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Impact of moisture uptake in uncured bond lines on joints performance

Master's thesis in Materials Engineering

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Cover: Structural adhesive applied on a component.

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Abstract

Adhesive bonding is a joining technique use to bond two surfaces together by using solidification or hardening that is non-metallic. For example, adhesive joining is mainly used in bonding sheet metal together in the car body in the automotive industry. The advantage of adhesive bonding is that it offers excellent flexibility and toughness. In addition, the risk for crack propagation can be minimized with adhesive bonding due to distributing the stress force over a larger area and reducing the stress concentration on the spotweld. However, moisture and elevated temperature significantly influence adhesive joints' performance. Most research has investigated how uptake moisture in cured bond lines influences the final properties. At Volvo cars, it is essential to understand how the transportation chain between supplier which is bonding the frame together and send it to Volvo cars for curing and assembly influences the mechanical performance of the joint performance and see if the adhesives follow Volvo cars standards before implantation in the production chain.

For this thesis, two adhesives are tested, steel-to-steel adhesive and multi-material adhesive, and the following climate are conditions are 25°C/80%RH, 30°C/80%RH, 35°C/90%RH and 40°C/80%RH. The uncured bond lines will be exposed to different humidity and temperature for age-time between 0 to 62 days before being cured and tested. Indication of influences has been seen and increasing the ageing time shows a loss in mechanical properties for both investigated adhesives. Multi-material adhesive for lap shear and T-peel showed a more significant average loss than steel-to-steel adhesive. Also, it has been identified that the number of cycles for failure decreases with age-time for both adhesives. Although today's Volvo Cars standard regarding exposure time for uncured adhesive can be applicable for steel-to-steel adhesive, the multi-material adhesive showed more significant losses and exceeded the acceptable loose limit.

Keywords: bonding, adhesive, moisture, temperature, car-body, humidity, temperature

Contents

List of terms

Terms	Definition
Adhesive joining	Joining method by using adhesive as a bonding between the component.
Adherend	One of the bodies held to another by adhesive.
Cohesive failure	Type of failure in a bond, in cohesive failure is a failure when the bond breaks. Basically, it is still adhesive left in both adherend/substrate.
Adhesive failure	Type of failure occur due to lack bonding between the adhesive and adherend. One adherend ends up it the adhesive and the other one without adhesive.
Surface near cohesive	Mixer of cohesive and adhesive failure. When having little bit of adhesive on the surface left after failure.
Body in white (BIW)	It is a stage in the automotive industry where the car's frame is joined.
Closed bond	Basically, when you have two adherend and adhesive between them. Then the bond is closed.
Open bond	Having one adherend with applied adhesive.
Plasticization	Refers to changes in the thermal and mechanical properties of the polymer.
T-peel	Test specimen for testing the peeling strength.
Lap-shear	Test specimen for testing the shear strength between two components.
VCS 1029, 5477	Test method standard for testing structural adhesive
VCS 5601,69	Volvo cars’s standard for testing fatigue testing of weld-bonded joints.
ISO 11399	Standard for testing t-peel test for flexible-to-flexible bonded

1 Introduction

The chapter contains short information about the project's aim, purpose, a small introduction to Volvo Cars and the use of adhesive joints.

1.1 Background

Volvo Cars is a multinational vehicle manufacturer, having production plants in Sweden, Belgium, Malaysia, China, and the USA. First Volvo cars were rolled out from the factory on 14 April 1927 in Gothenburg. Volvo Cars are known as one of the safest cars. In 1959, Volvo engineers introduced the three-point safety seatbelt, which until today is a significant role in the whole automotive industry and is estimated to have saved over one million lives. Their ambition for 2030 is to turn fully electric and only sell electric cars, and to reduce the emission per vehicle by 40% between 2018 and 2025

Body in white (BIW) is the stage in the production chain where the car body frame is joined together before the painting and interior assembly. The stage of BIW involves different joining techniques such as welding, riveting, clinching, adhesive bonding and laser brazing. The automotive industry is constantly seeking to produce cars with low consumption, and the vehicle's mass has a significant impact on efficiency**.** Decreasing the mass in the body frame can reduce weight and lead to a significant loss in performance. Back in the day, most metals in the car body frame were steel. Today, different materials can be found. Structural adhesives and weld-through sealants are used in combination with mechanical joining or spot welding in the BIW, giving possibilities of optimizing the body frame and joining dissimilar materials, which can lead to minimized weight and excellent performance.

According to Volvo standards all adhesive or sealant parts shall be spot welded as soon as possible to minimize the risk of moisture uptake. Adhesive can be uncured in an environment of 25°C/60% humidity for up to 31 days unless it is a closed bond. For open bond lines, the maximum open time is 72 hours (3 days) before curing the adhesive. For slightly higher temperatures 30°C and 80% humidity, an open bonding must be cured within 8 hours otherwise requires rework. The bond can be stored for up to 14 days in a closed environment. A maximum reduction of 20% in the mechanical performance of adhesive joints due to moisture uptake in bond lines is allowed.

1.2 Problem formulations

The car manufacturing process consists of many steps, and a delay or production stop has a significant impact on the production chain. In BIW manufacturing, parts using adhesive as joining requires a curing process to achieve expected performance. Due to vacation or production stops, the adhesive might be exposed to climate conditions for longer time influencing the adhesive joint's performance. Transportation between different sites such as supplier bonding and joining all the metal sheet together before sending into the production plant for curing and assembly has an impact on the joint’s final properties. Transport through a trailer or container in a closed environment with its own temperature and humidity, makes it is necessary to understand how much impact this production chain has on the adhesive joint's performance. Few suggestions have also come internally regarding the possibility of having open bond lines in the transportation chain, which gives opportunities to fit more components in the transport. Therefore, it is necessary to understand how the adhesive joints will behave due to moisture uptake in different humidity and temperatures.

1.3 Aim

This project aims to investigate two different adhesives used in the BIW and see how the impact of moisture uptake on uncured bond lines joints influences the joint's mechanical performance. In addition, investigate how increased temperature and relative humidity influence adhesive's fatigue performance and failure mode of the adhesive.

1.4 Constrains of the thesis

Due to a confidential agreement with the supplier and Volvo Cars, the name of the adhesives will not be mentioned. However, for this project, two different adhesives will be investigated. Adhesive one is a multi-material structural heat curing adhesive that can join dissimilar materials. In contrast, the second adhesive is a one-component structural heat curing adhesive used for steel components. Both are crash-resistant structural adhesives used in the car-body structure ana epoxy-based resin.

1.5 Delimitation

Two students from different universities will carry out this master thesis project. The decision has been taken that each of us shall write one report each where the results consist of one adhesive. This report will consist of results from the second adhesive steel-to-steel adhesive, but further down in this results and discussion comparison with the multi-material adhesive will be made.

2 Theory

This chapter provides more profound information about adhesive joints, types of stress adhesive might exhibit, and how moisture and elevated temperature influence the mechanical performance of the adhesive.

2.1 Adhesive joining

The growing demand for efficient vehicles has resulted in manufacturers producing lighter materials in the car body to reduce the vehicle mass, such as high strength steel, magnesium alloys, aluminium alloys, and composite. However, the process of substituting one material for another is not simple. Decreasing mass weight can lead to a loss in strength and toughness, which are inconvenient. But it has been shown that it is possible to achieve at least equivalent strength and less weight by optimization by using different materials.
Adhesive joining is an essential bonding technology in various industries, including today's automotive industry. It can reduce cost, corrosion, fatigue, crack resistance, and good damping properties characteristics. Furthermore, it offers a significant advantage over traditional joinings, such as arc welding, since it does not distort the joined component and create any heat affected zone (HAZ). Also, adhesive joining improves joint stiffness compared to mechanical fasteners or spot-welds because it creates a continuous bond rather than a localized point contact. As a result, stress is distributed more uniformly over a broader region. Due to reducing stress concentration along the joining edge, adhesive joints provide better fatigue resistance and inherently high shear strength. In addition, a well-designed joint can absorb much energy and has good noise and vibration dampening qualities. (Barnes & Pashby, 2000)

Adhesive joining is becoming more popular because it combines high strength with a high degree of flexibility and toughness. These are the most critical parameters determining the adhesive joint's strength and are essential for carrying loads effectively throughout the joint. The ductility of the adhesive allows the adhesive layer to absorb peak stresses at the overlap ends, preventing failure (Lucas F, Mariana D, Raul, & Ricardo, 2017). Different materials and joining geometries, on the other hand, cause different stress states in the adhesive layer, and the performance of a particular adhesive is inextricably linked to these factors. Adherend material is another factor which determines the performance of adhesively bonded joints, and its impact should be considered while designing adhesive joints. Although, increasing the adherend stiffness has shown to improve the strength, also overlap length affects the joint strength, the effect varies depending on the adhesive type, if it is ductile or brittle. It was discovered that, unlike in brittle adhesive joints, the strength of ductile joints improves practically proportionally with increasing overlap length (Lucas F. M, Mariana D, Tiago A.B, & Raul D.S.G, 2015)

Today, hybrid joints with adhesive bonding with spot-welding, self-piercing riveting or clinching are the main approaches for structural joints in car bodies. The use of adhesive started decades ago, especially for bonding windscreens for their design purpose and was also used to fill gaps in spot-welded flanges to prevent corrosion in the materials. Later, engineers realized that improving corrosion resistance with adhesive had unintended consequences of stiffening the automobile body, leading to using high modulus adhesives to improve the car's torsional stiffness. New adhesives with high strength and high energy absorption in collision situations were developed in the mid-1990s. The first purpose of the first structural adhesive bond was to enhance the car body's stiffness. Nowadays, adhesive joining has become a real structural part of a car, even in crash situations. Using a more lightweight design by allowing different materials has increased the usage of adhesive as a joining technology.

2.2 Stresses in adhesive joints

Adhesive joints might experience different stresses during service as in figure 1, which affects the adhesive layer differently. Therefore, for a successful design of joints, it is critical to understand different types of stresses in the joints.

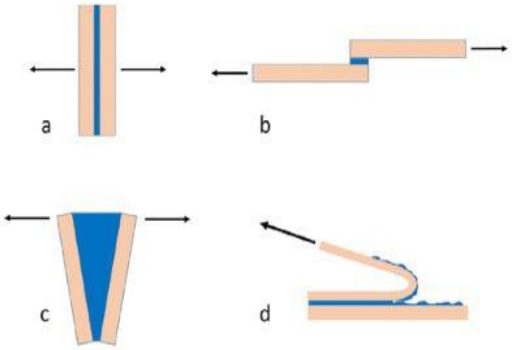


Figure 1: Illustration of different stresses an adhesive can experiences during services a) tensile stress, b) shear stresses, c) peel stress and d) cleavage. (Adams, 2021)

Tensile stress is produced when uniform tension is applied to the joint. Shear stress is generated when the joints are subjected to in-plane shear load. When the substrates are flexible, the adhesive layer is subjected to peel stress. Normal stresses are produced in the adhesive layer in both peel and tensile stress, but the fundamental difference is the shape of the stress distribution along the bond. Peel stress is not evenly distributed along with the adhesive layer, while tensile stress is evenly distributed across both bond lines. Finally, cleavage is caused by a bending moment, which is often created in joints with thick substrates. This loading condition causes nonuniform stress distribution throughout the bond line. A significant stress concentration is formed near the edge of the overlap, like the mechanism that generates peel stresses (Adams, 2021)

Joints will exhibit different forms of mechanical loads, such as forces and moments in service, leading to complex stress conditions. In understanding the behaviour of the adhesive joints, it is necessary to understand the mechanical load the adhesive is subjected to. If the load is constant, a quasistatic or creep analysis can be required to simulate the joints' behaviour. However, the main cause of failure is fatigue loads are applied for a brief period or cyclic. Therefore, adhesive properties as a function of strain rate should be appropriately studied to examine the stress distribution due to the impact load (Adams, 2021). Stress analysis of adhesives subjected to fatigue loads are challenging because the rate of degradation of adhesive properties due to fatigue must also be taken into account. Regardless of the loading condition, the shear and peel stresses are usually essential stress parameters when considering the strength and toughness of the adhesive joints (Carbas, o.a., 2019).

Swelling stress is caused due to the adhesive being exposed to a wet environment, causing water to absorb as free water and bound water, and this can weaken the adhesive joints. Swelling stress does not change the volume of the adhesive but can later in the process, cost the adhesive to swell. The swelling process is a prolonged phenomenon, and in most closed joints, the central point of the bonded are never experiencing saturation condition. It is more likely that the swelling is higher at the edge of the bonded area (Debruyne, Fevery, Hallez, & Vandepitte, 2020)**,** (Adams, 2021)

Thermal stress is another source that can lead to stress in the adhesive joints, which is mainly caused by the differences between the coefficient of thermal expansion (CTEs) of the substrates and adhesive. Usually, adhesives have higher CTEs than substrates and will expand more at elevated temperatures. The post-curing process of the adhesives has a significant impact on the residual thermal stress locked into the adhesive layer. Using stiff adhesive as a joint in metal or composite material tends to increase the thermal stress due to differences in the CTEs (Adams, 2021)**.**

2.3 Mechanism of adhesion

Establishing intrinsic adhesion forces across the interface is crucial to achieving good bonding. Since the quality of the interface between the solids has a considerable impact on the ultimate performance of the bonded materials. However, some factors determine how a polymer adheres to a specific surface and the most important material properties of the adhesive are viscoelasticity, molar mass distribution, and glass transition temperature. Other external factors which have a significant influence are temperature, humidity, surface roughness and the surface free energy of the substrates. (Baldan, 2012)

Mechanical interlocking is one of the adhesions mechanisms and occurs as the adhesive flows into the pores. Crack propagation at the interface is physically hampered by the interlocking. It also increases surface area, resulting in more total contact between the adhesive and the substrate**.** A study conducted that for strong adhesion, good wettability is not enough. The adhesive must also have suitable rheological properties to penetrate the substrate's pores. Having low adhesive viscosity promotes greater interfacial strength by allowing it to penetrate microvoids and pores wholly and quickly. Wettability is essential for providing good contact between the adhesive and adherend. Epoxy wets better in steels and gives good bonding than PE, PP, and PTFE. According to the adhesion theory, adhesion is formed by the interdiffusion of molecules between the adhesive and the adherend. The chemical bonding theory is the most well-known and oldest of all bonding theories and generated across the adhesive–substrate contact can considerably improve the level of adhesion between two similar or dissimilar materials. (Baldan, 2012)

A chemical bond is created by a chemical grouping on the adhesive surface and a chemical group in the substrate. The strength of the bond is determined by the number and types of bonds, such as a covalent bond or van der waals bond. Most of the industries use pretreatment/surface-treatment for their materials, producing different chemical compositions and oxide onto the surface. These morphological changes influence the nature of the chemical bonds. As mentioned before the strength of the adhesive joint is heavily dependent on the adhesion between the adhesive and substrate, which has led to the use of adhesion promotor molecules in the adhesive, typically called coupling agents which improve the joint strength between the adhesive and adherend. The coupling agents can react chemically on both sides of the interface, with the substrate on one side and the adhesive on the other, forming a chemical bridge. Almost all surface treatment affects the roughness of the surface in some way. For example, treatments such as abrasion and etching remove weak boundary layers and improve the wettability and reactivity of the surface. The adhesive must come into close contact with the adhered surface for good bonding. Surface roughness can obstruct adhesive spreading by stopping the glue from entering the adherend or causing it to gel before penetrating completely. (Baldan, 2012)

A well design adhesive bond can withstand and absorption a lot of energy, by due to the lacking in the bonding it might lead to unexpected failure and failure mode. The ideal mode of failure is 100% cohesive failure in the adhesive layer meaning that is it still adhesive remained in both surface after failure. On the other hand, adhesion failure is unwanted failure indicate lack of bonding between the adhesive and adherend. However, Failure mode should not be regarded as the primary criterion for determining whether or not a joint is beneficial. Some combination of adhesive and adherend might fail adhesively but exhibit superior strength than a similar joint having cohesive failure. The ultimate strength of a joints is important criterion compared to joints failure, but analyzing the failure can determining whether the failure was caused by a weak boundary layer or incorrect surface preparation (Ebnesajjad, 2014) (Landrock & Ebnesajjad, 2015)

2.4 Elevated temperature and moisture impact on bond lines

The main objective of both adhesives is to improve the stiffness, crash durability and fatigue performance. In comparison to typical metal joining techniques, adhesives significantly increase energy absorption during crash events, making them an appropriate choice. However, both adhesives are epoxy-based resin and absorb moisture, which can influence the adhesive's mechanical performance. Moisture uptake in a joint can occurs before curing, during manufacturing/assembly and after curing. Most researchers have studied the elevated temperature and moisture effect on cured adhesive bonded, and minor have been made on uncured bond lines.

The adhesive absorbs moisture as free water, and which occupies and fills the free spaces in the glue and causes plasticization. When water is absorbed, the water molecules tend to form single or multiple bonds with the adhesive's polymer chains, leading to swelling of the glue, plasticization, decrease in strength and glass transition temperature. However, if the water uptake is done at low temperatures, the adhesive's mechanical qualities are usually regained when cured. High temperature increases moisture uptake and has unavoidable degradation of adhesive properties, to short exposure times, the performance of bond tends to improve due to post-cure effects. However, over some time, its properties begin to deteriorate. Water and humidity are the enemies of adhesion since the water molecules usually bond to the same site as the adherend leading to poor adhesion. (C.P & S, 2004)

In an Investigation it was indicated that two different adhesive bonding indicates that they react differently by exposing them to an adverse environment. Some adhesive bonding tends to improve their bonding properties when increasing temperature and moisture due to the increasing cross-linking process of polymer chains. However, when the post-curing process is completed, the temperature and moisture negatively impact the performance of the glue. Other adhesive bonding might not exhibit a post-curing process, meaning that elevated temperature and moisture negatively degrade much earlier. (Lic, Lin, Lu, Wang, & Wang, 2011)

Majority of the bonded component in a car frame will be exposed to moist air and elevated temperature, and exposure to higher relative humidity and temperature for a long time has a certain influence on the strength of the joints. Research by Luo and Wong (2004) indicates that moisture can be absorbed by uncured epoxy and thus can affect the curing properties and the thermomechanical properties of the cured materials. For example, absorbed moisture can cause decreases in the glass-transition temperature, and the adhesion strength decreases dramatically when the samples are exposed to moisture before curing.

Another study conducted by Li, Lin, Lu, Wang L and Wang P (2011) investigating the moisture content influence on uncured adhesive bonding showed a reduction in static strength when increasing the moisture content. Most of the strength loss occurs rapidly initially, and as ageing continues, the bonding strength tends to level off. The same study indicated mixer of cohesive and adhesive failure modes and an increase in relative humidity tends to shift the failure from cohesive failure to adhesive failure.

Adhesively bonded joints that have been a moist show more plastic, ductile behaviour and absorb more energy, but generally withstanding lower maximum stress and allow high strains before failure. While bonded joints contain less moisture usually give a higher failure load and absorb less energy. Some studies have shown that the failure is also load-dependent on parameters such as overlap’s length or the thickness of the adhesive layer, making it essential to keep these parameters constant throughout the project. (F M A & M D, 2010)

3 Method

Method sections describe all necessary steps to perform this project, from literature study to analyzing test results.

3.1 Literature Study

At the beginning of the project, a literature review was conducted to further understand the topic regarding elevated temperature and moisture impact on the bonding performance before curing, more specifically, the bonding between metal components. The literature review describes previous results and conclusions, and relevant articles are collected for further thesis progression. Following databases are used to collect information Chalmers library search, Science direct, Access engineering and Access science. A summary of the result from the literature review can be seen in the theory section.

3.2 Test preparation

Testing followed international standards and Volvo cars standards. For the T-peel test ISO 11399 was followed, which describes the determination of the T-peel strength of an adhesive by measuring the peeling force of a T-shaped bonded specimen of two flexible adherents.

Lap-shear adhesion testing and Fatigue testing of welded bond joints Volvo Cars internal standards VCS 1029,5477 and VCS 5601,69 were followed. Both standards describe a test method for testing the joints' adhesion and shear strength also a test method for analyzing the fatigue behavior of adhesive welded joints. In Lap shear, two overlapping surfaces are joined together by adhesive to form a test specimen like in figure 2. The joint was later subjected to a lap-shear test, which is assessed with respect to the shear strength and failure mode. Fatigue testing is carried out using a hydraulic loading frame mechanism. Testing equipment must feature a control system that allows for a constant force amplitude as the specimens' stiffness decreases. In addition, used equipment should be able to measure and accumulate the overall displacement of the specimen.

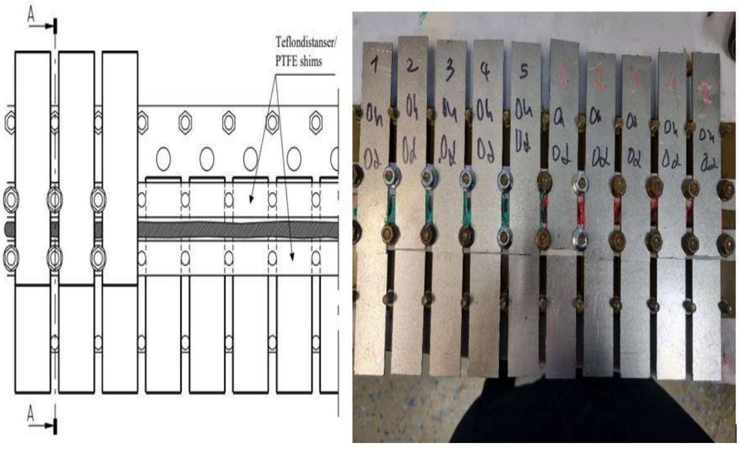


Figure 2: Illustration of how the lap samples are placed on a fixture suitable to hold the specimen together under curing, left picture is a in sketch while right image is how it looks like in practically.

DP600 is used as adherend material, a cold-rolled, uncoated dual-phase steel, and five test samples are made for each adhesive, condition, and ageing time. The width of the samples is 25mm, length 75mm and can be seen in figure 2. The workshop premakes the samples at Volvo Cars, and no surface treatment is applicable since the adhesive has to ability to absorb around 3g/m2 of oil during curing.

As lap shear, five samples for each adhesive, condition and ageing time are made for T-peel. The width of the samples is around 25mm and a length of 200mm. Adhesives were applied across the length of around 150m of total length and can be seen in figure 3. After curing, 50mm without adhesive was bent in the opposite direction, and force was applied along the bending direction.

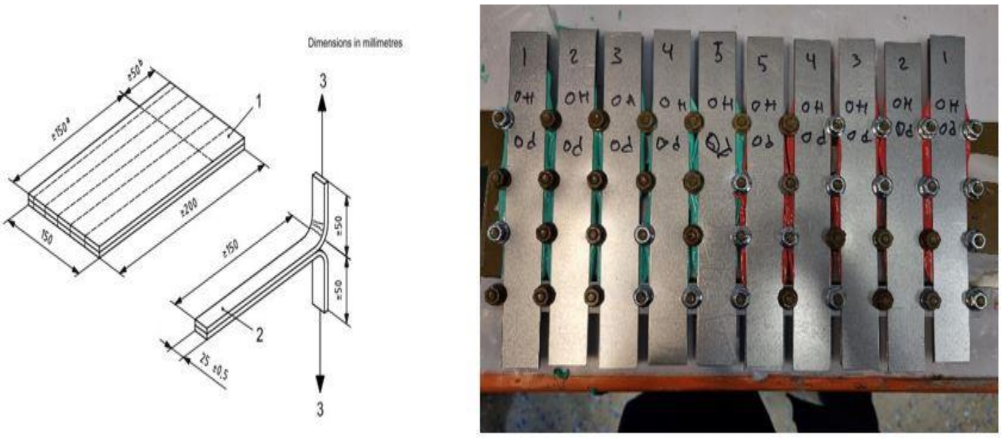


Figure 3: Illustration of the T-peel samples, left is a sketch while right picture is a real-life image.

Fatigue samples have two variations of specimens seen in figure 4, lap shear and T-peel and same preparation method are used for both specimens.

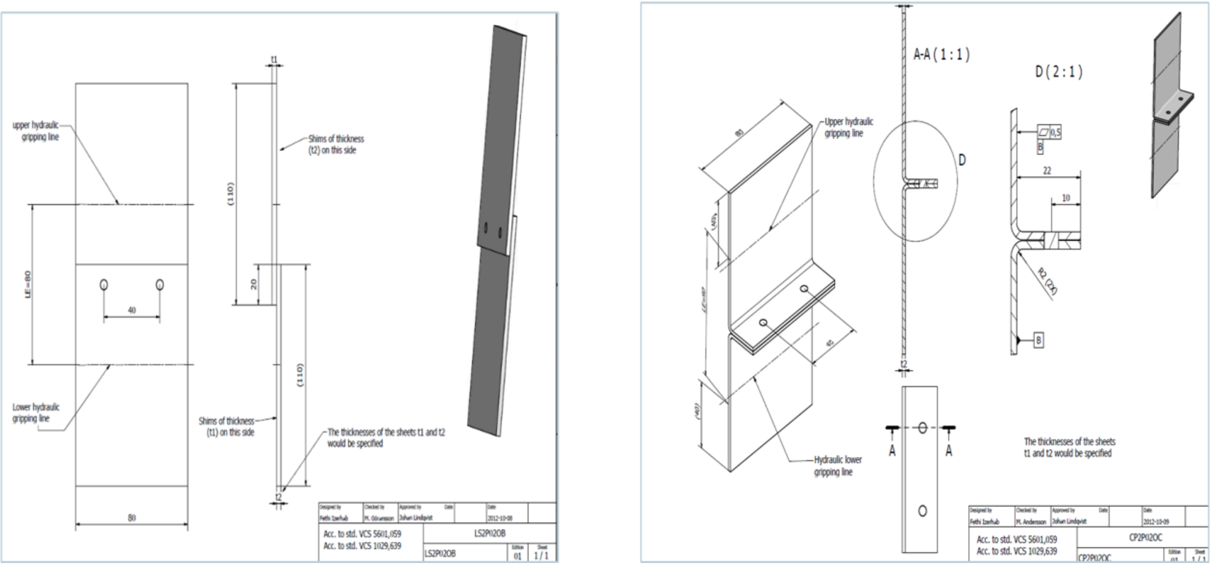


Figure 4: Drawing of how the lap-shear and t-peel are manufactured for fatigue testing.

For each adhesive, totally of 20 shear-test and 20 peel-test specimens were manufactured for respective ageing-time and climate. One of the adherents was placed in a fixture. Afterwards, adhesive was applied with a spatula or extrusion equipment to avoid air inclusions in the joints. The upper adherend was placed on the lower adherend and squeezed to ensure a good joint. It shall be slight overflow of material along the entire width of the joint. In the last step, the specimens were spot-welded.

3.3 Procedure of testing the samples

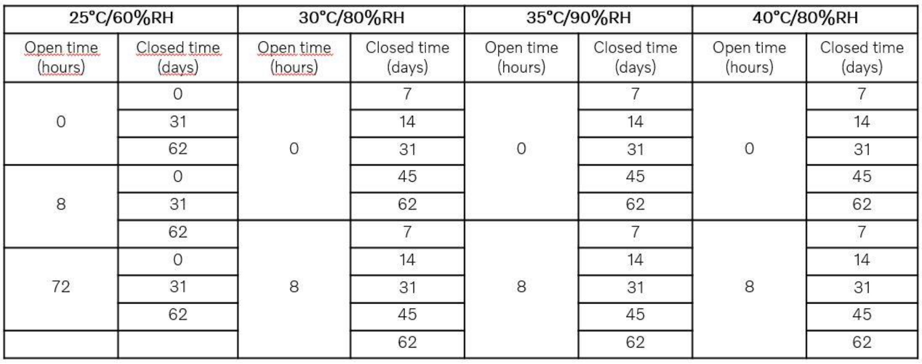
The tensile testing machine was capable of maintaining a predetermined constant crosshead rate and equipped with a suitable self-aligning grip to hold the specimen. Each grip shall firmly engage the outer 25mm of the unbonded specimen. The grip and the attachments were constructed to move in alignment with the specimen as soon as the force is applied. The test was performed at room temperature environment, tensile speed of the testing equipment was around $10 \pm 2\ mm/min$.

Fatigue samples were mounted in such a way that the shearing force of the joint is centered between the grips and the free length of the specimen, and grips shall be 80mm. Also, adjustable grips with the same material as the test samples was used during testing. Specimens were loaded with a sinuiosload with constant amplitude and constant frequency, and it is 15 HZ. Important that the testing occur in a room temperature climate. The testing was continued until it fails, or 200 000 cycles had been completed. To determine when a specimen fails, the stiffness was measured throughout the test, and it is defined as a load of range divided by the displacement range.

3.4 Testing of Climate condition

Four different in climate conditions in table 1 were investigated. Usually, the room temperature climate is around $23 \pm 2℃$ and 50 ± 5 % relative humidity. However, previous investigations on the production plant have shown that the climate there is around $25℃$ and 60 % humidity, and this climate condition can be seen as the room temperature. Open – time and close-time corresponds to the production, since in some scenarios the adherend is applied with adhesive and due to production stop, transportation, or off-days the adhesive is exposed to certain climate for longer time before bonding second adherend. With this investigation it is able to see what happens to mechanical properties of the adhesive due being exposed to open or closed bond.

Table 1: Describes the climate condition that will be tested and for how long the specimens will be aged.



25°C/60%RH		30°C/80%RH		35°C/90%RH		40°C/80%RH	
Open time (hours)	Closed time (days)	Open time (hours)	Closed time (days)	Open time (hours)	Closed time (days)	Open time (hours)	Closed time (days)
0	0	0	7	0	7	0	7
	31		14		14		14
	62		31		31		31
8	0		45		45		45
	31		62		62		62
	62	8	7	8	7	8	7
72	0		14		14		14
	31		31		31		31
	62		45		45		45
			62		62		62

In table 1, the different climate conditions have open-time from 0 hours to 72 hours. With open-time 0 hours, the specimen was closed as in figures 2-3 and placed directly into the climate condition. For open-time 8 to 72 the specimen was placed into the climate condition with one adherend applied with adhesive as in figure 5 for respective hours which before squeezing the adhesive with the second adherend.

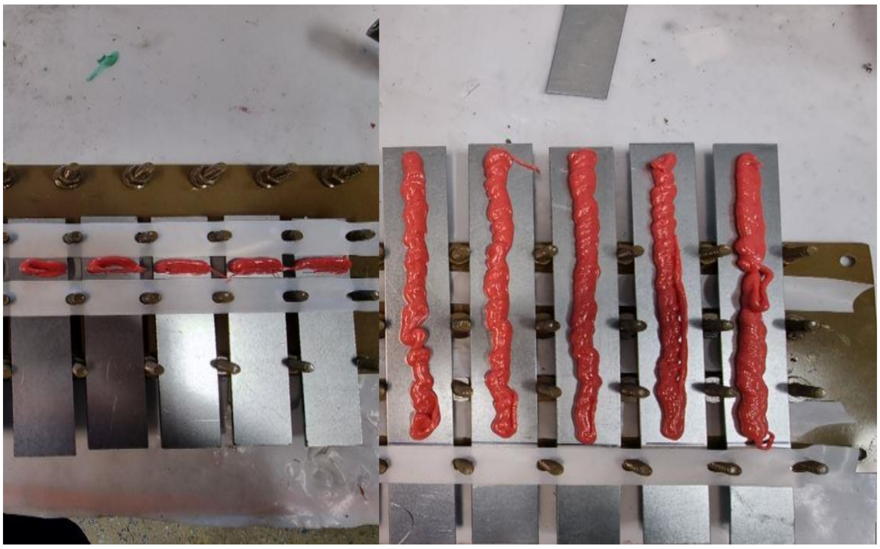
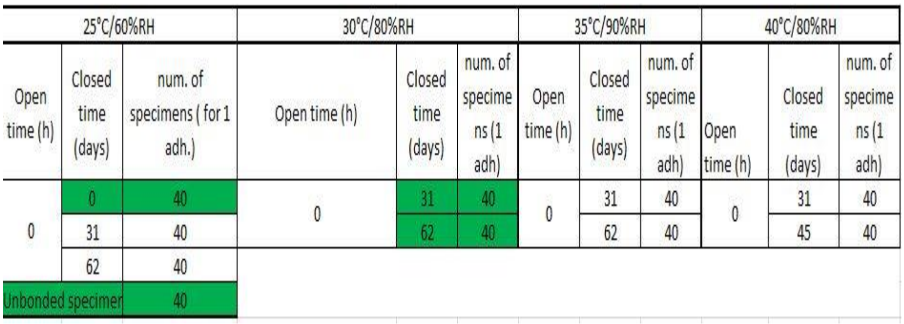


Figure 5: Image of the how the specimen with certain amount of open time are placed in their respectively climate condition. Left corresponds to lap-shear samples while right is T-peel specimens.

Compared to Lap-shear and T-peel, there is no such an open time for the fatigue samples, instead they have open-time for 0 hours which can be seen in table 2. Which mean they were applied with adhesive and directly spot-welded, later placed in a respective climate condition.

Table 2: Describes the original plan of testing fatigue samples, and their condition climate also ageing time.



25°C/60%RH			30°C/80%RH			35°C/90%RH			40°C/80%RH		
Open time (h)	Closed time (days)	num. of specimens (for 1 adh.)	Open time (h)	Closed time (days)	num. of specimens (1 adh)	Open time (h)	Closed time (days)	num. of specimens (1 adh)	Open time (h)	Closed time (days)	num. of specimens (1 adh)
0	0	40	0	31	40	0	31	40	0	31	40
	31	40		62	40		62	40		45	40
	62	40									
Unbonded specimer		40									

The original plan for the fatigue samples was to carry out testing for all the ageing time and condition climates. Due to testing performance occurring by an external company, a decision was taken that the climate condition and age-time with green marks in table 2 will be carried out primary, and the results will be included in this Master thesis. The rest condition time/ageing time will be a project that will be carried out internally.

3.5 Curing condition

Before performing mechanical testing after reaching exposed ageing time, the samples needs to be cured. Therefore, testing samples follows three different curing conditions as in figure 6.

Figure 6: Image of the different curing temperatures and times for the test samples.

For samples being cured, it is necessary to let the oven reach the curing temperature. Also, the countdown shall be counted when the sample's temperature reaches five degrees less than the curing temperature. Before starting the following curing condition, it is essential to let the samples cool off to room temperature.

4 Results

This chapter will present testing performance and failure mode for one of the adhesives. It is the Steel-To-Steel adhesive. Figure 7 shows type of different failure mode that have been indicated and how it might look.

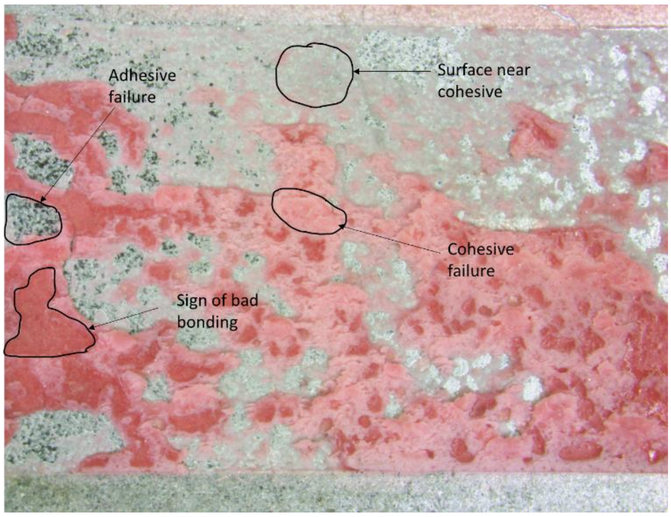


Figure 7: Illustration of type of failure that can be detected on the surface. Sign of bad bonding indicate that the adhesive has not stretched, and to be sure that it is adhesive failure corresponding adherend has to be look through.

4.1 Test result of Lap-shear

In table 3,,, and 6 describes how the shear strength and the average type of failure mode seen at the surface. While figure 8,,,,11 shows how the failure mode looks due to the ageing time.

Table 3: describes how climate condition 25°C/60% relative humidity in this case corresponds to the environment in the production plant influences the strength of the adhesive before curing. Also describes the average failure mode seen on the respective condition. Cohesive failure (CF) allows the fracture of a layer to remain in both surfaces. Surface cohesive failure (SCF) still have cohesive failure but instead small amount of adhesive is remained in one of the surface. Adhesive failure (AF) occurs when a failure between the adhesive and adherend.

Climate condition	Closed/open time	Days	Strength (MPa)	Standard deviation (Std)	Strength of loss %	Failure mode
25°C /60%	**0 hours**	**0**	33,93	0,90	0	90%CF, 10%SCF
		31	32,92	0,74	2,98	85%CF, 15%SCF
		62	31,57	0,72	6,95	90%CF, 10%SCF
	8 hours	**0**	38,07	2,14	+12,2	95%CF, 5%SCF
		31	32,63	0,28	3,83	90%CF, 10%SCF
		62	31,38	0,78	7,52	50%CF,50%SCF
	72 hours	**0**	33,53	0,99	1,17	100%CF
		31	32,62	1,36	3,86	100%CF
		62	30,44	0,92	10,29	65%CF, 35%SCF

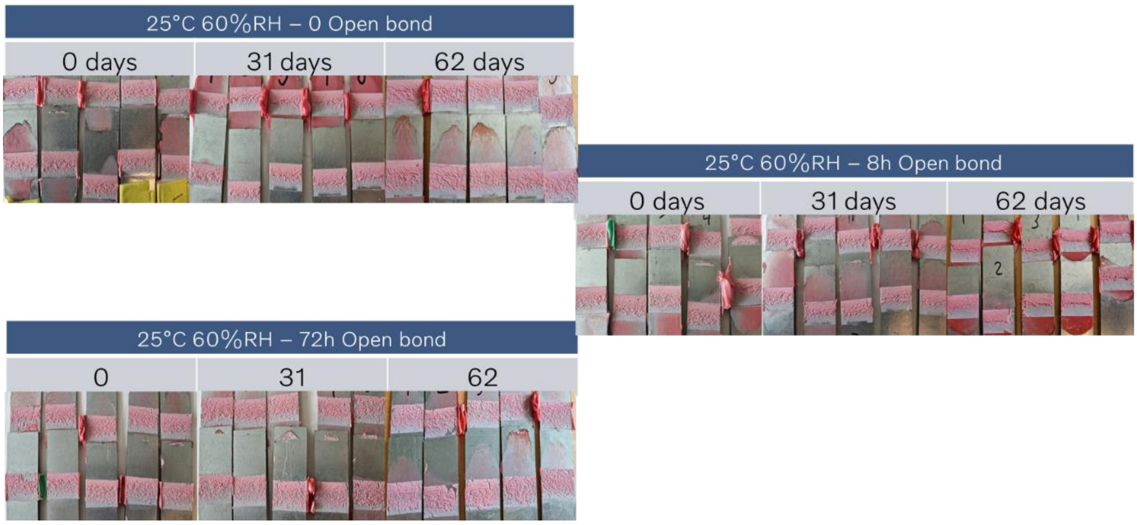


Figure 8: Image of the failure structure of the adhesive with three different initial point. Exposed to an open bond from 8 to 72 hours before ageing and one that is directly bonded, the percentage correspond to the average loss, plus sign indicate shear strength increases. In all three indications of cohesive failure can be seen, also small amount of surface near cohesive failure.

Table 4: average shear strength of the adhesive when specimens are exposed to 30°C and 80 % humidity.

Climate condition	**Closed/open time**		**Days**	**Strength (MPa)**	**Standard deviation (Std)**	**Strength of loss %**	**Failure mode**
30°C/ 80%	**0 hours**		**7**	31,61	1,80	6,84	98%CF, 2%SCF
			14	33,18	0,76	+0,35	90%CF, 10%SC
			31	32,60	0,41	3,91	100%CF
			45	30,95	0.60	8,88	85%CF, 15%SCF
			62	29,62	3,53	12,70	100%CF
	8 hours		**7**	33,24	0,97	2,03	100%CF
			14	30,91	3,02	8,90	100%CF
			31	29,31	1,44	11,85	98%CF, 2%AF
			45	26,86	3,63	20,83	95%CF, 5%AF
			62	24,72	3,53	27,14	70%CF,30%SCF

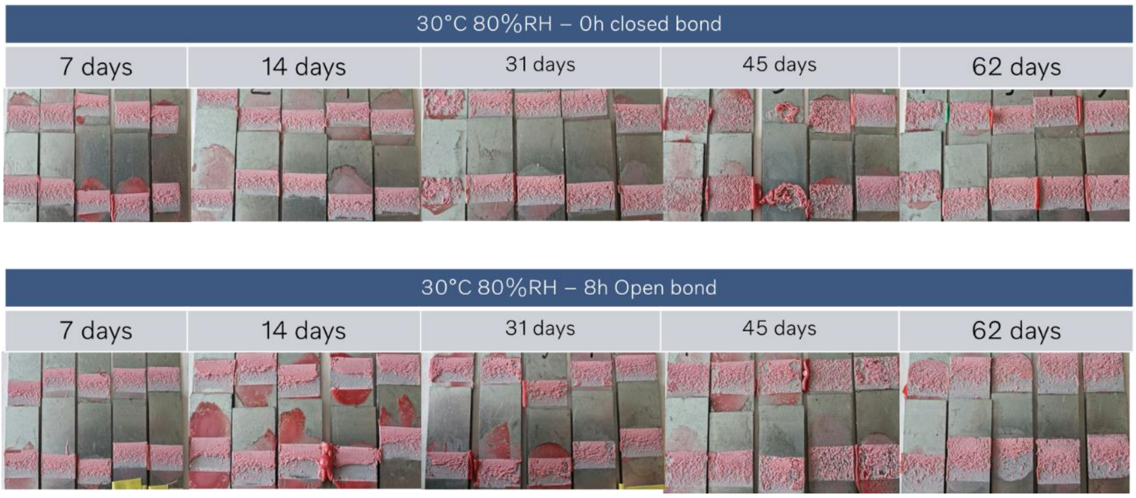


Figure 9: Images of surface structure of the failure mode in condition 30°C/80 % humidity, in both open and closed circumstances, the average loss and failure mode can be seen in table 4.

Table 5: Shear strength of the adhesive after being exposed to 35°C and 90% humidity before curing.

Climate condition	**Closed/open time**	**Days**	**Strength (MPa)**	**Standard deviation (Std)**	**Strength of loss %**	**Failure mode**
35°C/ 90%	**0 hours**	**7**	32,23	0,55	5,01	90%CF, 10%SF
		14	31,93	0,69	5,89	93%CF,7SF
		31	29,99	1,50	11,61	95%CF, 5%SF
		45	23,83	4,20	29,67	55%CF, 20%SCF, 25%AF
		62				
	8 hours	**7**	31,89	0,51	6,01	95%CF, 5%SF
		14	29,21	1,16	13,91	100%CF
		31	19,40	3,58	42,82	50CF,48%SCF, 2%AF
		45	21,27	0,37	37,31	50CF,45SCF, 5%AF
		62				

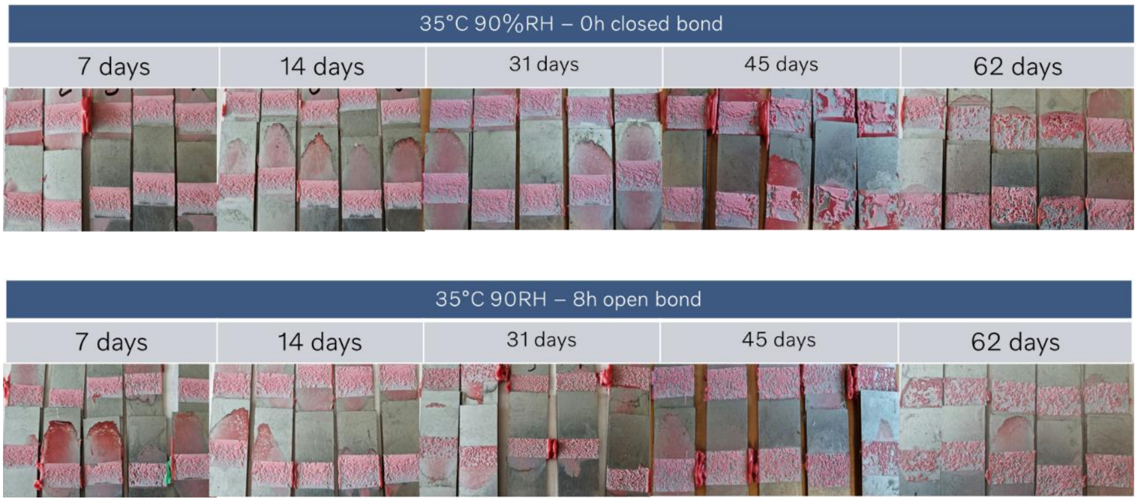


Figure 10: Failure mode of the adhesive at environment 35° C and 90% humidity. I can clearly be seen at closed bond produces cohesive failure and surface near cohesive. For 8h open bond 7 respective 14 gives cohesive failure and surface near cohesive, 31 days the surface appearances slightly change. Cohesive failure can been seen but hard to indicate surface near cohesive, and more avoids and pores are present on the surface compared to previous days.

Table 6: Lap shear results when bond line is exposed to 0 hours and 8 hours in climate condition 40°C and 80% relative humidity.

Climate condition	**Closed/open time**	**Days**	**Strength (MPa)**	**Standard deviation (Std)**	**Strength of loss %**	**Failure mode**
40°C / 80%	**0 hours**	**7**	31,75	0,71	6,42	**90%CF, 10%SCF**
		14	31,04	1,19	8,52	**90%CF, 10%SCF**
		31	30,55	0,38	9,96	**100%CF**
		45	29,18	2,32	13,99	**85%CF, 15%AF**
		62	26,73	0,81	21,22	**90%CF, 10%AF**
	8 hours	**7**	23,95	2.11	29,41	**95%CF, 5%AF**
		14	23,73	1,3	30,06	**98%CF, 2%AF**
		31	23,27	2,68	31,42	**98%CF, 2%AF**
		45	20,63	2,94	39,19	**80%CF, 20%AF**
		62	20,40	1,49	39,88	**70%SCF,20%CF,10AF**

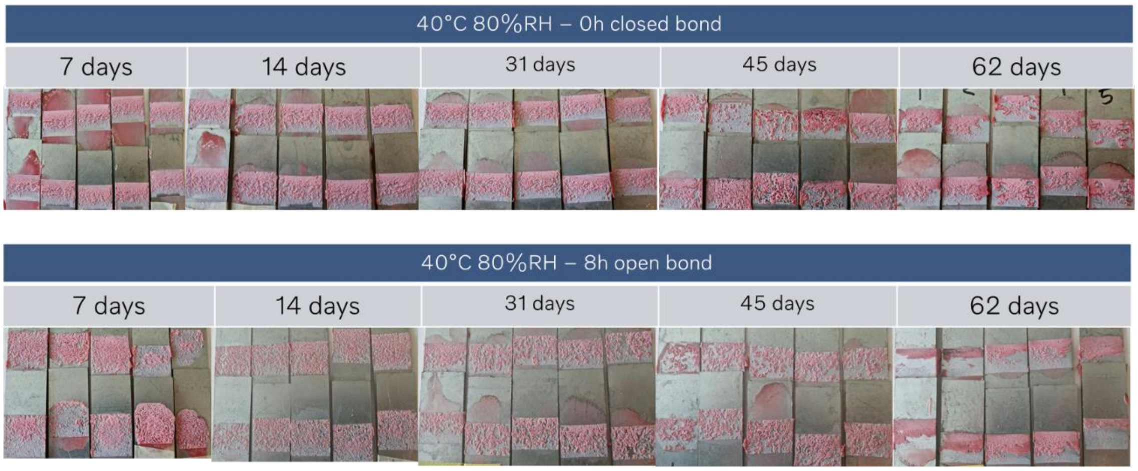


Figure 11: Describes and shows the structure of the failure mode of the adhesive, and how much of original shear strength is lost due to ageing time and exposure condition. For closed days failure mode indicate both cohesive failure and surface near cohesive, around 45 days the structure changes more appearances of pores and voids and darker adhesive voids can be seen, it indicates adhesive failure and bad bonding between adhesive and adherend. For open bond, 40°C/40% is not convenient around 30% of the shear strength losses and after 14 days voids and pores with darker adhesive bond appears on the bond surface.

4.2 Results on the peeling strength

T-peel is used to determine the force required to open a bond where two components are joined together with adhesive. T-peel strength is average load per unit width of bond line necessary to separate bonded materials at an angle of 180. Figure 12,,,and 15 shows the surface structure of the adhesive bond after different ageing-time. In table 7,,, and 10 the average load needed to open the bond can been seen.

Table 7: Peeling strength for the environment corresponding to the production plant.

Climate condition	**Closed/open time**	**Days**	**F average (Favg (N))**	**Standard deviation (Std)**	**Favg of loss %**	**Failure mode**
25°C /60%	**0 hours**	**0**	**215,2**	15,40	0	50%CF, 50SCF
		31	**219,7**	6,26	+2,04	70%SCF,30% CF
		62	**202,88**	8,44	6,07	60%SCF,40%CF
	8 hours	**0**	**212,5**	9,09	1,27	70%SCF,30% CF
		31	**209,11**	17,89	2,91	100%CF
		62	**212,65**	6,45	1,19	70%SCF,30% CF
	72 hours	**0**	**197,77**	6,78	8,81	100%CF
		31	**203,36**	9,10	5,82	50%CF, 50SCF
		62	**213,60**	6,89	0,74	50%CF, 50SCF

Figure 12: How open time/closed time and ageing days influences the peeling strength and failure mode. In all scenario appearances of cohesive and surface near cohesive failure can be indicate.

Table 8: Average shear strength of the adhesive when specimens are exposed to 30 degree and 80 % humidity.

Climate condition	Closed/open time	Days	F average (Favg (N))	Standard deviation (Std)	Favg of loss %	Failure mode
30°C / 80%	**0 hours**	**7**	211.2	10,40	1.8	100%CF
		14	216,1	11,36	+0,41	100%CF
		31	206,9	8,96	3.86	100%CF
		45	217,3	4,91	+0,98	100%CF
	8 hours	**7**	184,6	35,20	14,22	100%CF
		14	180,9	12,13	15,94	100%CF
		31	183,0	25,67	14,96	95%CF,5%AF
		45	173,4	5,76	19,42	100%CF

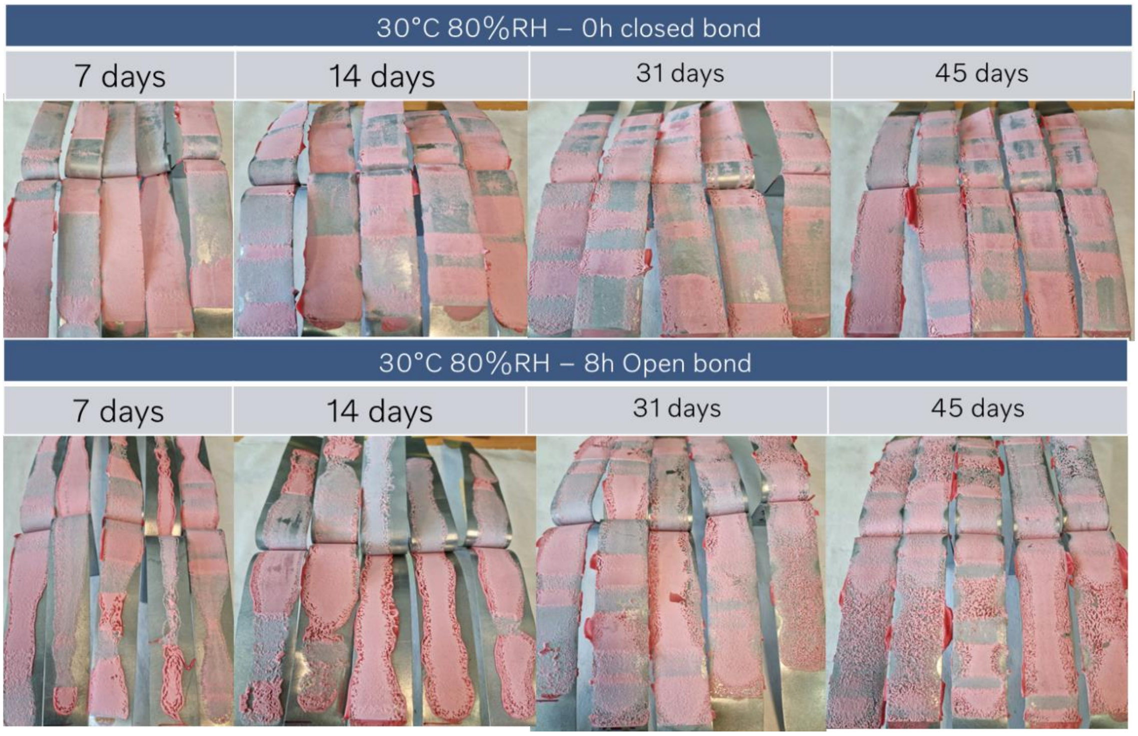


Figure 13: Directly closed bond shows small changes in the peel strength compared to a 8 h open bond. Open bond have higher tendency of producing voids and pores on the surface, also more indication of darker adhesive bond which might indicate adhesive failure on some of the voids. Otherwise closed produces good peel strength and cohesive and surface near cohesive failure.

Table 9: T-peel strength of the adhesive when exposing to a climate of 35°C and 90% humidity.

Climate condition	Closed/open time	Days	F average (Favg (N))	Standard deviation (Std)	Favg of loss %	Failure mode
35°C / 90%	**0 hours**	**7**	215,1	14,12	0,047	100%CF
		14	231,1	15,89	+7,34	80%CF,20%SCF
		31	215,8	13,35	+0,28	90%CF, 10SCF
		62	186,12	10,98	15,62	80%CF,15%SCF,5%AF
	8 hours	**7**	197,4	10,84	8,27	80%CF,20%SCF
		14	228,4	12,13	+6,13	90%CF, 10%SCF
		31	204,2	14,44	5,11	90%CF, 10%AF
		62	140,49	9,64	34,69	70%CF, 30%AF

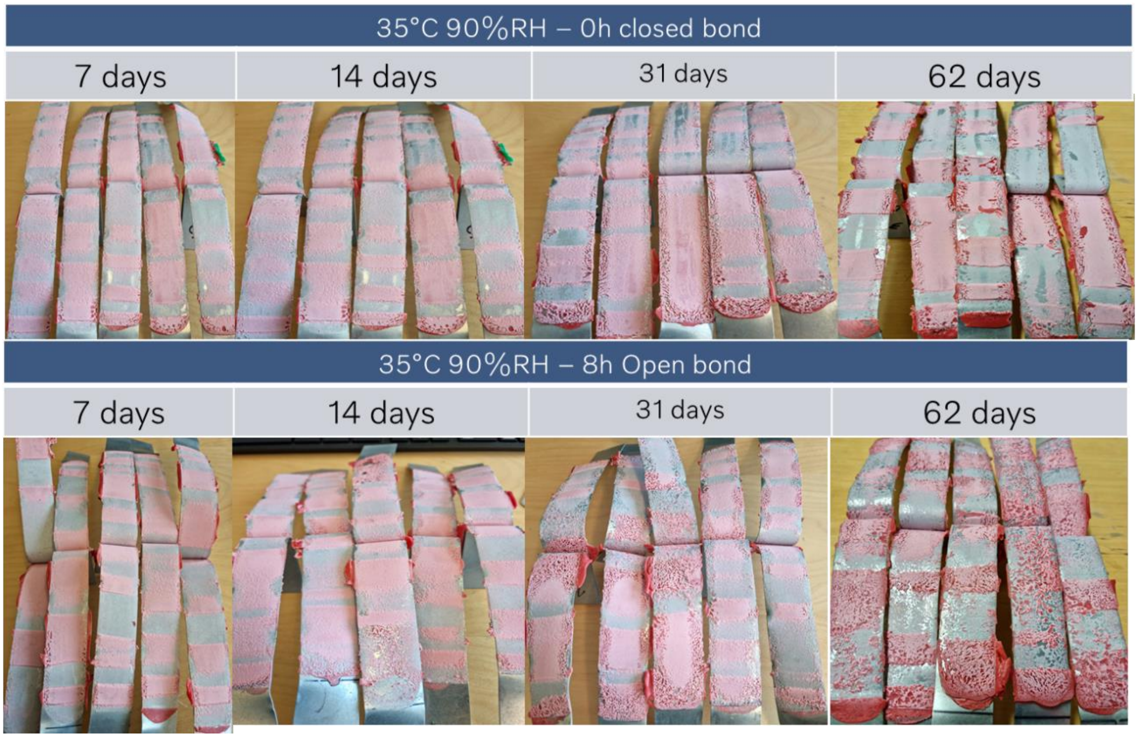


Figure 14: Failure mode of the climate condition on table 9, majority of failure mode are cohesive failure and surface cohesive failure. Small amount of adhesive can be seen in with 8 open-time and 31 days ageing-time.

Table 10: Strength various in the harshest climate condition 40°C and 80% relative humidity.

Climate condition	**Closed/open time**	**Days**	**F average (Favg (N))**	**Standard deviation (Std)**	**Favg of loss %**	**Failure mode**
40°C / 80%	**0 hours**	**7**	223,9	5,39	+4,04	60%CF,40%SCF
		14	191,7	31,87	10,92	100% CF
		31	198,7	27,08	7,67	100% CF
		45	198,7	10,96	7,67	90%CF, 10%SCF
		62	144.59	34,23	32,81	70%CF,30%AF
	8 hours	**7**	172,75	19,42	19,73	100%CF
		14	192,4	11,55	10,60	100%CF
		31	124,7	10,64	42,05	90%CF,10%AF
		45	110,32	11,41	48,74	50%CF,50%AF
		62	58,9	12,59	72.62	70%AF,30%CF

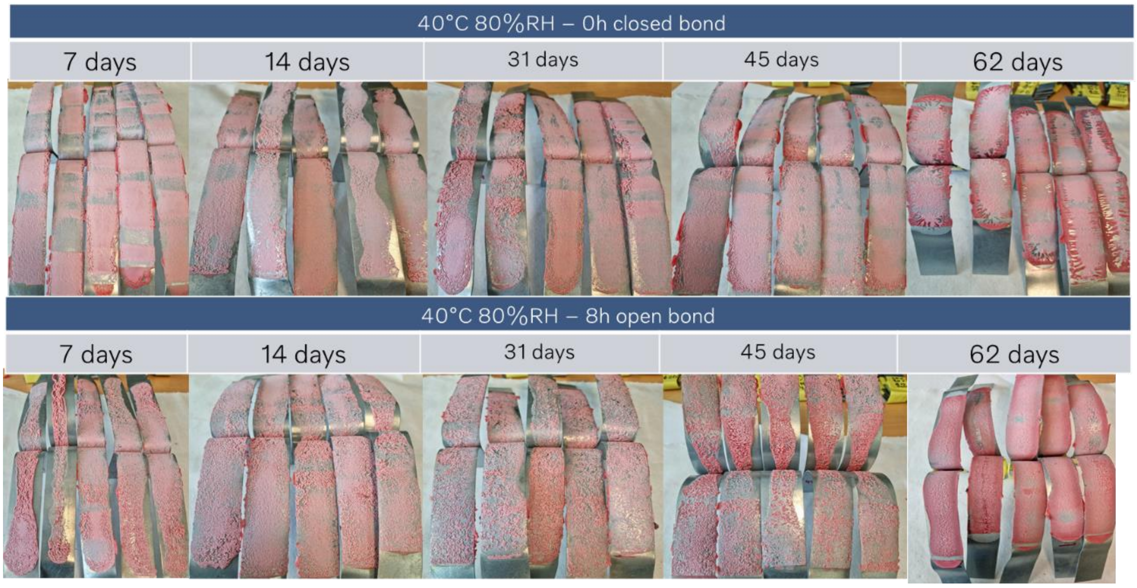


Figure 15: 40°C and 80% humidity was the harshest condition tested in terms of temperature, also long the bond is closed and bonded with in a week one, the moisture and temperature might not have huge influences. But open bond is highly not recommended where up to one week can lead to loss of 20%. The failure for closed starts to shift after 31 days where more pores and voids can be detected, while open bond produced voids and pores within 7 days. Some adhesive failure can be detected in some places of adhesive specially closed bond after 31 days and after 14 days for open bond.

4.3 Mechanical performance comparison

In this section the mechanical properties of both adhesives will be presented in histograms seen in figure 16,,,,,,and 23.

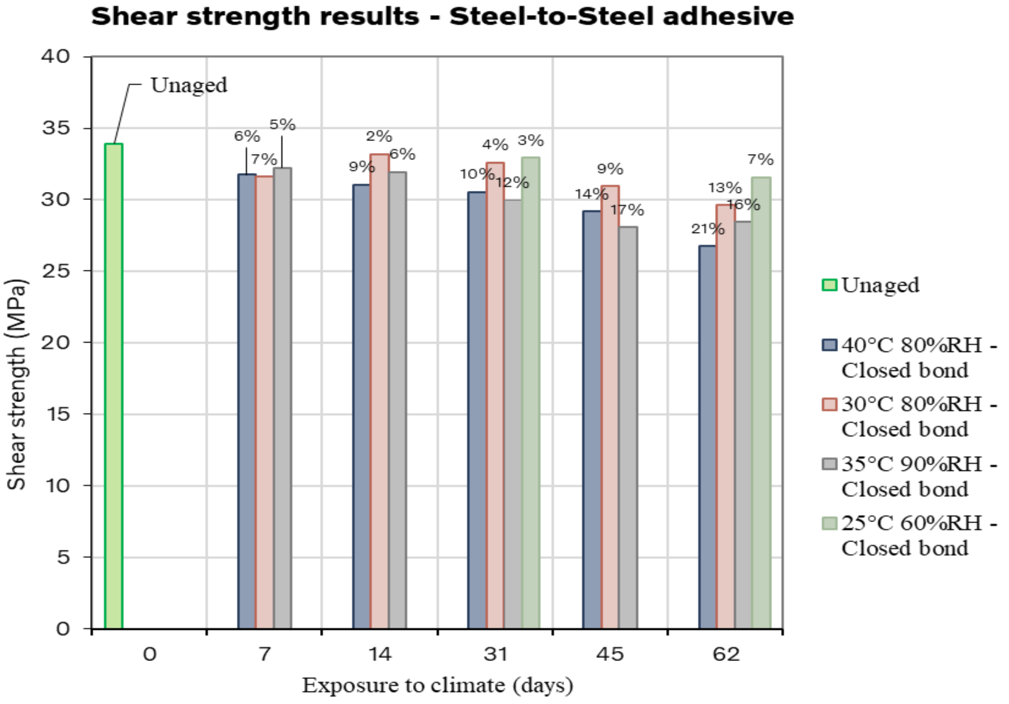


Figure 16: Shear strength result for closed bond steel-to-steel adhesive.

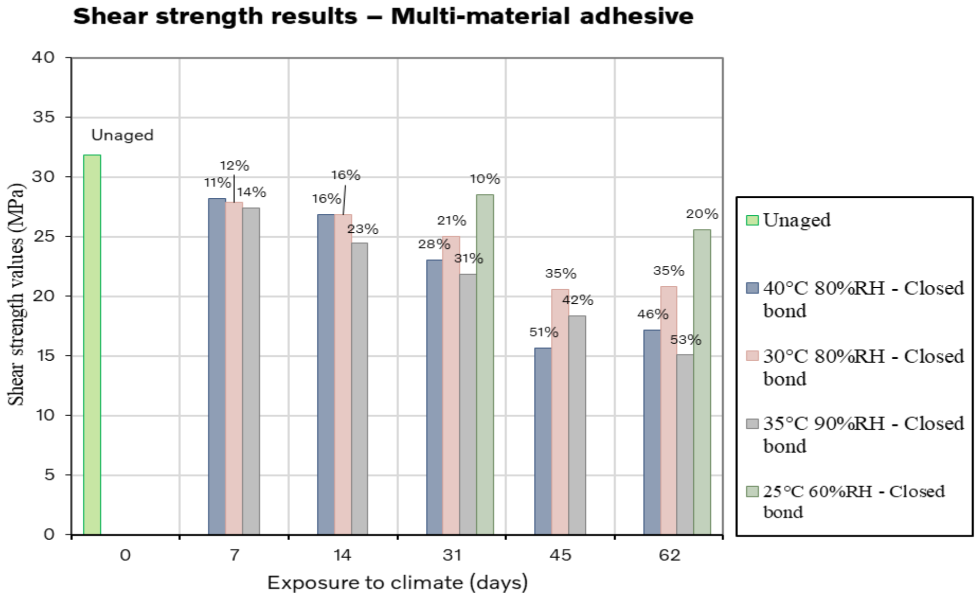


Figure 17: Shear strength result for closed bond multi-material adhesive.

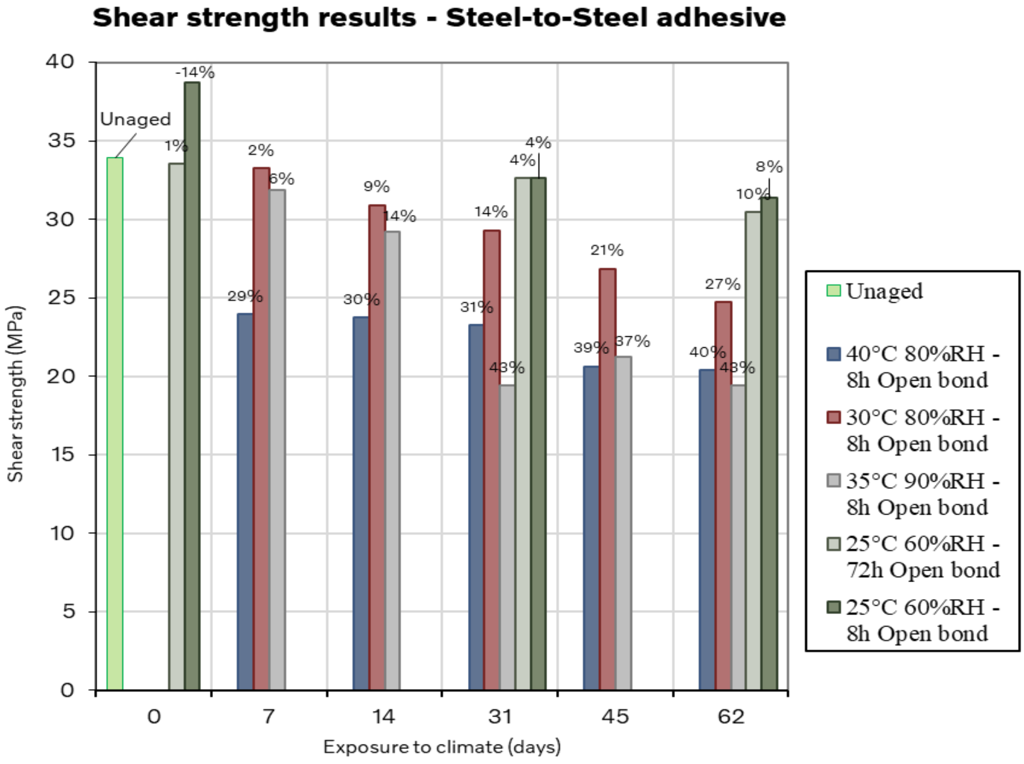


Figure 18: Shear strength results for steel-to-steel adhesive when having open exposure before boding adherends.

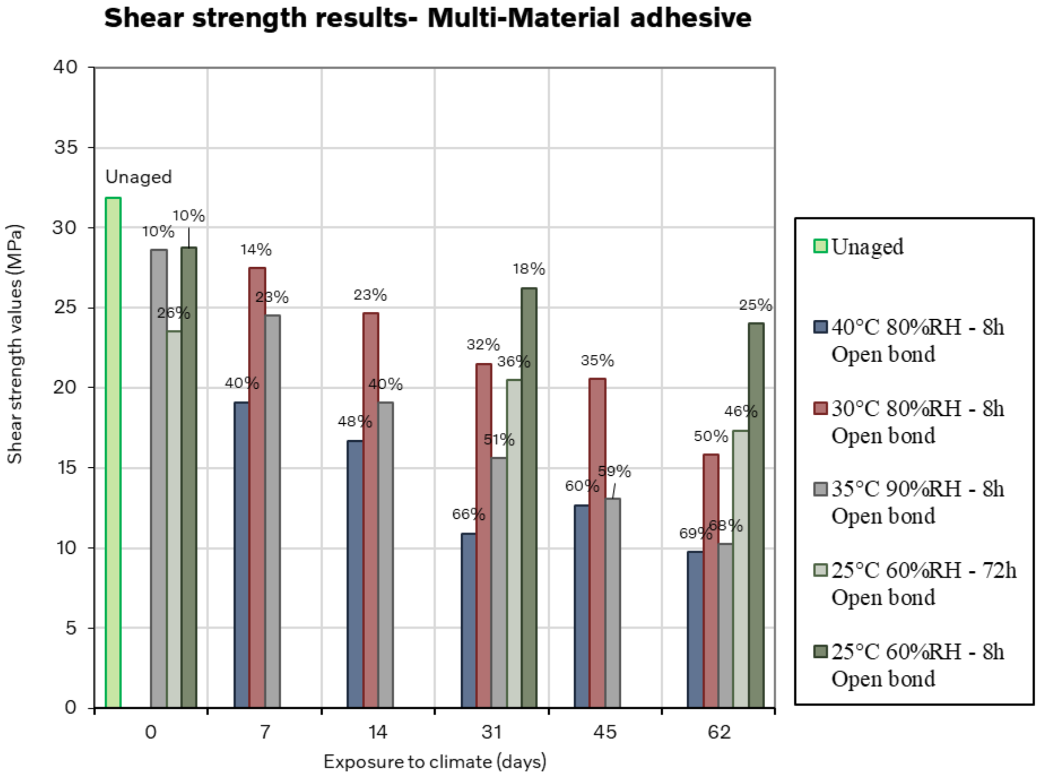


Figure 19: Shear strength results for multi-material adhesive with open exposure time.

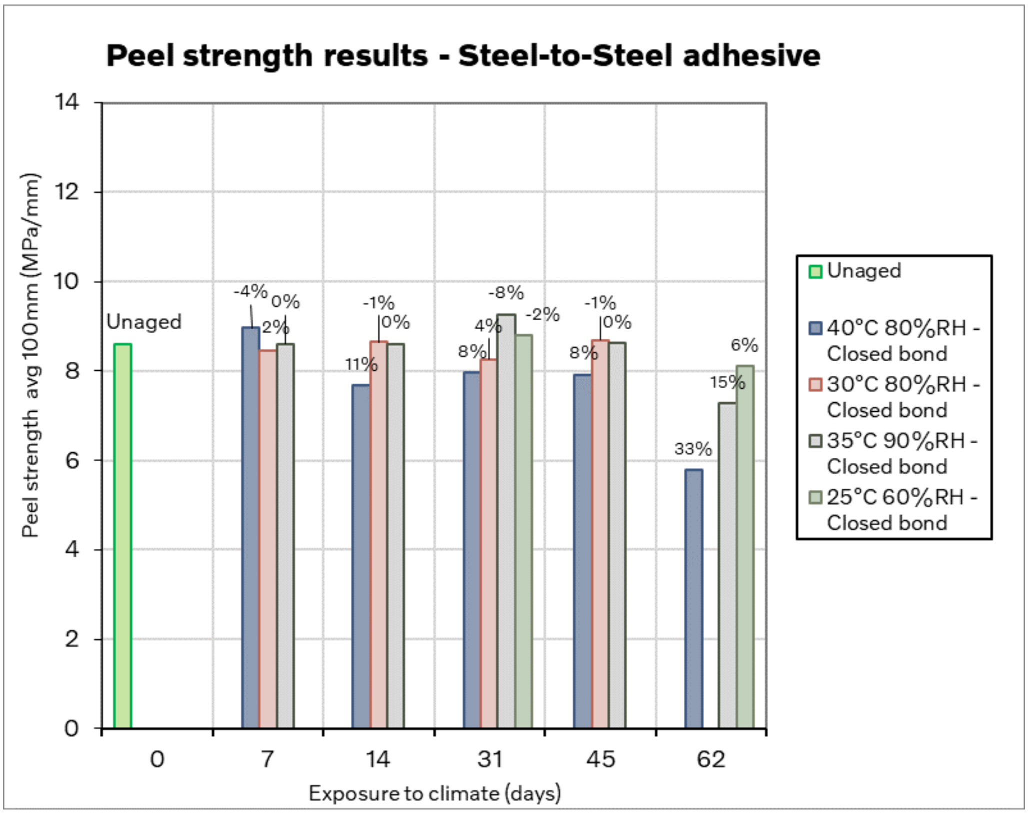


Figure 20: Peeling strength for the Steel-to-Steel adhesive with closed bond-lines.

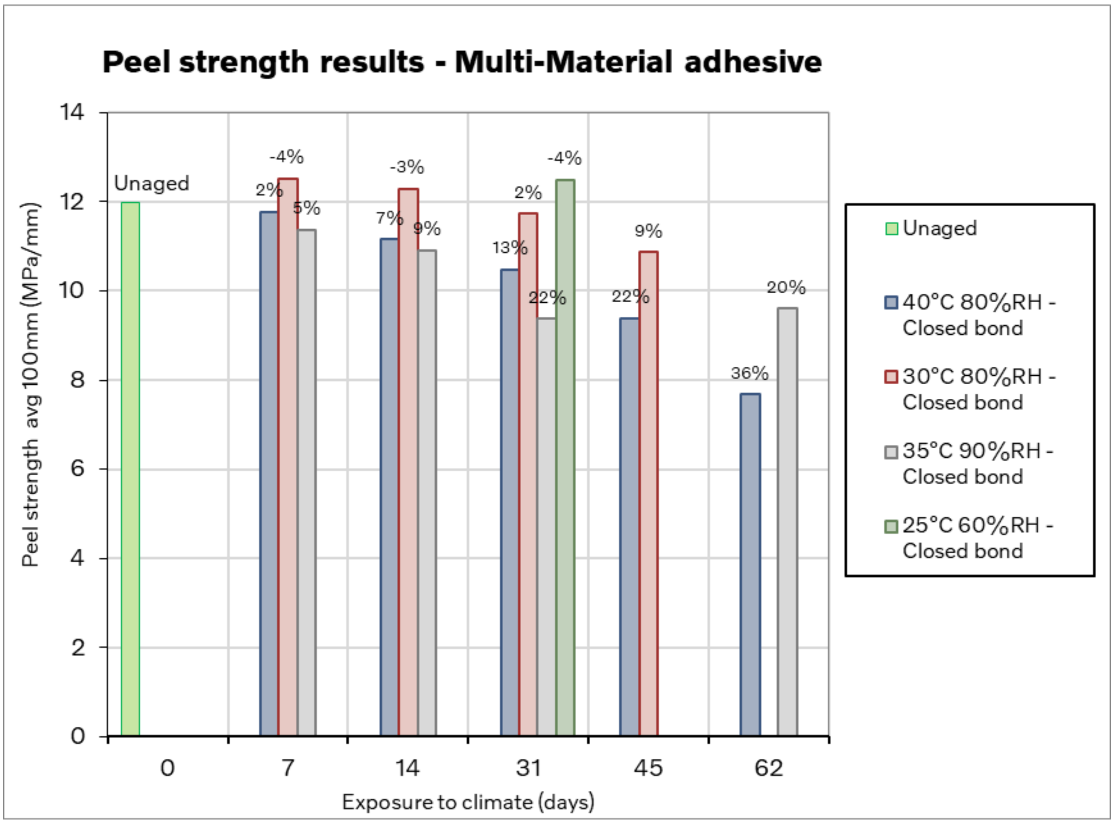


Figure 21: Peeling strength for the multi-material adhesive with closed bond-lines.

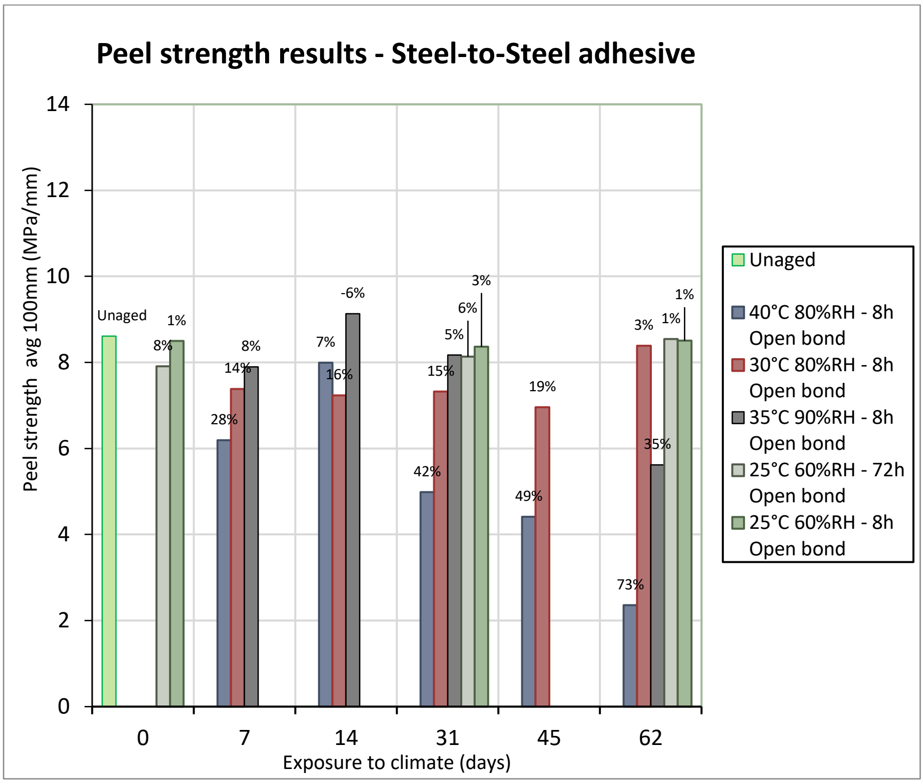


Figure 22: Ollustration of how peeling strength changes due to age-ing for Steel-to-Steel adhesive with open bond-lines.

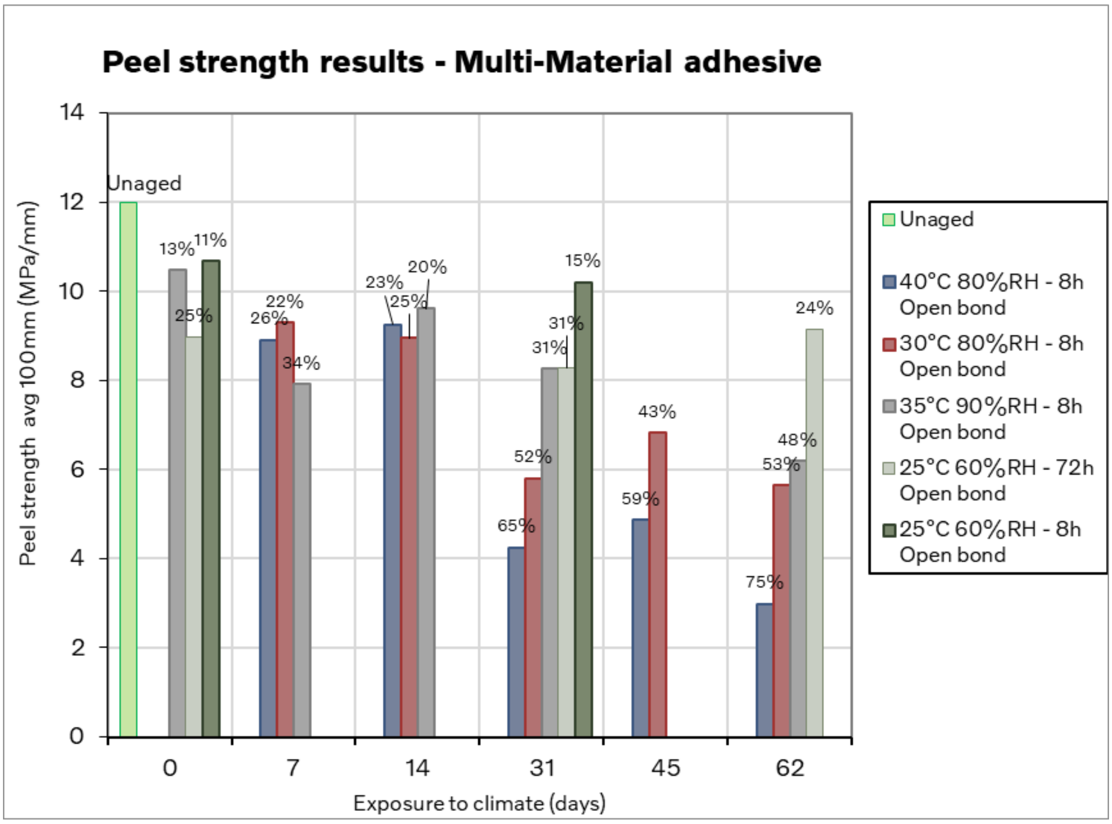


Figure 23: Peeling strength for multi-material adhesive with open exposure.

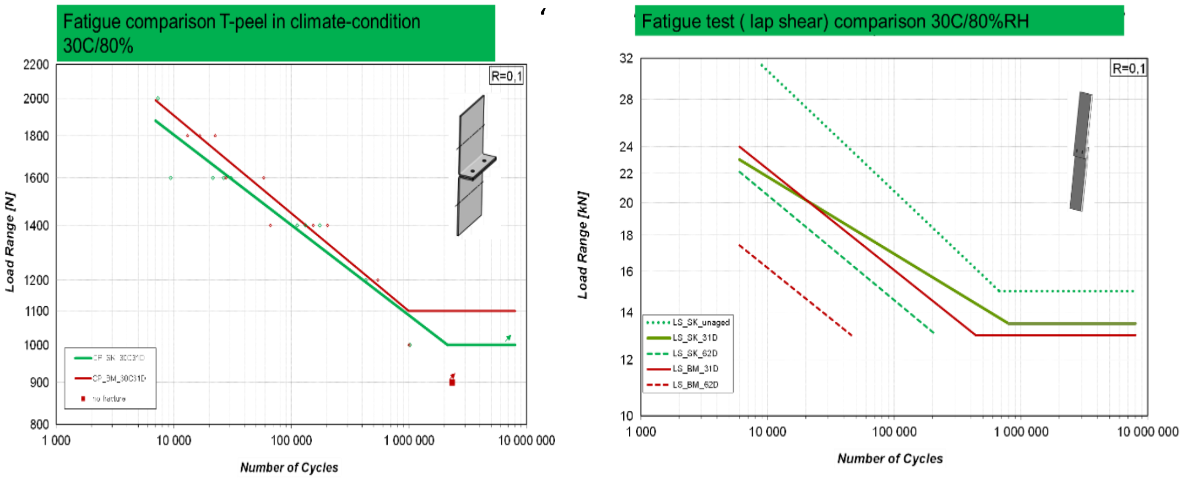


Figure 24: Describes how fatigue properties changes for T-peel and lap-shear testing in climate condition 30C/ 80%RH. Red line is the steel-to-steel adhesive while green line corresponds to multi-material adhesive. Dots-line is the unaged, line spacing is 62 days ageing and 3 is the fit line.

4.4 Fractography comparison

Moisture uptake in uncured bond-lines showed influences on the surface structure of the broken specimens. Comparison on how moisture affected the adhesive failure mode can be seen in figure 25, 26 and 27

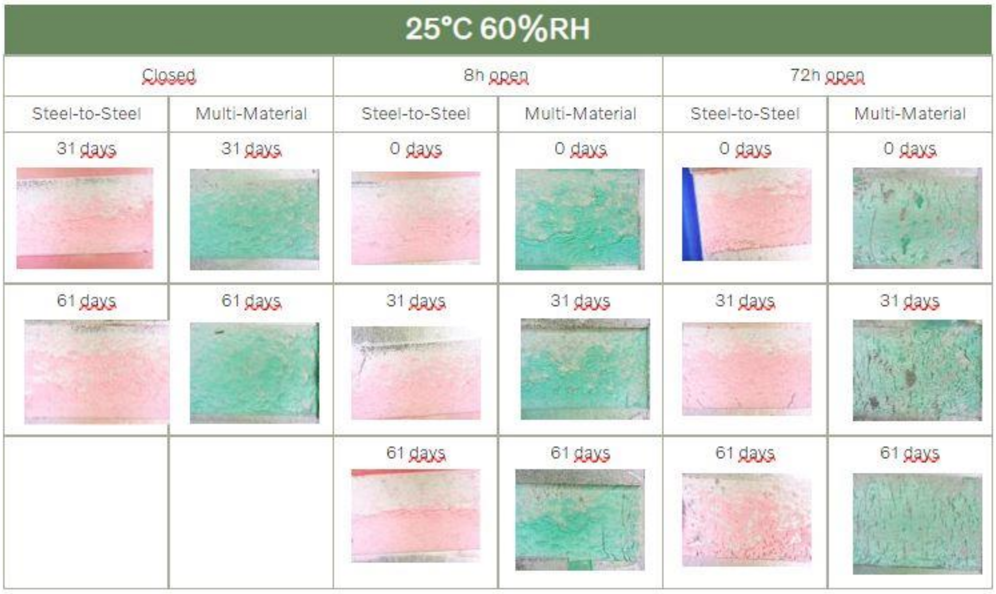


Figure 25: Demonstrates the surface structure for climate condition 25°C and 60% relative humidity.

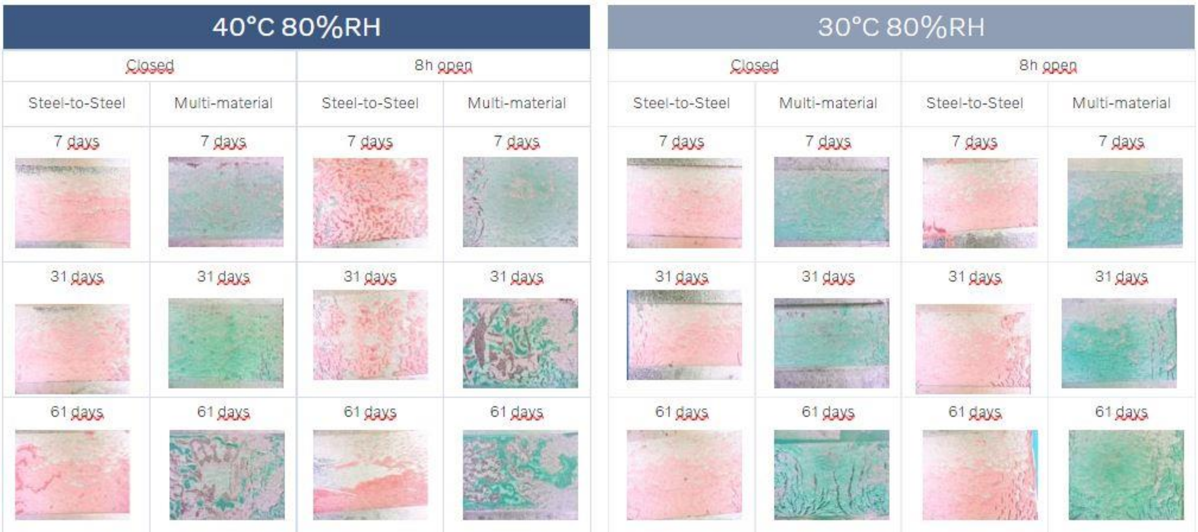


Figure 26: How climate condition 40°C/80%RH and 30°C/80% influences the failure mode of both adhesive.

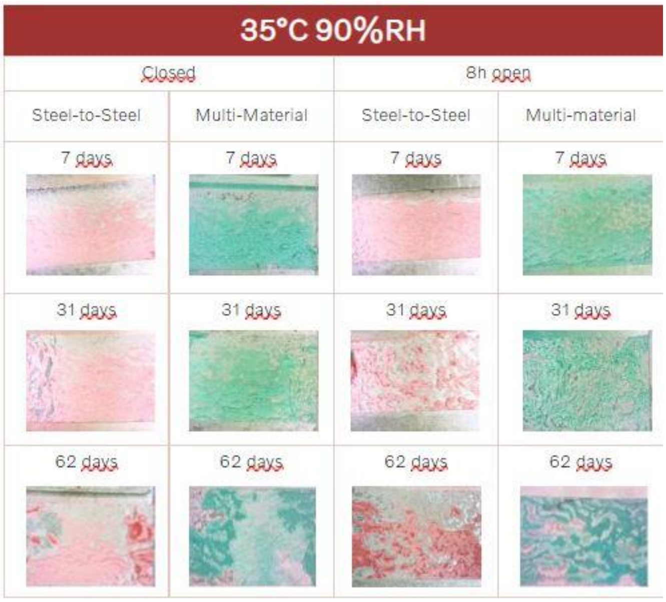


Figure 27: How highest relative humidity and temperature 35°C affect the failure mode of the adhesives.

5 Discussion

In this chapter a deeper understanding of the results will presented and see if both adhesive maintains good quality regardless moisture uptake, also if changes are need in Volvo cars standard regarding how long samples can be exposed to moisture and temperature before being cured.

The structural adhesive used for steel components presents superior shear strength to multi-material adhesive which can been seen in figure 16 and 17. Moisture uptake in lower temperatures under 30°C and 80% revealed that the multi-material adhesive could be stored up to 45 days without losing 20% of its shear strength but keeping multi-material in figure 19 open for 8 hours in the storage time reduces to 7 days. Moisture uptake in temperature, significantly above 40°C and 80%, is not acceptable due to high loss in shear strength, especially having an open bond for 8 hours which can lead to a loss of 29% in 7 days in figure 18. Multi-material adhesive is more sensitive to moisture uptake and has a greater impact on the shear strength. For the production plant environment keeping the bond open for 72h can lead to multi-material adhesive loss up to 24% while steel-to-steel losses around 6%. For temperatures around 40°C and 80% for closed bond lines in 7 days, multi-material adhesive loses 10% more shear strength than steel-steel adhesive.

Peeling strength of steel-to-steel adhesive showed no tendency of high loss in lower climate and 0 hours closed bond, most of the samples decrease or increase little bit which makes it hard to identify correlation. For 8 hours of open bond in figure 22, we observed some variation in the average force (Favg) loss, but everything points out that exposing open-bond lines for 8 or more hours can lead to greater loss in peeling strength compared to closed joints in figure 20. Increasing the temperature to 40°C and having open bond lines produces the highest losses in the peel strength. In figures 21 and 23 multi-material adhesive had huge loses in the peeling strength and open bond-lines shall be prevented since all condition resulted in more than 20% loss expect 25°C/60% 8h and 0h. Most closed bond samples observed cohesive failure and surface cohesive failure.

Analyzing the facture surface, we indicated that for elevated temperature with open bond-lines introduce defects such as pores on the surface specially for multi-material adhesive which can be seen in figure 25, 26 and 27. Steel-to-steel indicate mostly cohesive failure and surface near cohesive failure for majority of the closed bond environment, and after 62 days ageing-time some bad bonding and adhesive failure can be seen. But for the open climate condition 25°C/60%RH and 30°C/80% RH some bad bonding can be identified after 31 days for climate condition 30°C/80% RH. Compared to multi-material where some adhesive failure can be seen after 72h exposure time in figure 25. Open bond-lines for climate condition around and above 40°C/80%RH shall be avoidable due the huge loses in mechanical performance but also due to the high influences on the fracture surface of the adhesive failure, where some adhesive failure and bad bonding can be identified after 7 days.

Fatigue testing have only been done at climate condition 30°C / 80RH, and it can be seen in figure 24 that ageing-time have impact on the fatigue performance. It is also indicated that Steel-to-steel fatigue performance is more sensitive to moisture uptake compared to multi-material. Comparing both adhesive steel-to-steel produces greater fatigue performance for higher load-range with lower number cycles, and the opposite for higher number cycles and low load range where multi-material is superior.

According to Volvo Cars standard, parts with adhesives in climate condition of 25°C/60% RH can maximum have open bond line up to 72h before being cured, and parts directly closed can maintain uncured for up to 31 days. For slightly higher temperature 30°C and 80%RH open bond lines need to be cured within 8 hours and closed up to 14 days. A maximum of 20% of the continuous joint may contain defects, and a maximum of 20% losses in mechanical properties are allowed. For the steel-to-steel adhesive the results indicate it fulfill the Volvo Cars standard. It also shows that by keeping them 8 hours open in an environment over 40°C/80% more around 29% of the shear strength and 20% of the peeling strength decreases which make it necessary to avoid transportation of open bond-lines between production plant and supplier in uncontrolled condition. On the other hand, multi-material adhesive shows a loss of 24% in a climate of 25°C/60% RH for 72h exposure, which is not acceptable.

By adapting the production chain after multi-material adhesive, steel-to-steel adhesive will automatically fulfil the requirements. First, adjustment needs to be made to the standards and the production plant. Theoretical it will be perfect to directly bond the part together when applied with adhesive, giving room to keep them up to 31 days before curing. However, since this might be practically harder to implement in the production chain**,** another way is to reapply the adhesive after being open for 8 hours, for 8 hours open only leads to a loss of 8% in the shear strength.

5.1 Conclusion

- Steel-to-Steel adhesive produces greater lap shear and lower peeling strength. Multi-material is more sensitive to moisture uptake
- Climate exposure of uncured bond lines has an influences and effect on the final performance of the joint,
- The degradation is increased for harsher climate condition, especially when bond lines are directly exposure to high condition
- Higher climate condition shows a tendency of introducing defects in form of pores on the cohesive failure mode.
- Multi-material adhesive doesn’t fulfil Volvo Standards, and the time should be lowered for the climate condition 25%/60°C.

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