5.1 Sample preparation

In order to obtain samples of tape carrier free from adhesive, tape A, B and C were cut in 10 cm pieces and release liner pulled off. Each piece was put on the bottom of a trough with the adhesive side facing up. 99, 2% ethanol was poured in to a trough with the sample making sure the samples were covered with ethanol. Trough with tapes and ethanol was covered with a glass disc. After 24 hours the sample were taken out and the adhesive was pealed of from the carrier. Tapes were washed until there was no adhesive left on the carrier and left for dry.

Reinforcement threads were pealed of from tapes A and B and washed with 99, 2 % ethanol making sure the adhesive was washed away. When threads were dried, small samples enough to fit sample holders were done by cutting pieces and rolling them in shapes of small spheres.

3.6 FTIR

FTIR analysis can be used for identification of a polymeric material as different chemical bonds absorb energy at varying wave numbers.

Small slices of sample were analysed using a Nicolet 6700 FT-IR instrument (Thermo Electron Corporation) equipped with a micro-ATR. The analyses were performed by collecting at least two spectra (32 scans) from each sample at a resolution of 4 cm⁻¹.

The collected spectra were baseline substracted before they were matched to reference spectra. Background spectrum was done before analysis. The ATR-kristall on which the samples were analysed on was cleaned with acetone before and between analyses of different samples.

Reinforcement thread was pealed from the tape A and B and washed with 99,2 % ethanol making sure the adhesive was washed away. A small sphere shaped sample was done and put to the eye of the FTIR device.

3.7 Shear test

A shear test was performed on the samples a2, a3, b2 and b,3 film that were aged in 10 days (chapter 3.3). This test was made to see if there was a way of evaluating any effect

on the adhesiveness of the surface of the film after ageing. This was an attempt of doing it through comparing the aged film with non aged film.

Samples were prepared according to SP-method 1380 with the exception of not including any tape during he ageing process. Two pieces of film were joint together with splicing tape and cut to obtain samples with 20mm width(b). The distance between the clamps on the tensile tester were 100mm with the joint centred in between.

The shear(σ)[N/m] was calculated by dividing the average and the max force(F)[N] taken from the critical state demonstrated by Figure 17. with the width(b)[m] of the sample as shown in equation 3.



Figure 17. Demonstration of where data was taken from the shear test plot. Critical state is the area of where the adhesive was breaching between the two pieces of film and the tape.

4. Results

4.1 Splicing tape

4.1.1 T1



Figure 18. Tape A change in range between measure points relative to the initial range (x_a-x_0) at 45, 65 and 90°C.

Tape A shows a clear tendency in shrinking in different temperature, the rate of change was high between days 1 and 2. After day 2 it does not shrink any more and begins to expand but in a slower rate according to Figure 18.



Figure 19. Tape B change in range between measure points relative to the initial range (x_a-x_0) at 45, 65 and 90°C.

Tape B does not show a clear tendency (Figure 19), the samples took a wavy like shape that made it hard to measure and obtain accurate data.



Figure 20. Tape C change in range between measure points relative to the initial range (x_a-x_0) at 45, 65 and 90°C.

There was a clear tendency in tape C (Figure 20), the rate of change slows down after two days at 90°C and after one day at 45 and 65 °C. Tape C show most shrinkage compared to Tape A and B(Figure 18 and 19). Measurement on sample tape C at 45°C day 10 was not able to be done because it got stuck to the oven. In the process of trying to retrieve it, it got destroyed.

The problems encountered with wavy samples were solved in T2 by removing the reinforcement threads from tape A and tape B before ageing.

4.1.2 T2

In test T2 samples A and B the reinforcement threads were removed making it easier to measure and obtain more accurate data. This test was performed in the time range of two days, as test T1 showed that most shrinkage occurred during the first two days.



Figure 21. Tape A change in range between measure points relative to the initial range (x_a-x_0) at 45, 65 and 90°C.



Figure 22. Tape B change in range between measure points relative to the initial range (x_a-x_0) at 45, 65 and 90°C.



Figure 23. Tape C change in range between measure points relative to the initial range (x_a-x_0) at 45, 65 and 90°C.

Samples were easier to measure at the two measure points used in this test. Tape B showed shrinking effects at 45 and 65°C as opposed to the effect at 90°C where the tape shows effect of expansion shown in Figures 21-23.

4.1.3 T3

In interest of obtaining more data regarding tape A and B a third test was performed on them. As in T2 the reinforcement threads were pealed of and this time samples were exposed during 1, 2, 5 and 10 days as in the first test.



Figure 24. Tape A change in range between measure points relative to the initial range (x_a-x_0) at 45, 65 and 90°C.



Figure 25. *Tape B change in range between measure points relative to the initial range* (x_a-x_0) at 45, 65 and 90°C.

The results from Tape A samples were consistent in showing about the same result as in T1 and T2 (Figure 18 and 21). Tape B Shows different behaviour at different temperatures (Figure 25), it shrinks at 45°C, the difference was almost 0 at 65°C and at 90°C it expands the first day and after peaking it shrinks in a lower rate.

4.2 Construction film

4.2.1 Film A



Figure 26. Film A, average change in range between measure points relative to the initial range (x_a-x_0) at 45, 65 and 90°C.



Figure 27. *Film A, change in range between measure points relative to the initial range* (x_a-x_0) at 45°*C*. Sample *x, y and z plotted with the average line shown as dashed line.*



Figure 28. *Film A, change in range between measure points relative to the initial range* (x_a-x_0) at 65°*C*. Sample *x, y and z plotted with the average line shown as dashed line.*



Figure 29. *Film A, change in range between measure points relative to the initial range* (x_a-x_0) at 90°*C*. Sample *x*, *y* and *z* plotted with the average line shown as dashed line.

The general effect was shrinkage when film A was aged at 45, 65 and 90° C. Judging by the average value (Figure 26) the film at 45 and 65°C follows a pattern. But when analysing the curves without considering the average of the triplicates, there was no tendencies except for the tape ageing at 45°C(Figure 27), although the average value was low the curves seems to follow the same pattern (Figure 28 and 29).

4.2.2 Film b

As opposed to film A (Figure 26-29) the effect of ageing film B was different, instead of displaying a shrink it shows expansion. Judging by the average values (Figure 30) the films follow a pattern were they tend to expand more at higher temperature. But when the average of triplicate was not considered (Figure 31,32 and 33) more data was obtained when aging the film at 65 and 90°C. The pattern of the curves in Figure 33, seemed to follow the same patter as the average curve.



Figure 30. *Film B, average change in range between measure points relative to the initial range* (x_a - x_0) at 45, 65 and 90°*C*.



Figure 31. *Film B, change in range between measure points relative to the initial range* (x_a-x_0) at $45^{\circ}C$. Sample x, y and z plotted with the average line shown as dashed line.



Figure 32. *Film B, change in range between measure points relative to the initial range* $(x_a - x_0)$ at 65°C. Sample x, y and z plotted with the average shown as dashed line.



Figure 33. *Film B, change in range between measure points relative to the initial range* (x_a-x_0) at 90°*C*. Sample *x*, *y* and *z* plotted with the average line shown as dashed line.

4.3 Splicing tape combined with construction film

4.3.1 SP-method 5138

As in the test T1(chapter 4.1) sample tapes with threads took a wavy shape making it hard to obtain accurate data. When doing visual evaluation, it was easy to see when a channel between construction film and tape was either partial or fully developed. Examples of channels can be seen in Figure 34 and 35. The materials in Figure 34 and 35 has not been used in this study.

As described in Table 10 the samples containing tape B were the ones that showed fully developed channels in first and second test. Same tape without thread did not show any fully developed channels, only partial in sample Ba* first test and Bb* that were aged without thread (Table 10). Table 11 shows samples that were aged in 45° C. Despite the lower temperature tape samples Ba and Bb still shows full developed channel, in contrast to the rest of the samples in Table 12 where there were no signs of partial or fully developed channel.

In order to capture the time of initiation of channel formation, sample Ba aged in 65° C was recorded. Analysing the movie produced with the pictures, initiation of channel formation was observed after 10,5 hours after insertion of samples. According to the recorded temperature logs of the ovens the temperature was $40\pm1^{\circ}$ C at initiation and $51\pm1^{\circ}$ C when it was fully developed.



Figure 34. An illustration on how initiated channelling between tape and construction film looks like. Keep in mind that the materials in the picture were not included in this study. The initiated channel formation is a set up for demonstrational purpose.



Figure 35. An illustration on how a fully developed channelling looks like. Keep in mind that the materials in the picture were not included in this study. The fully developed channelling is a set up for demonstrational purpose

SP-method 5138 (*)= without thread								
Tape/Film	Change(mm)		Change (%)		Average Change (%)		Channel(x)	
	Film	Таре	Film	Таре	Film	Таре	Initiated	Full
Aa								
1	-1,0	-9,9	-0,2	-1,5	-0,2	-1,5	х	
2	-1,5	-10,5	-0,2	-1,6			х	
1*	-0,5	-11,0	-0,1	-1,7	-0,1	-1,4		
2*	-0,5	-7,5	-0,1	-1,1				
<u>Ab</u>								
1	3,0	-7,5	0,5	-1,1	0,5	-1,1	х	
2	3,0	-6,5	0,5	-1,0			х	х
1*	-17,0	-29,0	-2,6	-4,4	-1,1	-2,7		
2*	2,0	-6,5	0,3	-1,0				
Ba								
1	-3,5	-9,5	-0,5	-1,4	0,0	-1,3	х	х
2	3,5	-8,0	0,5	-1,2			х	х
1*	-1,0	-12,5	-0,2	-1,9	-0,1	-2,0	х	
2*	0,0	-13,5	0,0	-2,0				
Bb								
1	3,0	-0,5	0,5	-0,1	0,5	-0,7	х	х
2	3,5	-8,0	0,5	-1,2			х	х
1*	3,1	-9,2	0,5	-1,4	0,5	-1,8		
2*	3,5	-14,5	0,5	-2,2			х	
<u>Ca</u>								
1	-1,5	-4,5	-0,2	-0,7	-0,2	-0,8		
2	-1,5	-5,5	-0,2	-0,8				
Cb								
1	1,5	-1,8	0,2	-0,3	0,3	-0,2		
2	2,0	-0,5	0,3	-0,1				

SP-method 5138(*) = without thread45°C								
Tape/Film	Change(mm)	Change (%)		Average Change (%)		Channel(x)	
	Film	Таре	Film	Таре	Film	Таре	Initiated	Full
Aa								
1	0,5	-6,5	0,1	-1,0	0,1	-0,9		
2	0,5	-5,5	0,1	-0,8				
Ab								
1	2,0	-7	0,3	-1,1	0,3	-1,1		
2	2,0	-8	0,3	-1,2				
Ba								
1	0,0	-8	0,0	-1,2	-0,1	-1,2	х	x
2	-1,0	-8	-0,2	-1,2			х	x
Bb								
1	3,0	-9	0,5	-1,4	0,4	-1,4	х	х
2	2,5	-9,5	0,4	-1,5			х	x
<u>Ca</u>								
1	-1,5	-3	-0,2	-0,5	0,0	-0,3		
2	1,5	-0,5	0,2	-0,1				
Cb								
1	0,5	-4	0,1	-0,6	-0,2	-0,8		
2	-3,5	-6	-0,5	-0,9				

4.3.2 Tape on air and vapour barrier

Table 12 and 13 represents samples with tapes applied on sheets of film without joint and sheets alone without tape. The test was performed at 45°C and 65°C. The initiated channelling was observed on samples Ba and Bb with full developed channels on Bb at 65°C. At 45°C only initiated channel formation was observed on samples Ba and Bb. Samples combined with tape without reinforcement thread did not show any sign of channelling.

Tape on air	and	(*)=	Without						
vapour barrier hread									
65°C									
Tape/Film	Change(mm)		Change (%)		Average Change (%)		Channel(x)		
	Film	Таре	Film	Таре	Film	Таре	Initiate d	Full	
Aa									
1	-1,5	-10,5	-0,2	-1,6	-0,2	-1,6			
1*	-0,5	-7	-0,1	-1,1	-0,2	-1,1			
2*	-2,5	-7,5	-0,4	-1,1					
Ab	Ab								
1	3,7	-8,5	0,6	-1,3	0,6	-1,3			
1*	1	-8,5	0,2	-1,3	0,3	-1,3			
2*	2,5	-8	0,4	-1,2					
<u>Ba</u>									
1	-2	-14,5	-0,3	-2,2	-0,3	-2,2	х		
1*	-0,5	-17,5	-0,1	-2,7	0,0	-2,3			
2*	0	-12,5	0,0	-1,9					
<u>Bb</u>									
1	4	-19,5	0,6	-3,0	0,6	-3,0	х	х	
1*	5	-16	0,8	-2,5	0,7	-2,4			
2*	3,5	-15,5	0,5	-2,4					
<u>Ca</u>						1			
1	-0,5	-5 <i>,</i> 5	-0,1	-0,8	-0,1	-0,8			
<u>Cb</u>						1			
1	1,5	-3	0,2	-0,5	0,2	-0,5			
<u>Film</u>	1								
<u>a</u>		1	1			r			
1	-1,5		-0,2		0,0				
2	1,5		0,2						
<u>b</u>			1						
1	0,5		0,1		-0,2				
2	-3,5		-0,5						

SP-method	<u>5138</u>	(*) = wit	hout hre	ad					
45°C									
Tape/Film	Change(mm)		Change (%)		Average Change (%)		Channel(x)		
	Film	Таре	Film	Таре	Film	Tape	Initiated	Full	
Aa									
1	0	-8	0,0	-1,2	0,1	-1,0			
Ab									
1	1,5	-7,5	0,2	-1,1	0,1	-1,0			
Ba									
1	-1,5	-16	-0,2	-2,4	0,1	-1,0	х		
Bb									
1	0,6	-17,4	0,1	-2,6	0,1	-1,0	х		
Cb									
1	-0,5	-9,5	-0,1	-1,4	0,1	-1,0			

Table 13

4.4 DSC results

Table 14 shows data were T_g and T_m was taken from second heat up. Crystallization temperature (T_c) was taken from temperature drop. When evaluating the DSC curves from samples made of tape carriers and films an appearance of a transition that looks much like a T_g occurs under the first heat up but disappears in the second heat up. As told in chapter 2, PE has a lower T_g than the temperature range used for the test (Figure 36-40). T_g values were therefor not obtained for tape carriers and films.

Material(Sample)	T _g [°C]	T _m [°C]	T _c [°C]	°Crystallinity[%]
Tape carrier				
Car_A	-	109	98,3	38,8
Car_B	-	110	106	38,4
Car_C	-	101	88	16,14
<u>Thread</u>				-
Thr_A	86	255	207	42
Thr_B	78	256	207	40
<u>Film</u>				
а	-	117	108	43,7
b	-	108	99	37



Figure 36. Sample of a carrier from tape A. Time and temperature on x-axis and specific conductivity on y-axis.



Figure 37. Sample of a carrier from tape *B*. Time and temperature on x-axis and specific conductivity on y-axis with a double peak at the area of melting transition.



Figure 38. Sample of a carrier from tape C. Time and temperature on x-axis and specific conductivity on y-axis with a double peak at the area of melting transition.



Figure 39. Sample of film a. Time and temperature on x-axis and specific conductivity on y-axis with a double peak at the area of melting transition.



Figure 40. Sample of film b. Time and temperature on x-axis and specific conductivity on y-axis with a double peak at the area of melting transition.



Figure 41. Sample of reinforcement thread pealed of from tape A. Time and temperature on x-axis and specific conductivity on y-axis.



Figure 42. Sample of reinforcement thread pealed of from tape B. Time and temperature on x-axis and specific conductivity on y-axis.

4.5 FTIR analysis of materials

The tape carriers and film spectra were matched with LDPE film spectrum and the threads were matched with PET spectrum as reference shown in Figures 43-50. The average match between sample spectrum and reference spectrum is presented in percentage by Table 15.

Sample	Reference	Match(%)
Car_A	LDPE	80
Car_B	LDPE	79
Car_C	LDPE	84
Film_a	LDPE	81
Film_b	LDPE	82
Thr_A	PET	94
Thr_b	PET	92



Figure 43. Sample of Tape A carrier matched with a LDPE film spectrum.





Figure 44. Sample of Tape B carrier matched with a LDPE film spectrum.



Figure 45. Sample of Tape C carrier matched with a LDPE film spectrum. Peaks at wave numbers 1737 cm^{-1} and 1238.4 cm^{-1} seem to be interference from adhesive traces left on the sample.



Figure 46. Spectrum from the adhesive of tape C, with wavenumbers 1737.1 and 1238.7 cm⁻¹ marked on the peaks that matches with the interference from the sample made of the carrier from tape C (Figure 38).



Figure 47. Sample of film a matched with a LDPE film spectrum.

Film b:



Figure 48. Sample of film b matched with a LDPE film spectrum.

Reinforcement thread from tape A:



Figure 49. Sample of reinforcement thread from tape A matched with a PET spectrum.

Reinforcement thread from tape B:



Figure 50. Sample of reinforcement thread from tape B matched with a PET spectrum.

4.6 Adhesiveness

Film a:



Figure 51. Shear test film a aged at 65 and 90 °C in 10 days, the max and average value compared to the shear value of non aged film.



Figure 52. Shear test film b aged at 65 and 90 °C in 10 days, the max and average value compared to the shear value of non aged film.

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Shear value decreased when film a and b were aged at 90°C (Figure 43 and 44). It was almost constant when aged at 65°C except for the average value of film b aged at 65°C decreases.

5. Discussion

One of the aims with this study was to use SP-method 5138 to try to induce channel formation between tape and film; this was made with SP-method 5138. The results from the test showed that samples Aa and Ab had initiated channel formation (Table 10). Ba and Bb had fully developed channels in both tests (Table 10). In an evaluation according SP-method 5138 tape Ab and Aa would be classified as an uncertain result and airtightness tests would have been performed. Ba and Bb would not pass due to the fully developed channel formation (Table 10). It is worth noticing that Ba and Bb would not have passed the test either in 45°C. Sample Ca and Cb both passed the test and did not show any sign of channel formation, even though tape C is the tape that showed most shrinking behaviour among the three tapes (Figure 20 and 23).

As said in chapter 3 modifications of the test were made in order to be able to obtain as accurate data as possible (T2 and T3). One of the modifications was to take away the reinforcements threads from the splicing tape before exposing it in test T2 and T3 (Chapter 4.1.2 and chapter 4.1.3), as tape B took a wavy like shape when aged with threads making it impossible to straighten out and get accurate data.

• What is the underlying reason for this effect?

When sample A and B was prepared without reinforcement threads the results look better for both. Sample Aa* and Ab* passed the test and sample Ba* and Bb* did not show any fully developed channel formation (Table 10). There was clearly an effect in removing the threads and it indicates that they have a big role in the process of channel formation during exposures at elevated temperatures. It was hard to find out whether the thread had shrunk or expanded due to the way it curled up and got entangled during ageing. One suspicion was that the polyester of the threads was of the type PBT with a T_g at 40°C region (Klason et al. 2001), and if that had been the case the threads would have passed the glass transition temperature when increasing the temperature up to 65° C in a test. It seems that this is not the case, as the results seen in Table 14 showed that T_g is around 83°C, which is closer to a PET material (Klason et al. 2001).

The shrinkage and the expansion behaviour of tape B tested in T2 and T3 is worth noticing. As tape A and C showed a pattern where the rate of change were highest at 90°C and decreased at 65°C and 45°C, tape B showed most shrinking behaviour at 45°C, almost no shrinking at 65°C and expansion in 90°C. This behaviour of tape B sets it apart from the other tapes in T1, T2 and T3 (chapter 4.1).

The reason for the effect seems to be a combination of the behaviour of the carrier and the threads applied to it. All the tapes undergo a certain amount of change in dimensions during ageing. Considering the good results of tape 3 that shrunk the most in T1, T2 and

T3 but not forming any channels, it seems not to be the amount of change in dimension of the tapes it selves that determines if there is going to be a channel formation effect.

More tests on other splicing tapes containing threads should be done to gather more data from the effect of the reinforcement threads. Products can vary between production batches, which is reason of suggestion repeating the tests for different batches.

- At what temperature and when during the process of ageing does the channelling occur?
- Can the appearance of channels be correlated to the behaviour of thermal characterization of the materials via DSC?

Time lapse recording of tests according to SP-method 5138 were made to capture the time of when channel formation occurs. According to the ovens temperature this occurred at temperature between $40\pm1^{\circ}$ C at initiation and $51\pm1^{\circ}$ C when it was fully developed. This was also confirmed with visual observation through the window of the oven after the time lapse was done and gave good indications of when to go and observe the samples.

When evaluating the DSC curves in chapter 4.4 something that looks like a glass transition is found at approximately $40 \pm 3^{\circ}$ C. It looks like some kind of transition occurs in that temperature region and it seems to correlate to the temperature where channel formation initiation occurs. What the carrier and the building film have in common is that they tend to be made of PE and additives(Figure 43-48) and T_g for PE is lower than -100° C(Table 1).

There was a double melting peak on sample car_B indicating that it could be a copolymer. Experiments has been done by Fonseca, C.A. and Harrison, I.R on LDPE/HDPE blends were double melting peaks was shown on DSC curve (Fonseca and Harrison, 1998). Tape B is the one that showed different behaviour in the dimension stability test and did not pass the test according to SP 5138. This needs to be investigated more in order to know what component is causing the second peak in the DSC curve, and thereby maybe find out if it can cause this correlation.

- How does the ageing in elevated temperature affect the adhesiveness on surface of the building films?
- Is there any way of cancelling out the ageing effect of the construction film on the samples?

The shear test showed that the shear value is affected more on the film samples that were aged at 90° C than the samples that were aged at 65° C. Even though there were changes in the shear test between aged and unaged samples, they were small. There is doubt if the adhesiveness of the surface on the construction film is significantly affected by ageing. Inducing the dimensional change of the film by an exposure at an elevated

temperature for a certain time before the film is used in SP-method 5138, could therefore be part of a procedure when testing splicing tape.

• Is there any way of shortening the time it takes to evaluate the splicing tape in the process of approval of certification based on the result of the tests performed in this thesis?

The results of the tests made in this thesis shows that the rate of change in dimensions tends to slow down after two days, and the formation of channels occur already during the temperature increase in the beginning of the exposure used in SP method-5138. This indicates that the exposure time of the test method can be shortened drastically in respect of the properties studied in this thesis.

6. Conclusion

Information gathered from this thesis can be used to try shortening the time of evaluating tapes according to SP-method 5138. A suggestion is to change the exposure time to two days instead of 10 days.

Overall the underlying reason for channel formation effect was not determined in this thesis but good indications on what future works could concentrate on in the thrive of finding the answer has been done.

7. Future work

- A dimension stability tests to the threads alone should be considered, to see the behaviour in elevated temperatures, due to limited amount of time and limited oven space it was not done in this thesis.
- More DSC evaluations on tape carriers and film analysing the temperature region of 40°C where something that looks like a T_g is observed.
- Investigation on the occurrence of melting double peak on DSC curve with sample Car_B.
- To cancel out the change in dimensions of building films when evaluating splicing tape. A suggestion is to heat treat the film at 65°C before using it in sample preparation. Such a test should be done.

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