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Design of new test setups for characterization experiments of adhesives

Internship at Corporate Research and Advanced Engineering, Robert Bosch GmbH

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Preface

At the end of spring 2018, I started looking for an internship. I already made the choice that I would like to do the internship somewhere outside of The Netherlands, but preferably within driving distance from The Netherlands. This was quite challenging, as there are not many English internships available.

Luckily, I found this internship at Robert Bosch GmbH in Renningen, Germany. This internship fitted really well with my interests and I also had a good feeling with the company. It was quite challenging to get started within a short notice, but I am glad I accepted this internship, as I had a great time in Germany.

In this report, I will discuss what I did during my time in Germany. As my internship lasted half a year, it is not possible to discuss everything in high detail within one report. With this report, I tried to give a good overview without making this report too long. Of course, I can provide more detail on certain choices and results if wanted.

I would like to thank Philipp Weißgraeber for giving me this opportunity to work at Bosch on this nice and interesting topic. I learned a lot about working with adhesive and I liked collaborating with you. Also, I would like to thank Christoph Üffing for helping and mentoring me and making sure that I always had enough candy to eat. Finally, I would like to thank Andre de Boer for accepting this opportunity to do this internship.

Summary

In order to simulate the brittle failure of an adhesive, the material properties of this adhesive must be known. For this, many so-called single lap joints must be made and tested. Currently, the adhesive layer thickness of the joints is controlled by placing spacer wires within the adhesive. However, this might influence the results as the wire might introduce nucleation points for cracks. In this report, the development process of a new jig to effectively and accurately produce single lap joints without spacer wire is discussed. This new concept is compared with the existing methods to produce the single lap joints. In total, three iterations of this jig are developed.

Using these new jigs, experiments have been performed to determine the material properties of an epoxy adhesive. The influence of different overlap lengths and adhesive layer thicknesses of the joint is tested. The samples made with the new jig are compared with similar samples made with the traditional method using spacer wires. Furthermore, the influence of the fillet and curing method is investigated and discussed.

Besides the tests with the single lap joint, three other projects are discussed in this report. First, a method has been tested that can produce bulk specimens of epoxy-based adhesives. Results and possible improvements of this method are discussed. Second, a Matlab tool has been developed to simulate the internal forces within a single lap joint's adhesive layer. The development process, including further improvements, are treated in the report. Finally, two different tests with ductile silicone adhesive are executed. In the first experiment, the internal pressure created due to thermal contraction of the adhesive is estimated. In the second experiment, the nucleation and growth of cavity bubbles during cooling down of the adhesive is followed.

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Key words: Adhesive, Bulk specimen, Cavitation bubbles, Epoxy, Fillet, Matlab, Single Lap Joint, Silicone, Spacer wire, Stress analysis

Contents

1	Intr	oducti	ion	5						
2 Single Lap Joint Jig										
	2.1		ustification	6						
	2.2		ng methods	6						
		2.2.1	Spacer Wires	6						
		2.2.2	Finger samples	8						
	2.3	New c	oncepts	14						
		2.3.1	Requirements	14						
		2.3.2	•	15						
	2.4		•	16						
	2.1	2.4.1	1	17						
		2.4.1 2.4.2		18						
		2.4.2 2.4.3	0	19						
		2.4.3 2.4.4		19						
		2.4.4 2.4.5		19 19						
		-		-						
	0.5	2.4.6		21						
	2.5		0	21						
		2.5.1	Further improvements	22						
3	Cha	ractor	isation Experiments	23						
0		23								
	$3.1 \\ 3.2$			$\frac{23}{23}$						
	0.2	3.2.1	•	$\frac{23}{23}$						
		3.2.1 3.2.2	0	$\frac{23}{25}$						
			8							
		3.2.3	0	26						
		3.2.4	8	28						
		3.2.5		28						
	3.3			30						
		3.3.1	1	30						
		3.3.2		32						
		3.3.3	1	32						
		3.3.4		34						
		3.3.5	0	35						
	3.4	Furthe	er experiments	37						
		3.4.1	Thick Adherent Shear Test	37						
		er Pro	• ,	90						
4	J ~	39								
	4.1			39						
		4.1.1		39						
		4.1.2		40						
		4.1.3		40						
		4.1.4	0	41						
	4.2			41						
		4.2.1	Idea	42						
		4.2.2	Execution	42						
		4.2.3	Possible Extensions	43						

		4.3.1	e Adhesive Cavita Internal pressure dT Experiments							 		 			 		44
A	A Robert Bosch GmbH									48							
в	Refl	ection															49

Chapter 1

Introduction

The main goal of this internship is to develop a new way to prepare so-called Single Lap Joint samples. These samples are mainly used to determine fracture properties of different adhesives, as discussed in section 3.1. During the execution of this main assignment, a few other assignments have been performed on the side. As describing the internship chronically will result in constant jumping between these assignments, the report will look at the assignments one-by-one.

First, attention will be paid to the design and iterations of the Single Lap Joint jig, see chapter 2. Some tests have been executed using this jig. These experiments are explained and evaluated in chapter 3.

The second part of the report is about the other projects, in arbitrary order. First, the design process of a jig to prepare bulk specimens is treated in section 4.1. Next, an easy-to-use Matlab tool has been developed to predict internal stresses inside the adhesive layer of a Single Lap Joint. This tool will be discussed in section 4.2. Finally, two experiments conducted on a silicone adhesive will be discussed in section 4.3

After this, a quick description of Robert Bosch GmbH, the Renningen R&D campus and the CR/APP department is provided, see Appendix A. A reflection of my functioning during the internship can be read in Appendix B.

Chapter 2

Single Lap Joint Jig

2.1 Test Justification

An important part in the assembly of electrical components within Robert Bosch, is by gluing different components together. Many different adhesives are used for different purposes. Some of these adhesives, like epoxides, show a brittle failure behaviour. Contrary to a ductile material, where a yield criterium can be applied to elements in a FEM-study to predict the failure load of such an adhesive, brittle failure cannot be easily implemented because of the high stress gradients around an initialized crack. Different models have been put up to predict the fracture behaviour of brittle materials.

For ductile failure, the required parameters like the yield strength can be determined directly from a tensile test of the behaviour. However, the required parameters for brittle failure modelling cannot be easily determined. Multiple tests must be executed and compared to approximate the right value.

A common test to evaluate an adhesive is a so-called Single Lap Joint (SLJ) test. Here, two adherends are glued together with a determined overlap and adhesive layer thickness, see Figure 2.1. When testing these bonds, the adherends will bend slightly because of the alignment difference between the top and bottom adherend. Within the adhesive, this results in a combination of normal and shear stresses. At a certain force, the adhesive fails. The maximum force sustained by the sample is a measure for the toughness of the adhesive for that joint configuration. The main goal of this project is to predict the failure load for these and other adhesive joints using brittle adhesive. Many models and criteria have been proposed to determine to predict the fracture behaviour of these joints. By comparing data of practical experiments with different geometries, an appropriate model for brittle failure can be found. From this model, the material properties needed to determine the failure load can be calculated.

In order to do so, at least 6 different configurations of the single lap joint are needed to determine these material properties. As a rule of thumb, around 6 samples per configuration are needed to give statistically accurate values for each configuration. This means that in order to analyse one adhesive, 36 samples must be prepared and tested for each adhesive. Having a good method to prepare those samples can increase the accuracy of the results and decrease the amount of time needed to perform such experiments.

The main goal of this internship is to design a new way to prepare these samples, as existing methods have their limitations. In this chapter, these excising methods will be compared. Based on this, a new concept will be discussed. Multiple iterations have been made of this concept and the changes between different concepts will be motivated. Finally, some further optimisations of the final concept are discussed.

2.2 Existing methods

2.2.1 Spacer Wires

Until the start of this project, all single lap joint samples at Bosch were prepared using spacer wires to control the adhesive layer thickness. A simple jig is used to align the adherends with each other, see Figure 2.2.



Figure 2.1: Single Lap Joint sample clamped in a testing machine.



Figure 2.2: Gluing the specimens using the normal method. Below is the jig with the slider to control the overlap length. On top are glued samples and the glue dispenser.

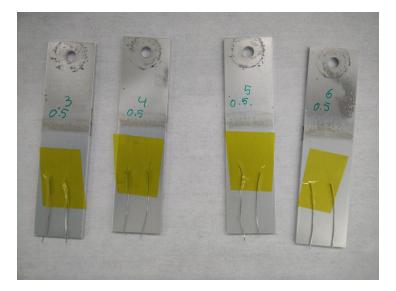


Figure 2.3: Spacer wires used to maintain the right distance.

To use this jig, a manual screw clamp is placed into the appropriate holes in the jig. On the bottom adherend, prepared with a suitable surface treatment, two wires are placed in the lengthwise direction of the samples. These wires have an accurately defined diameter. Kapton tape is sometimes used to keep the wires in place, see Figure 2.3. A slider on the other end of the jig controls the overlap length. An excess amount of adhesive is placed on the adherend. Normally, this is done using a glue gun to place a drop of adhesive on the adherend. A spatula is used to spread the adhesive evenly over the surface of the adherend. This step is important to ensure good wetting of the adhesive on the adherend. After this, the second adherend is placed on top of the first and the screw clamp is tightened as can be seen in Figure 2.4. The two wires limit how far the adherends can be pressed on top of each other and thereby control the adhesive layer thickness. Once all samples are finished, they are generally heat cured in a convection oven. Curing times and temperatures depend on the adhesive used.

This method is easy to execute and requires, besides one simple jig, no special tools. This explains why this method is used extensively within Bosch to produce such samples. There are however a few disadvantages to this method.

First, it is quite a messy method. Spreading the adhesive with the spatula results in adhesive sticking everywhere, especially if the adhesive is stringy. Also, using the spatula introduces air bubbles in the adhesive. It is therefore important to wait a few minutes for most of the air bubbles to rise to the surface and then pop them. This makes the process rather slow and some unwanted air bubbles might still be present in the final sample.

Also, when pressing both adherends together, excessive adhesive oozes out. This means that the jig must be cleaned with a solvent between each sample. In order to prevent adhesive sticking to the clamp, they must be covered in aluminium foil. It is quite a lengthy process tearing the aluminium foil to the right size and wrapping it around the clamp in such a way it does not fall off. All in all, it takes around 8 minutes per sample to prepare.

The main disadvantage, however, is the present of the spacer wires inside of the adhesive layer. Mainly for brittle adhesives, these wires will cause local stress concentrations. These points can act as the nucleation point of a crack, influencing the failure load of the sample. This effect is further investigated in subsection 3.3.3. It is therefore important that the new method does not use any spacers within the adhesive layer.

2.2.2 Finger samples

To get rid of the spacer wires, a new way to prepare the samples was previously developed at Bosch. This method works by moving the spacers outside of the adhesive layer on separate 'fingers', see Figure 2.5. Precision shim washers are used to control the adhesive layer thickness. In this design, 4 samples can be made per set. The samples are laser cut out of a sheet of steel or aluminium but are still connected with small taps that can be cut away after gluing. Adjacent to the samples, strips with holes are used for alignment. Three sets of holes are available to create samples with different overlap lengths.



Figure 2.4: The sample assembled in the jig.

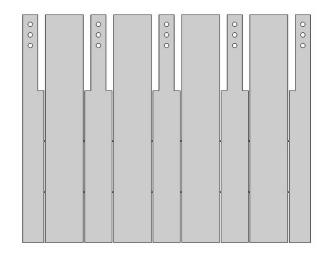


Figure 2.5: Geometry of the finger samples. The precision shim washers are placed at the holes in between the adherends.

Sample	Aimed	thickness	Measured	average	Standard	deviation
	(µm)		thickness (µm))	measureme	ents (µm)
1	100		68		37	
2	100		50		27	
3	300		69		32	
4	300		60		39	
5	500		201		46	
6	500		56		24	

Table 2.1: Average and standard deviation of the measured data of the finger samples

The samples are prepared in the following method, also graphically displayed in Figure 2.6. Bolts are placed through an aluminium bar and placed inside a jig. To prevent adhesive from sticking to the bar, a thin PTFE sheet is placed on top of this bar. Holes are cut into this sheet to fit the screws through. The metal plate with the adherends is aligned on to these screws. Now, shim washers as thick as the required adhesive layer thickness are placed on the bolts. After the adhesive is spread on the samples, the second plate is placed on top. Finally, another bar is placed on top and winged nuts are used to tighten everything together. The assembly can be loosened from the jig and cured in an oven. A schematic view of this assembly can be seen in Figure 2.7.

Although this method makes use of some special parts, it is still relatively simple to produce and to work with. The jig that goes into the oven, only consist of a standard extruded bar with 5 holes drilled into it, some screws and washers. This means that it is easy and not expensive to have many parts made so that multiple assemblies can be produced and cured at the same time.

Also, this method is a bit faster than the previous method, as four samples can be prepared at once. It does, however, take some time to cut the PTFE sheets to the right dimensions, as they often cannot be reused.

There are also disadvantages. First, it was hard to remove the assembly out of the jig. This is because the system is heavily over constrained, with 7 places where the bar is aligned with the jig. This was easily solved by enlarging the holes in the aluminium bar. Also, it is hard to cut the taps in between the samples. The slit in between the samples is 1 mm, exactly as thick as a cutting blade of a Dremel. If the Dremel is not aligned exactly, the cutting blade with become stuck and break. This makes cutting the samples quite a hard task to do. The final problem is the accuracy of the adhesive layer thickness. This will be discussed in the next section.

Adhesive layer thickness tolerance

The main goal of the finger samples setup is to produce samples without wires or other discontinuities in the adhesive. The major advantage of using wires, is the very high accuracy of the adhesive layer thickness that can be reached. It is important that a new setup must also reach an accurate adhesive layer thickness.

In order to test this with the finger samples setup, a few samples have been analysed. This is done by making 3 cuts through the overlap area of the samples. The strips are embedded in epoxy, polished and analysed under a microscope, see Figure 2.8. On each cut, 3 thickness measurements are executed, so a total of 9 measurements are done. Because the measurement points are spread evenly across the overlap area, it is possible to see any skews in the adhesive layer thickness of the samples.

Looking to Table 2.1 and Figure 2.9, 6 samples with 3 different adhesive layer thicknesses can be seen. The measured values are not consistent with the aimed thickness. There is no visible correlation between the aimed thickness and the measured thickness. The standard deviation is also high, with an average deviation of 48% of the mean measured value. Looking to Figure 2.9, it can be seen that there is a large spread of thickness within each sample. Also, clear skews in the thicknesses can be seen. The direction of the skew depends per sample.

Furthermore, the thickness is mostly smaller than the set thickness, and rarely larger. It seems that the samples are somehow pushed together. Looking to the design, it strikes that there is a large distance of more than 10 cm of unsupported material between the holes for the washers and the overlap area, see Figure 2.10. Testing it out in practice, it requires only a little bit of force to move the end of the sample whilst it is clamped into the jig. A back of the envelope calculation assuming a cantilever beam showed that a force of 1 N on the end of the sample already results in at least 54 µm. It is assumed that this lack of stiffness plays an important part in explaining the inaccurate adhesive layer thicknesses.



(a) The jig with a luminium bar and bolt and winged nuts.



(b) The bar with bolts placed in the jig.



(c) PTFE sheet and first plate placed on the jig.



(e) Adhesive spread over the sample with a wooden spatula.



(d) Shim washer being placed over all bolts.



(f) Top plate and bar placed. The winged nuts are tightened to compress the package together.

Figure 2.6: Production method of the samples using the finger samples

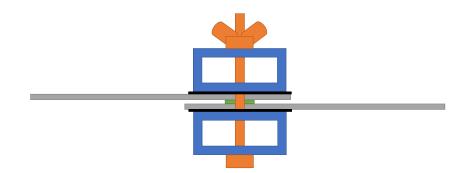


Figure 2.7: Schematic cross section view of the assembly that is put in the oven. The blue rectangles are the aluminium bars. The black layers are the PTFE sheets, preventing adhesive sticking on the aluminium bars. The adherends are grey. The precision shim washer is green.

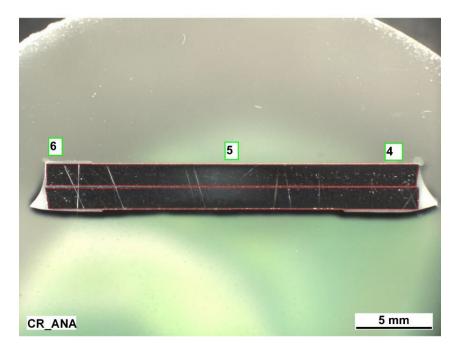


Figure 2.8: Cross section of a finger sample as seen under a microscope. The numbers indicate where measurements are taken. The adhesive layer thickness is greater on the left than on the right.

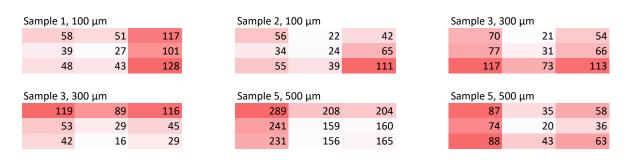


Figure 2.9: Adhesive layer thickness measurements of the finger samples. The redder the colour, the larger the adhesive layer thickness.

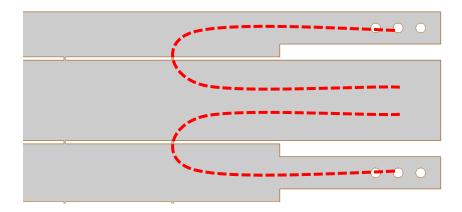


Figure 2.10: Graphical indication of the total length of unsupported material.

Other causes may also explain the inaccurate results. Some small practical studies have been executed on these causes and other causes possibly explaining the inaccurate results of the adhesive layer thickness. They will be discussed here in arbitrary order:

• The steel sheets are not completely flat.

It was found out that the steel sheets can be easily bend along the slots in between the samples. The bending persists if the sample is clamped in. The plates should thus be handled carefully in order not to bend them. Increasing the length of the tabs holding the samples together, will decrease the change of bending the plates. However, cutting the samples will become much more tedious to do.

• The samples are bend under their own weight.

Some back of the envelope calculations, assuming a cantilever beam with a uniformly distributed force, showed that this only accounts for a displacement in the order of $1 \,\mu$ m. Other mechanisms have larger effects.

• Non-uniform thickness of the steel plates.

The edges of the laser cut samples were quite rough. Analysing the cross section of the samples, shows that the thickness around the edges is up to 0.1 mm thicker than in the middle. However, looking to the results in Figure 2.9, the adhesive layer thickness is generally greater on the outside than on the inside. So, although the non-uniform thickness is not beneficial for the accuracy, this does not explain the bad accuracy of the adhesive layer thickness.

• Shrinking adhesive during curing.

For an epoxy base adhesive, as used in the tested samples, a shrinkage of 2.3% will occur during curing of the adhesive. Also, the thermal contraction when cooling down the sample will add another 0.5%. Combined, this only explains a deviation of $14\,\mu\text{m}$ on an adhesive layer thickness of $0.5\,\text{mm}$. This is thus also not the main reason for the inaccurate results.

• Thickness tolerance precision shim washers.

According to DIN 988, which defines the precision shim washers, a tolerance of $30 \,\mu\text{m}$ for the 0.1 mm thick washer and $50 \,\mu\text{m}$ for the 0.3 and 0.5 mm washer is allowable. Some washers have been measured, and they all meet this norm. Mainly for the 0.1 mm adhesive layer thickness, the washer itself can play an important role in the accuracy of this layer thickness. Using these washers to set the adhesive layer thickness is not ideal.

• Positioning

The way the jig is designed, makes it hard to guaranty that the plates are placed parallel to each other. Because the plates are only fixed at 5 points on one line, the plates may rotate along this line, see Figure 2.11. This mechanism can explain some of the found skews in adhesive layer thickness.

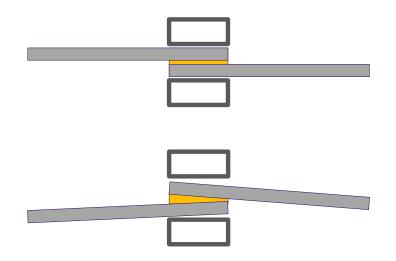


Figure 2.11: Possible positioning error with the finger samples. Diagram by Philipp Weißgraeber.

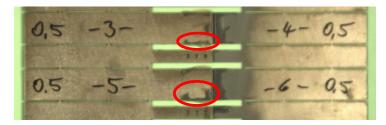


Figure 2.12: Adhesive on the outside of the samples, possibly pushing the adherends to each other.

• Debris between holder and samples.

The aluminium bar is not cleaned between different sets of samples. In Figure 2.6b it can be seen that there is some cured adhesive from the previous sample still left on the bar. These will push the samples inwards. Besides this, also the TPFE sheets were reused with still some adhesive on it. Adding to this, PTFE is a soft material, which can act as a spring between the aluminium bars and the samples. It is expected that this is the main contributor to the inaccurate adhesive layer thickness.

• Capillary action of the adhesive.

With many samples, a small adhesive layer on the top and bottom of the sample is formed, see Figure 2.12. This layer might be formed during curing, where the liquid adhesive creeps in between the sample and PTFE foil. It is not sure if this effect pushes the two adherends to each other, decreasing the adhesive layer thickness, or if the adhesive flows in to the void that is already formed due to other effects.

2.3 New concepts

Using the knowledge obtained in the previous section, new concepts to produce single lap joints are compared. First, requirements of the new setup are set up. After this, some concepts are compared with the requirements to choose the best concept. These concept with their advantages and disadvantages will be discussed in this section.

2.3.1 Requirements

The main goal of the new setup is to produce single lap joint samples with accurately controllable overlap and adhesive layer thickness. To meet this goal, different requirements for the new jig are set up:

• The setup should fit at least 5 samples for statistically significance, with:

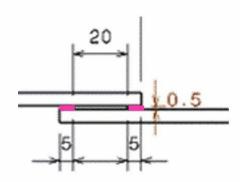


Figure 2.13: The idea of Stuparu et al. [11] The pink lines indicate where the wax is dispensed.

- Width adherend: $25\,\mathrm{mm}$
- Length adherend: $100\,\mathrm{mm}$
- Thickness adherend: 2 mm, with option for 3 mm
- Material adherend: Any material, mostly aluminium
- Overlap length: $12.5\,\mathrm{mm}$ and $25\,\mathrm{mm};$ accurate to $0.1\,\mathrm{mm}$
- Adhesive layer thickness $0.2\,\mathrm{mm},\,0.5\,\mathrm{mm}$ and $0.8\,\mathrm{mm}$
- Accuracy adhesive layer thickness within a few hundreds of a millimetre
- Easy to use
- Quick to use
 - Fast to heat up and cool down
 - Not much preparation needed in between batches
- Adhesives should not stick to the jig
- $\bullet\,$ Heat resistant to at least 150 $^{\circ}\mathrm{C}$

Preferred options:

- Easy to produce
- Possibility for other samples, like the thick adherent shear test (subsection 3.4.1)
- Control adhesive fillet shape and size (subsection 3.3.4)

2.3.2 Literature study

As single lap joints are often used in researches, different methods of preparing these samples are developed. Some other interesting methods that have been found are discussed here.

First, L.A. Stuparu et al. [11] screen-printed wax on two outside edges of the overlap area, see Figure 2.13. When the samples are compressed on to each other with the wax in between, the excess adhesive is forced out until the right thickness is achieved. The thickness of the screen determined the thickness of the wax line. Although this method can reach good accuracy, it cannot be used for heat curing adhesives as the wax will melt away.

The KU Leuven designed a jig which used an aluminium plate with cut out slots, see Figure 2.14. 3D-printed spacers were used to accurately position and clamp the adherends in place. Because the 3D-printed parts are easy and cheap to produce, it is easy to alter the geometry of these parts to make samples with different overlap lengths, adhesive layer thicknesses and adhesive fillets. Performing some research on different 3D-printing materials, no cheap materials and processes are available that can withstand a temperature of 150 °C. Also, the layer height of a common 3D-printer is not small enough to reach an accurate adhesive layer thickness. This makes this method not ideal for this purpose.

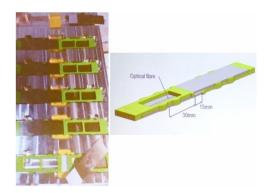


Figure 2.14: The method proposed by the KU Leuven on the EURADH 2018. The green parts are 3D-printed.

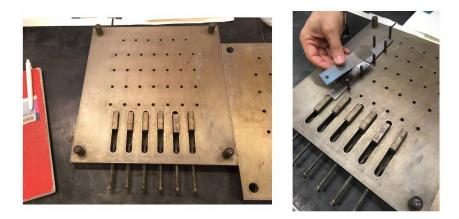


Figure 2.15: The jig designed and used by Da Silva. Photos made by Philipp Weißgraeber.

Arenas et al. [2] used a jig with two elevated levels. The bottom adherend is laid on the bottom level and after applying the adhesive, the top adherend is placed on top. By changing the height difference between the bottom and top levels, the adhesive layer thickness can be set.

Da Silva et al. expanded this method by adding alignment pins and a lid to the jig to fix the samples in all directions, see Figure 2.15. This jig and method are explained in his book [5]. The jig is from steel. Pins can be placed into drilled holes in the jig to create cavities to lock 6 samples in place. Four large alignment pins are placed on the outside to align the top lid to the bottom lid. On the bottom, 6 slots with sliding blocks can be used to lock the samples in the lengthwise direction. These blocks can be positioned by turning the 6 screws that can be seen sticking out of the bottom in Figure 2.15.

The raised platform is now replaced with a detachable shim with the correct thickness and length. By having different set of steel shims, different overlap lengths and adhesive layer thicknesses can be made. By changing the shape of the edge of the shim, the fillet shape can also be altered as is shown in Figure 2.16. The jig is placed in a heat press to press all the samples together and quickly heat up the jig. This concept looks promising and works well according to Da Silva. This method meets most of the requirements or can adapted to meet them. Therefore, the final concept follows this concept closely. Next section explains how the jig and its components are designed.

2.4 Final concept

From the original design, only pictures are available. The first step of the design process is to backwards engineer the original jig and alter the original design and dimensions to meet the requirements and fit within the available heat press.

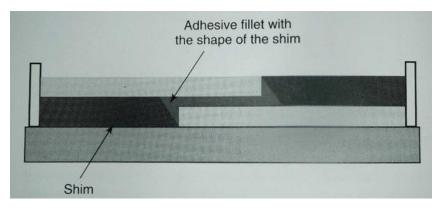


Figure 2.16: Side view of the built up of the jig. The light parts are the adherends, the dark parts are the shims. The grey part in the middle is the adhesive. [5]

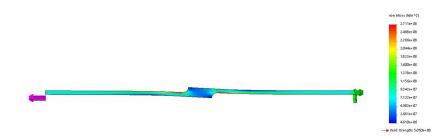


Figure 2.17: Result of the SolidWorks simulation. The bending of the adherends can be clearly seen.

There are two additional requirements set up for this specific concept. First, because this jig must be used in conjunction with a heat press, the maximum size is constricted to the size of the heat press. In the clean room, only one heat press was available with an area of 250 mm by 250 mm. To have some play, the maximum size is chosen to be 235 mm by 235 mm. Second, because the jig is used at elevated temperatures, thermal expansions is important. In this case, most of the adherends will be made from aluminium. If the jig is made from steel, the differences in thermal expansion can lead to high thermal stresses or lead to the samples getting stuck in the jig. It seemed best to also make this jig out of aluminium instead of steel. An bonus is the reduced weight of the jig.

2.4.1 Adherends

Before the final design of the jig can be determined, the dimensions and tolerances of the adherends must be chosen. The adherends are based on DIN EN 1465, a norm for single lap joints. They are 100 mm long, 25 mm wide and for this set of experiments 2 mm thick. A tough aluminium must be used, as the adherend may not plastically deform during the tensile test. The toughest aluminium available in 2 mm sheets at the supplier was 7075-T6, with a yield strength of above 430 MPa. Performing a simple stress simulation in SolidWorks, shows that the internal forces inside the adherends will remain below the yield strength with a safety factor of 2, see Figure 2.17.

It is important that these adherend have an accurate thickness and flatness. A bent sample might lead to an inaccurate adhesive layer thickness. First, a flatness and thickness tolerance of 0.01 mm was put on the adherend. This was, however, hard to produce as this meant that each sample must be ground to the right thickness. This makes the adherend expensive for a single-use consumable.

After feedback from the metal working companies, the thickness tolerance is lifted to ± 0.09 mm. This is the normal thickness tolerance of the aluminium sheet, according to NEN-EN 485-4. This means no further processing of the thickness is required. With some samples, the thickness is measured and they all easily fit in between the tolerance boundaries.



Figure 2.18: The first jig with the bottom and top part.

Because the samples may not be bent during cutting, the cutting process is important. Water jet cutting or punching may bend the samples. Laser cutting is therefore chosen as best option, with an added requirement that any burrs that might have formed during production must be removed. The tolerances on the cutting lines are a tenth of a millimetre, as this is the best tolerance that can quite easily be achieved with laser cutting without much struggles. On one end, a hole is cut out to be able to hang the samples on a wire or rod during the surface treatments.

2.4.2 Jig

The basic design consists out of two, 15 mm thick aluminium plates. A photo of the two halves can be seen in Figure 2.18. The jig is designed such that most operations can be done in a 3-axis milling machine without having to re-clamp the work piece. The main area of attention were the tolerances on the holes and in between the holes, because this determines the accuracy of the alignment of both adherends and thus the final accuracy of the samples.

For the pins, hardened pins with internal thread according to DIN 7979 are used. Using these off the shelf pins is much cheaper than making custom pins for this project. The variant with internal thread is chosen so a bolt can be turned into them. If a pin gets stuck in the jig with some glue, a bolt can be screwed into the pin which can be used to loosen the pin.

The pins have a diameter of 5 mm with a m6 tolerance. This means that for a normal H7 hole, it is a tight fit that requires some force to mount the pin. However, for the jig, the pins must be easily removable. Also, a yet unknown release agent will be applied on both the jig and the pins. Depending of the thickness of this coating, it might also reduce the diameter of the hole. Because of this, a loose fit is chosen. Following table 2-9 of Roloff/Matek [15], a lower play of 20 μ m is taken. The upper limit is taken to be 100 μ m. Given an m6 shaft, a D11 hole gives a tolerance that fulfils these requirements. Also, this wide tolerance is easy to reach, even without reaming the holes. This save a lot of time, as there are 40 holes that must be drilled. The chosen tolerance results in a play of 0.018 mm to 0.101 mm between the pin and the hole, excluding any release coatings. This tolerance is also chosen for the four 10 mm pins on each corner of the jig.

The adherends must fit snugly in between the pins. The adherends are 25 ± 0.1 mm. To make the adherends always fit in between the pins, the distance between the holes is chosen to be 25.2 ± 0.1 mm. The added tolerance varies thus from 0 mm to 0.4 mm, again excluding a release coating.

The holes on the lid have a larger diameter than the ones in the base plate. In this way, the top lid can be easily placed over the pins. For the small holes, a diameter of 5.8 mm is chosen. For the larger holes, the diameter is 11 mm.

The position of the holes is chosen such that all possible overlap lengths gets enough support from the pins. Also, extra holes are added to make it possible to prepare the shorter TAST samples. With this arrangement, 6 samples can be made in one batch. On the sides of the base plate and lid, 4 M8 threaded holes are made. Here, some handles can be attached to make the 4 kg jig easier to handle.

The slots for the sliding blocks are made 7 mm wide, 1 mm wider than the blocks themselves. The depth of the slots is 10 mm. At the outside edge, a M4 thread is drilled through were some rod end will be used to position the block. The blocks themselves can be cut out of a standard 6 mm by 10 mm wrought iron bar, although the milling company milled them by themselves, probably because they did not have the right material in stock. The height of these blocks is 22 mm.

2.4.3 Shims

To control the overlap, a set of shims must be made for each combination of adhesive layer thickness and overlap length. The thickness of the shims is equal to the thickness of an adherend plus the required adhesive layer thickness. The length is equal to the length of the adherend, minus the length of the required overlap. First, 4 sets are made to make samples with an adhesive layer thickness of 0.2 mm and 0.5 mm and an overlap of 12.5 mm and 25 mm. Each set consists out of 14 pieces, two for each slot in the jig, and two as backup.

The length and width tolerance are set to $0.1 \,\mathrm{mm}$. The thickness tolerance is set to $0.01 \,\mathrm{mm}$, so a 5% deviation is acceptable for the smallest adhesive layer thickness. This accurate tolerance does mean that the shims must be ground to the right thickness, making them quite hard to make. To make the shims more resistant to bending and scratches, they are made from steel instead of aluminium.

2.4.4 Extra holes

After receiving the jig, it was decided that extra holes for the pins were needed to be more flexible with the placement and the dimensions of the samples. These holes are placed in between the existing holes. This is done by the on-campus workshop.

2.4.5 Second iteration

A few changes have been made for the next iteration, see also Figure 2.19. The main disadvantage of the first jig, was the large the large lateral clearance that is possible. In the worst case, a alignment difference of 0.6 mm can occur. This resulted in a poor alignment between the two adherends. As the adherends are already produced with a high accuracy for a laser cutter, only the jig can be improved. First, the distance in between the pins is lowered from $25.2\pm0.1 \text{ mm to } 25.1 \text{ mm}_{-0.0}^{+0.1}$.

Besides this, after a phone call with the manufacturer of the release agent, it became clear that the release coating only adds around $5 \,\mu\text{m}$ of thickness. Also, in practice, it became clear that there was quite some play in the pins. This meant that the tolerances on the holes for the pins could be tightened without problem. The tolerance is decreased from D11 to D8. Now, the maximum added lateral tolerance is 0.388 mm instead of 0.6 mm.

On the lid of the first jig, the tolerance on the four large pins was larger than the tolerance on the small pins. In practice, this resulted that the lid should be aligned with all 40 5 mm pins, whereas the four pins on the outside did not contribute to the alignment at all. It therefore can take some time to align the lid with all the pins. To fix this, the clearance on the large holes were decreased and the clearance on the small holes increased. Now, the lid aligns only on the four large pins. Once these are aligned, the small pins automatically align with their corresponding holes in the lid. This worked well in practice.

Furthermore, there were holes adjacent to the overlap area when the jig is in use. This resulted in adhesive flowing into these holes during curing that could not easily be removed, see Figure 2.20. After using the jig, this adhesive must be removed as good as possible. This takes some time and does scratch up the jig. In this iteration, these holes have been removed as they are not needed for the alignment of the adherends.

The main alteration to the design is the possibility to use the jig without heat press. The pressure is now delivered by six M8 bolts. Two bolts are located at either outside edge and two are located in the middle. The middle row of pins is duplicated to make space for these bolts. As aluminium threads are not strong enough when you fully tighten the bolts, slits are milled in the bottom of the base plate to fit M8 nuts.

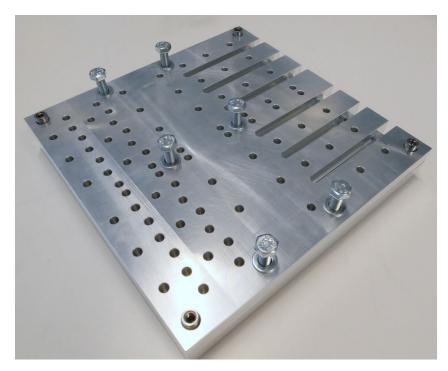


Figure 2.19: The second jig with the six bolts.

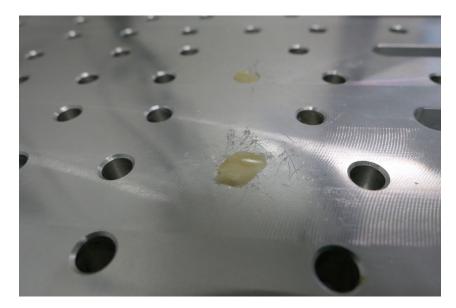


Figure 2.20: Adhesive stuck in one of the holes. Scratches in the jig can be seen from previous removal attempts.

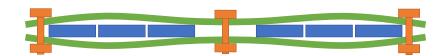


Figure 2.21: Effect of tightening the bolts in the second jig.

The idea of the bolts is that you tighten the bolts to the correct torque to have a certain clamping pressure. Because normal thread an no lubrication is used, the required torque per bold can be approximated according to [15]:

$$M_A = 0.17 \cdot F_{VM} \cdot d \tag{2.1}$$

Here, F_{VM} is the required clamping force and d the nominal diameter. For example, if a total force of 56 kN is required, each bolt should be tightened to 12.8 Nm. This is done using a torque wrench.

Using only 6 bolts to tighten the jig means that not each sample receives the same clamping force. Because the aluminium plates are 15 mm thick, it was assumed that the high stiffness of these plates will ensure that the load is equally spread over all six samples. However, when tightening the bolts, the plates do bend a small bit like in Figure 2.21. This means that the two middle samples of each set of 3 samples were not properly clamped after tightening the bolts and could even be manually moved. This system was thus no good replacement for a heat press.

Solutions of solving this problem were thought off. This include using glue clamps or adding extra stiffness members. These are possible, but it is nicer to have a new revision which get rid of this problem. Therefore, a third iteration of the jig is designed and ordered. The second jig can of course still be used in conjunction with a heat press.

2.4.6 Third iteration

In the next iteration, the six M8 screws are replaced with more smaller screws to spread the load evenly across each sample. The bolts are strong enough to handle the required torque. However, the aluminium might not be strong enough if the bolts are not turned in enough. Using nuts, like with the second iteration, is also not the best solution because it will take a long time to align all nuts with the corresponding slots. Therefore, threaded inserts are used to tighten the screws directly in the bottom half of the jig.

More details on the final design is omitted in this report due to confidentiality restrictions.

2.5 Release agent

Of course, if the adhesive touches the jig, it will adhere to it. This will probably mean that the jig will be glued shut after the first use. In order to prevent the adhesive from sticking to the jig, a release agent must be used.

An important requisite is that the release agent does not have free silicones. Silicones can greatly affect the bonding strength of a non-silicone adhesive. Therefore, it is important that no tools and surfaces gets contaminated with silicones. At Bosch in Renningen, this is ensured by having a separate room with its own equipment were silicones are used. Equipment used there, may not leave the room to be used somewhere else.

Most release agents are silicone based. As there is no proper heat press in the silicone room, it is not an option to use release agents with free silicones on the jig. Multiple release agents have been compared, but only one did not mention it was silicone based. This release agent, Marbocote 227CEE [9], is normally used to release carbon and glass fibre composites from their mould. Based on good previous experiences with releasing the car body of a hydrogen car of Green Team Twente, this product is chosen.

Before applying the release agent, the jig must be cleaned. The official description prescribes to use the Marbocote Mould Cleaner [10]. This is a chemical cocktail of different hydrocarbon solvents. It takes around 3 wipes to clean the jig. Cotton swabs are used to clean the holes in the jig. Because the vapours of this liquid are quite unhealthy to breath, the process must be done in a fume hood. To reduce the exposure to these vapours, the parts are first cleaned with acetone with a final cleaning wipe with the Mould Cleaner. The release agent must be applied in multiple wipe-on layers. For the first time, 4 to 5 layers are required. As each layer should cure for 15 minutes, it is quite a lengthy process. However, the release agent is quite durable. Around 6 releases are possible with the single lap joint jig and 3 releases for the bulk mould. After this, only one new layer needs to be applied. This means it does not take much time to maintain the coating.

After a call with Marbocote, it became clear that the release agent did contain silicones. However, the concentrations of free silicones is generally low, so no contamination problems should occur. Also, one layer of the release agent only adds around $1 \,\mu\text{m}$ of thickness. This means that the tolerance on the holes in the jig will not change that much, also making it possible to reduce the tolerance of the holes.

2.5.1 Further improvements

Still, after three iterations, some improvements are possible. The worst property of this system is that the excess adhesive flows out of the sides of the samples. This must be removed manually which takes time and might influence the strength of the joint. Having a rim or mould around the overlap area that controls the flow of adhesive will help reducing the production time and possible increase the accuracy of the samples.

The second main disadvantage of using these jigs is that only 6 samples can be cured at once, whereas with the old method with spacer wires this limit does not exist. Giving the concept with the jig, there are two methods to work around this problem. First, a larger jig can be made that can accommodate more than 6 samples. However, this jig will not fit any more in the heat press available in the clean room. The jig therefore can only be clamped together using bolts and heated in an oven.

Second, two or more jigs can be used at the same time. It was found out that having 3 jigs is optimal. In this case, 1 jig is curing in the heat press, 1 is cooling down and 1 is being glued. Cycling the jigs through these steps, a new batch can be made around every 45 minutes, assuming a curing time of 30 minutes.

A final disadvantage with using the jig without heat press is that an oven is needed. Also, it takes quite a long time to heat up the jig using forced air convection. This can be solved by incorporating heating elements, like stick-on heating mats, on either side of the jig. A simple off the shelf temperature controller can be used to set and control the temperature. This eliminates the need of an oven and will heat up the jig faster.

Chapter 3

Characterisation Experiments

3.1 Description

An department within Bosch asked CR/APP to develop a method to identify the material criteria that can be used in the assessment of brittle failure in adhesive joints. Unlike with ductile material failure, were the average stress in a material can be used to describe the material behaviour, brittle failure depends on local stress concentrations. The parameters used to predict these concentrations and failure mode cannot directly read of the test results. Instead, multiple tests with different variables must be executed. The data of these experiments can be used to fit and compare multiple fracture models and subsequently find the parameters that describe the failure properties the best.

In this case, an one-component, heat curing epoxy adhesive is tested. This adhesive is commonly used to mount electronic parts like relays and sensors. At room temperature, this adhesive has the viscosity comparable with warm Nutella and is also quite stringy. The adhesive must be cured either at $120 \,^{\circ}$ C for 1 hour or $150 \,^{\circ}$ C for half an hour.

To determine the fracture parameters, six different configurations are tested: two overlap lengths of 12.5 mm and 25 mm and adhesive layer thicknesses of 0.2 mm, 0.5 mm and 0.8 mm.

Besides the six experiments, other comparative experiments have been executed, namely the effect of a spacer wire, curing method and the influence of the fillet shape. For all these experiments, the samples are made using the same procedure. To start, this procedure will be explained in the next section.

3.2 Preparation and Testing Procedure

3.2.1 Etching

Before the adherends can be glued together, the surface needs to be prepared. The best surface preparation will differ based on the adherend material and the adhesive used. In this case, the aluminium adherends are being etched. The procedure can be graphically seen in Figure 3.1.

To start, a etching solution is made and heated to the right temperature. In the meantime, the adherends are hung from a threaded rod with a hook on either side. Thick washers ensure there is enough space between each adherend for the etchant to flow. The adherends are then hung in a square beaker with acetone and cleaned in an ultrasonic bath for 5 minutes. The samples are dried an pre heated in an oven for at least an hour.

After this, the adherends are hung in the pre-heated etchant for the right amount of time. After this, the adherends are rinsed in multiple water baths to ensure there is no etchant left on the surface. The adherends are dried off in an oven before packaged and stored in a desiccator.

In total, around 150 adherends can be etched in one batch. If more is needed, a fresh batch of etching solutions is needed. One person can prepare around 2 batches in one, long working day.



(a) Two batches with adherends hanging from the threaded rods.



(b) Adherends cleaning in acetone in ultrasonic bath.



(c) Adherends drying in the oven



(d) The water baths used to rinse the samples after etching.



(e) Etched and rinsed samples, ready to be dried and stored.

Figure 3.1: Etching procedure of the single lap joint adherends.



(a) Removing adhesive from previous batch using wooden spatula. After this, the pins can be placed in the jig.



(c) The top adherend is carefully placed on top. The top lid of the jig is placed on the adherends.



(b) The adherend and shims are fitted in the jig. The adhesive is placed using a pneumatic adhesive dispenser.



(d) The jig placed in the heat press.

Figure 3.2: Gluing procedure when using the jig with an heat press. Pictures made by Philipp Weiß-graeber.

3.2.2 Gluing

After etching, the samples can now be glued using the jig and heat press discussed in the previous chapter. The final procedure followed for most of the samples will be discussed here. This is the procedure that, after multiple trials, resulted in the fasted workflow and best results. The method will be discussed in a concise way, as a detailed step-by-step guide with pictures has already been made and is available within Bosch. Some pictures of this process can be seen in Figure 3.2.

Before the actual gluing can start, the jig first must be cleaned from any possible adhesive residue from the previous batch. If needed, a new layer of release agent must be applied. Starting from the left (for a right-handed person), the right pins must be placed in the holes. The first adherend and the 2 shims can now be placed in the cavity. Next, the screw and block on the end are tightened. Attention must be paid by not overtightening the bolt, as this might push up the adherend.

The adhesive can now be dispensed. The best way is to use a pneumatic adhesive dispenser. This consists out of a syringe pushed in with a controlled air pressure. The air pressure can be turned on and off with a foot pedal. As the syringe and syringe tip is much smaller than a complete 300 ml cartridge of adhesive, this method is easier and give neater results.

The overlap area is filled by placing multiple lines of adhesive against each other. In this way, the chance of air bubbles getting trapped is minimized. Also, using a spatula to spread the adhesive is unnecessary, further reducing the risk of air bubbles in the adhesive. Furthermore, by applying an excessive amount of adhesive, excess adhesive is pushed out the side, taking the last possible air bubbles with it.

Placing the second adherend is done carefully. First the adherend is placed upright on the bottom shim. The adherend is laid flat slowly, so that no air bubbles get trapped. Now, the adherend is pushed on the shim, forcing excess adhesive out of the joint. This excess is removed with a spatula. This process is repeated for the next five samples.



Figure 3.3: The first generation jig in the heat press.

When each sample is done, they are labelled. Now the top lid of the jig can be placed on the bottom jig. The whole jig can now be placed in a pre-heated heat press. Aluminium foil on the bottom of the heat press is used to catch any adhesive flowing out of the jig.

When using an oven instead of a heat press, the screws on the jig must be tightened after closing the jig. From the middle, the screws are first loosely pre-tightened. After this, the crews can be tightened using a torque wrench. When heated up, the aluminium jig expands harder than the steel screws, increasing the clamping force. Experimental trials have found the optimal tightening torque of the bolts at room temperature so that the optimal clamping pressure is reached when the jig is heated up.

3.2.3 Curing

The curing cycle depends on whether a heat press or an oven is used. Both methods will be discussed in this section.

Heat Press

To save time, the heat press can be preheated to the required temperature, in this case either 120 $^{\circ}$ C or 150 $^{\circ}$ C. When the samples are glued, the jig can be put in the heat press. Some aluminium foil must be placed beneath the jig to catch any adhesive that might flow out of the jig during curing. If everything looks good, the heat press can be closed.

The required pressure must be set. In this case, an average pressure of 2 MPa over the surface of all samples was taken as arbitrary value. In the original planning, the influence of this pressure value on the results was part of the assignment. However, in the end no time there was not enough time to look at this. 2 MPa seemed to give good results, so there was no need to change this.

After closing, the temperature of the heat press of course drops because the jig is still at room temperature. Because of the large contact area between the heat press and the jig, see Figure 3.3, it only takes around 5 minutes for the jig to reach the set temperature. After this, a timer is started to cure the adhesive according to the data sheet. Next, the jig can be cooled down. The fastest way would be to use the water cooling built in the heat press, which can cool down the press and jig within 5 minutes. However, the drainage pipes in the clean room were not suited for the high temperature water and steam, so this was not an option. It was found out that the next fastest method is to open the jig and cool it down in a cold convection oven. The moving air cools down the jig about 2 time faster than cooling it in the open air. After about 40 minutes, the jig is cooled down to about 60 °C and the samples can be removed.

It is best to remove the pins of the jig when the jig is still warm. If the jig is cooled down, the thermal contraction makes it harder to remove the pins and the samples. After taking out, the samples are labelled and stored in a controlled environment.

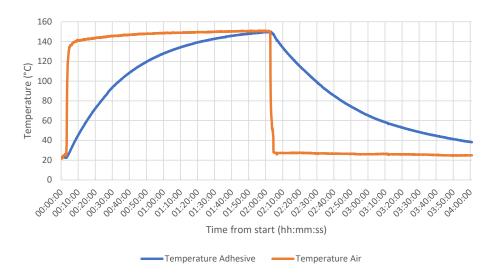


Figure 3.4: Time-temperature graph of the first oven test. The oven temperature is kept at 150 °C. The closed jig is cooled by natural convection.

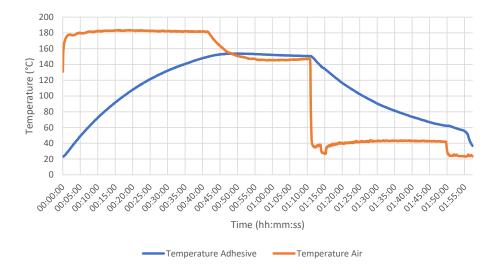


Figure 3.5: Time-temperature graph of the second oven test. The oven is preheated to 180 °C and lowered to 150 °C once the samples reached 150 °C. The jig is opened and cooled by natural convection.

Oven

In the heat press, there is a direct metal-to-metal contact between the jig and the heating element. In a convection oven, the jig must be warmed up by convection with the air. Therefore, it takes longer to heat the jig up. Two experiments have been done to see what the best curing cycle was. The temperature is measured by placing a temperature probe in the adhesive of one of the samples. During the first experiment, the temperature in the oven is set to a constant 150 °C. The result can be seen in Figure 3.4.

It takes about 2 hours before the temperature of the jig also reaches 150 °C. Cooling down is done with the jig closed and put in a cold convection oven. It takes about an hour before the jig is cooled down enough to take the samples out.

In order to try to speed up this cycle, the oven is pre-heated to $180 \,^{\circ}\text{C}$. Once the temperature of the jig and samples reaches $150 \,^{\circ}\text{C}$, the temperature of the oven is lowered to also $150 \,^{\circ}\text{C}$. The result can be seen in Figure 3.5.

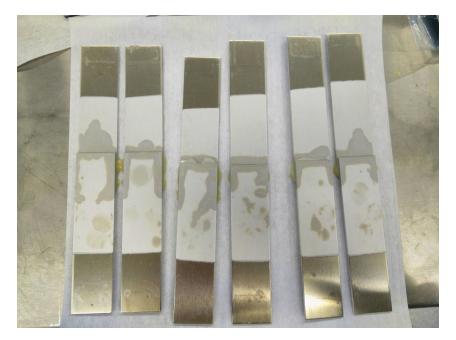


Figure 3.6: Single lap joint samples after curing. Note the excess adhesive still present at the sides of the samples.

Now, it only takes 40 minutes to heat the jig up to 150 °C. The overshoot is also acceptable with 3.8 °C. Opening the jig for cooling speeds the cooling process from 1 hour to 40 minutes. The whole process now takes 2 hours instead of more than 3 hours. Using a heat press is still faster with around 45 minutes for the whole process, if water cooling can be used.

Raising the temperature of the oven even more, might speed up the heating time, but it can also influence the strength of the aluminium jig by additional ageing. If the process must be sped up, having two jigs will be the easiest way, as then every hour a new batch of samples can be made.

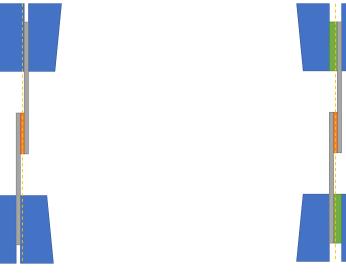
3.2.4 Sanding

During the gluing and curing, not all excess adhesive can be removed. This can be seen in Figure 3.6. This adhesive should be removed manually, as the extra adhesive might make the bond stronger. It is important to remove the material without altering the material properties or introducing cracks in the adhesive. On advice of Da Silva [5], the excess adhesive is removed by filing, as this should not impose too much stresses on the adhesive. Furthermore, filing the samples compensates for any width wise misalignment of the two adherends. After this, the samples are stored in a controlled environment until testing. The filing does mean that the width might be a bit less than the original 25 mm. The results are linearly normalized for this.

3.2.5 Testing

Testing of the single lap joints is done in a universal testing machine. The procedure followed is based on DIN 1465. It is important that the clamps of the testing machine are aligned well. The force should go through the middle of the adhesive layer. Normally, the clamps are placed at a small offset to count for this, see Figure 3.7a. However, if samples with many different adhesive thicknesses are tested, like in this test, constantly changing the alignment of the clamps does cost some time.

To solve this problem, so-called Zwick Spacers are made that can be fit through the holes in the sample, see Figure 3.8. With this, the clamps can stay in line with each other, as can be seen in Figure 3.7b. There is a spacer for each adhesive layer thickness. This method worked well, as it does not take much time to position the spacers in the samples. However, it is quite costly to produce the spacers, so this is something to weigh up against the saved time. Also, during testing the extension between the clamps might vary a bit, because it sometimes takes some force to press the spacer on to the sample. If the strain of a measurement is important, an external extension must be used.



(a) Common method by realigning the clamps (blue) of the testing machine

(b) New Zwick Spacers (green) to align the samples

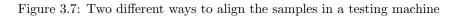




Figure 3.8: A Zwick Spacer besides a centimetre ruler.

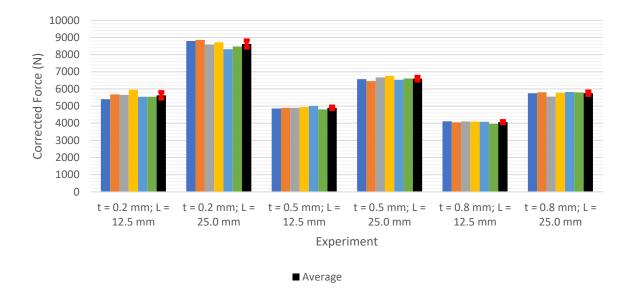


Figure 3.9: Force per sample of samples with different overlap lengths and adhesive layer thicknesses. The black bar indicates the average value of the six tests. The red error bars indicate the standard deviation.

The testing speed is chosen such that the samples break in $65 \,\mathrm{s} \pm 20 \,\mathrm{s}$. For the first sample in a batch, a guess of the speed must be made. It is possible that this test will not meet the time interval. Per sample, the sample width, maximum force, testing time, testing speed and failure mode are noted in the logbook of the sample. The failure mode is analysed according to ISO 10365. For the analysis, only the maximum force, normalized for a width of 25 mm, is used.

3.3 Results

Different tests have been performed using the new setup. All tests are done with the same epoxy adhesive. The tests are executed with different thicknesses and overlap lengths, as the data of this research is used to get the material properties of this adhesive. The effect of the spacer wire and the tolerance of the adhesive layer thickness are also investigated in order to compare the new method with the previously used spacer wires. Also, the influence on curing method, curing temperature and using the oven or heat press, is investigated. As a separate research, the influence of the fillet shape on the strength of the adhesive bond is investigated. The results are also presented in more detail in the AJDM report available within Bosch [14].

3.3.1 Thickness and Overlap

As said before, six different configurations are researched: two different overlap lengths (12.5 mm and 25 mm) and three different adhesive layer thicknesses (0.2 mm, 0.5 mm and 0.8 mm). Of each combination, six samples have been tested to provide statistical significance.

The result of each test can be seen in Figure 3.9 and in Table 3.1. The average values and standard deviation are also plotted in Figure 3.10. Two effects can be clearly seen. First, the larger the adhesive layer thickness, the lower the sustained force. This seems contrary, as stress models indicate that the internal stresses for a thicker adhesive layer thickness are lower than for a thin adhesive layer thickness[3, 4]. Some research points this effect to the strength of the adhesive layer thickness also means there is more elastic energy available for fracture growth [6, 13]. This means that the strength of an adhesive joint cannot be determined with simple strength calculations, but also fracture toughness is important to consider.

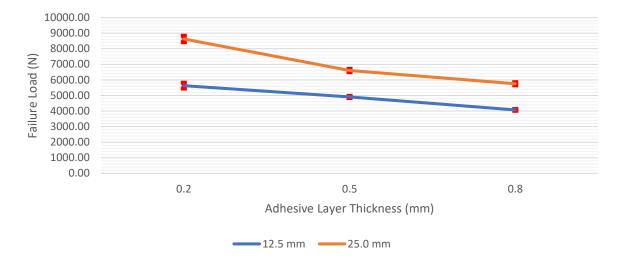


Figure 3.10: Comparison of the mean force per adhesive layer thickness between the $12.5 \,\mathrm{mm}$ and $25 \,\mathrm{mm}$ overlap length. The red error bars indicate the standard deviation.

Table 3.1: Results of the single lap joints with different overlap lengths and adhesive layer thicknesses.

Experiment	Mean failure load (N)	Standard de- viation (N)	Coefficient of variation (%)	Mean failure load per unit area (N/mm2)	Standard deviation (N/mm2)
t = 0.2 mm;	5630	186.7	3.32	18.02	0.60
L = 12.5 mm					
t = 0.2 mm;	8628	206.9	2.40	13.80	0.33
L = 25.0 mm					
t = 0.5 mm;	4903	72.19	1.47	15.69	0.23
L = 12.5 mm					
t = 0.5 mm;	6601	105.1	1.59	10.56	0.17
L = 25.0 mm					
t = 0.8 mm;	4071	57.27	1.41	13.03	0.18
L = 12.5 mm					
t = 0.8 mm;	5753	100.9	1.75	9.20	0.16
$L=25.0~\mathrm{mm}$					

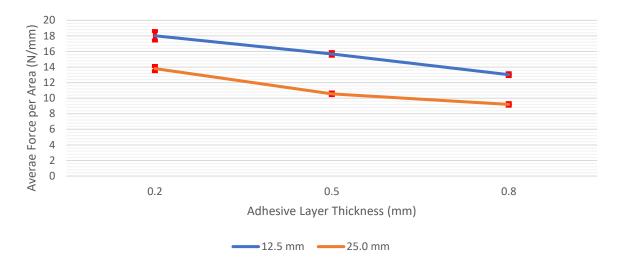


Figure 3.11: Comparison of the average force per area per adhesive layer thickness between the 12.5 mm and 25 mm overlap length.

Experiment	Mean failure load (N)	Standard deviation (N)	Coefficient of variation (%)
t = 0.2 mm; L = 12.5	5630	186.7	3.32
mm t = 0.2 mm; L = 12.5	6018	272.8	4.53
mm; With Wire			
t = 0.5 mm; L = 12.5	4903	72.44	1.48
mm t = 0.5 mm; L = 12.5	5577	271.8	4.87
mm; With Wire			

Table 3.2: Results of the single lap joints with and without wire.

Second, the 25 mm bonds are stronger than the 12.5 mm. However, if this strength is normalized for the overlap length, as can be seen in Figure 3.11, it can be seen that this relation is not linear. Doubling the overlap length does not result in double the strength. This is because the load bearing capacity of the adhesive is not constant but is concentrated at the edges of the overlap. When the overlap length is increased, the adhesive in the middle does not transfer much load between the adherends, but the stress at the edges does not change that much. This can be seen in Figure 3.12.

3.3.2 Adhesive layer thickness tolerance

3.3.3 Spacer Wire

Before the new setup, spacer wires were used to ensure the right adhesive layer thickness, as already shown in Figure 2.3. The diameter of the wire equals the required adhesive layer thickness. However, these wires might act as discontinuities in the adhesive layer. This can result in local stress concentrations and thus act as nucleation points for fractures. On the other hand, the spacer wires are a bit longer than the overlap area, effectively increasing the overlap length of the adhesive.

To look for the differences between samples with and without spacer wires, four sets of samples have been tested. The adhesive thickness is 0.2 mm and 0.5 mm and the overlap is 12.5 mm. The results of each sample can be seen in Figure 3.13. Also, the averages and standard deviation of each experiment is plotted in Figure 3.14 and Table 3.2.

The samples with the spacer wires are stronger than the samples without wire. However, the scatter in between samples is greater for the samples with wire. Also noticeable, during testing clear 'pings' could be heard with most of the samples with the wire before the sample breaks. These 'pings' were probably caused by the spacer wire breaking loose from the adhesive, before the adhesive itself broke.

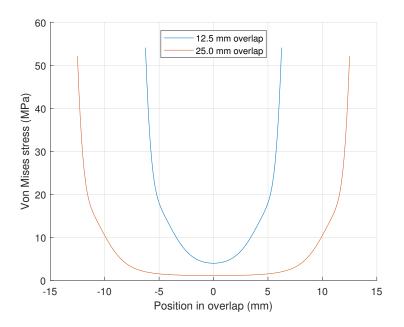


Figure 3.12: Comparison of the Von Mises stress in the adhesive layer between $12.5 \,\mathrm{mm}$ and $25 \,\mathrm{mm}$ overlap length.

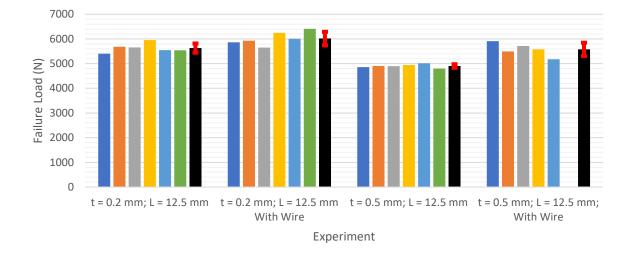


Figure 3.13: Force per sample of samples with and without spacer wire. The black bar indicates the average value of the tests. The red error bars indicate the standard deviation.

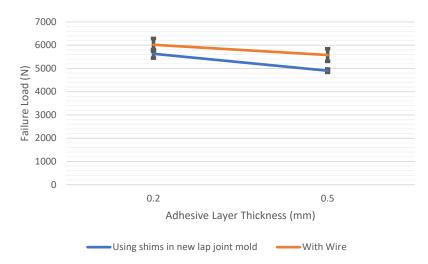


Figure 3.14: Comparison of the mean force per adhesive layer thickness between the samples with and without spacer wire.

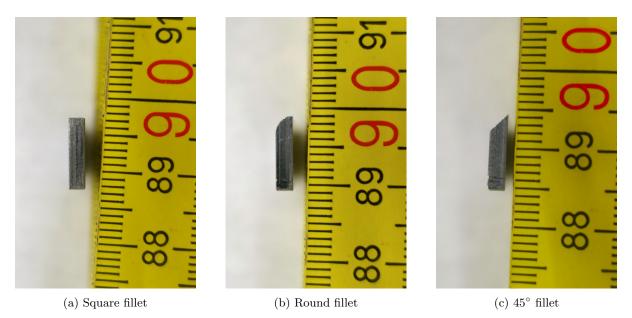


Figure 3.15: The 3 different fillet shims. The scale is in centimetres (small lines in millimetres).

It is also important to consider that the samples with wire are cured in an oven and the other samples were cured in a heat press. The curing cycle in an oven is more gradual and takes longer. This also influences the strength of the adhesive, see subsection 3.3.5.

3.3.4 Fillet

In a single lap joint, the highest stresses are around the edges of the overlap area as can be seen in Figure 4.5. Having a square edge in the adhesive, results in local stress concentrations. Placing a fillet at their edges, will result in smaller local stresses. Therefore, the fillet shape can influence the strength of the joint. In this test, multiple fillet shapes can be compared with samples without fillet.

For this set of experiments, a special set of shims have been made. Each shim is now divided into two parts. The largest part is a normal shim with the appropriate thickness and length. Besides this, there is a set of small interchangeable parts. There are three sets for three fillet shapes, see Figure 3.15. The standard set has square edges, resulting in a square fillet. There are also rounded edges and a 45° edges for these fillet shapes. The adhesive layer thickness cannot be changed and is set to 0.3 mm.

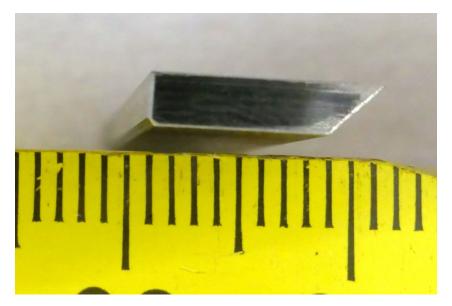


Figure 3.16: Close-up of the small fillet of 0.3 mm on the left of the 45° shim.

Experiment	Mean failure load			,
	(N)	ation (N)	ation $(\%)$	Square fillet
45° Fillet Large	5923	152.6	2.58	8.92
Round Fillet	5616	348.0	6.20	3.28
Large				
Square Fillet	5438	65.81	1.21	-

Table 3.3:	Results o	f th€	e single	lap	joints	with	three	different	fillet	shapes.
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The last two sets have two sizes of fillets to choose from. One size is as large as the adhesive layer thickness plus the adherend thickness. The other side is as large as the adhesive layer thickness only, see Figure 3.16. Due to time restrictions, only the large fillets have been investigated.

The fillets made using this process turned out good. As can be seen in Figure 3.17, the adhesive takes on the shape of the shim accurately. Being able to control the fillet is a large bonus of this setup, as this was not possible with the previous method. A disadvantage is that the adhesive tends to flow between the sample and the fillet. Therefore, the adhesive layer thickness will be less accurate compared to using the normal shims.

Looking to the results of the tests, Figure 3.18 and Table 3.3, it can be seen that the results are close together. There is no significant difference between the square fillet and the round fillet, but this is also because of the large scatter in the results of the round fillet. There is a significant difference between the square and the 45° fillet. The samples with the fillet are almost 9% stronger than the samples without the fillet.

3.3.5 Curing Method

According to the datasheet of the epoxy adhesive, there are two possible curing cycles: 1 hour at 120 °C or half an hour at 150 °C. To see if there is a difference between the two cycles, experiments have been executed. Besides this, the difference between curing in the heat press and in the oven is investigated. For the oven, the two cycles discussed in section 3.2.3 are investigated. The results can be seen in Figure 3.19 and Table 3.4.

The samples cured in the heat press at $150 \,^{\circ}$ C are significantly stronger than the samples cured in the heat press at $120 \,^{\circ}$ C. The difference is $5.7 \,\%$. Possibly, the higher temperature results in a higher degree of cross-linking.

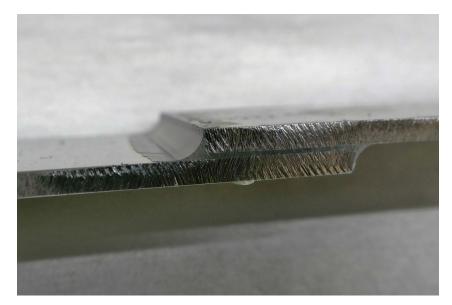


Figure 3.17: The result after using the large round fillet shim. Also note the small layer of adhesive left on the adherend on the left.

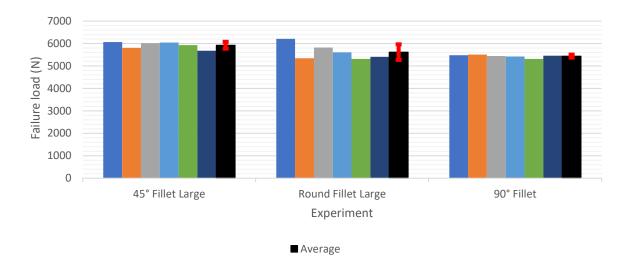


Figure 3.18: Force per sample for three different fillets. The black bar indicates the average of each set. The red error bars indicate the standard deviation.

Experiment	Mean failure load (N)	Standard deviation (N)	Coefficient of variation (%)
Heat press 120 °C	5442	58.60	1.08
Heat press 120 °C	5753	100.9	1.75
Oven 150 °C 2h	6378	150.5	2.36
Oven 180 °C	6419	134.9	2.10

Table 3.4: Results of the single lap joints with 4 different curing conditions.

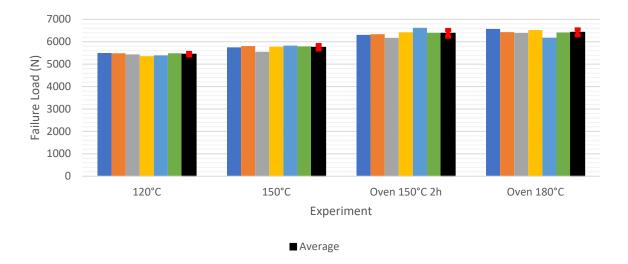


Figure 3.19: Force per sample for three different fillets. The black bar indicates the average of each set. The red error bars indicate the standard deviation.

Besides this, the samples cured in the oven are stronger than both sets of samples cured in the heat press. The difference is around 11% compared with the samples cured in the heat press at 150 °C. The fact that heating and cooling in the oven takes longer, means that the adhesive stays at an elevated temperature longer compared with the heat press. This might also lead to a higher degree of cross-linking, resulting in a stronger bond. In that case, the proposed curing cycles in the data sheet of the adhesive does not result in the strongest bonds. For better results, the adhesive should be cured longer. Also, a difference of up to 11% can be expected when comparing experiments cured with the heat press with experiments cured in an oven.

3.4 Further experiments

3.4.1 Thick Adherent Shear Test

When performing a tensile test on a single lap joint, a combination of shear and normal forces occur in the material. This is because of the bending of the adherend as seen in Figure 2.17. If only the shear strength of an adhesive must be known, a different adherend geometry must be used that does not bend under load. This kind of test are called Thick Adherent Shear Test (TAST). The adherends are thicker, preventing bending as can be seen in Figure 3.20. Also, the adhesive layer thickness and overlap are set by making different samples.

In order to keep the adherends as simple as possible, they are made from standard 25 mm by 12 mm extruded bar of aluminium. On one end, a cut-out is milled according to the overlap length and adhesive layer thickness. A hole is also added to hang the samples from. The hole can also be used to hold the samples during the tensile test.

The TAST adherends can be glued and cured using the same jig as used for the single lap joints. The only difference is the increased thickness, that means that the four large pins should be replaced by longer ones.

In between the samples, spacers are used to prevent adhesive to stick on the ends of the adherends. First, these spacers were cut from a plate of PTFE. However, these spacers were not accurate and could only be used once. Steel spacers with a release coating are designed and made to make them more accurate and durable. Sadly, there was no time to test a full set of TAST specimen.



Figure 3.20: A TAST sample tested in a testing machine. On either side of the overlap area, PTFE spacer can be seen.

Chapter 4

Other Projects

Besides the main project of designing the single lap joint jig and preforming the experiments, some smaller projects have also been addressed parallel to the main projects. These projects are further discussed in this section.

4.1 Bulk mould

The previous researches are executed to determine the strength of an adhesive bond. However, in order to correctly model the behaviour of an adhesive, the material properties of the adhesive itself must also be known. In order test these, so-called bulk specimen is made and tested.

To do so, a plate of pure adhesive is made. Until now, this is done by sandwiching uncured adhesive between two PTFE sheets and pressing it down in the heat press. Brims of the correct thickness control the thickness of the resulting plate of adhesive.

This method has a few disadvantages. First, the thickness of the adhesive plate is not well controlled and is not constant. This is probably caused by the limited stiffness of the PTFE causing it to deform in the heat press. Also, it is hard to produce plates without air bubbles.

After curing, the samples must be cut in the right shape. For tensile tests, most commonly a dog bone shape is used. For flexible adhesives and elastomers, this can be done with a punch with the correct shape. For a brittle adhesive, like the epoxy adhesive, this must be done at an external water jet cutting company. This can take some time. Also, the force applied by the water jet might introduce small cracks in the material, acting as nucleation points for cracks, and therefore influencing the test results. It would be beneficial if a method is designed that can mould the adhesive directly in the right dog bone shape.

4.1.1 Idea

Da Silva [5] developed a method that should produce square plates with constant thickness and without air bubbles. This is done by pouring the adhesive inside a silicone rubber frame. This frame is placed in a steel mould, restricting any movement of the silicone frame. Inside the cavity that the frame forms, the right amount of adhesive is poured so that the cavity is filled. The top lid of the mould is placed and the whole mould can then be placed in a heat press. Pressing on the mould, ensures that the adhesive has a hydrostatic pressure because the silicone frame presses onto the adhesive from the side, see Figure 4.1. This should force out any remaining air bubbles from the adhesive.

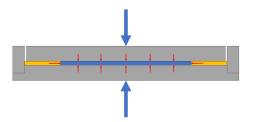


Figure 4.1: Schematic representation of the hydrostatic forces acting by the mould (grey) and the silicone frame (yellow) on the adhesive (blue).



Figure 4.2: The opened bulk mould with the bottom plate, the four sides and a silicone frame. The top lid is not in the picture.

4.1.2 First Design

The design consists out of six parts: a bottom plate, a top plate and four removable sides, see Figure 4.2. The sides are clamped on the bottom plate by four screws. Between the top plate, and the sides there is some extra play for excess adhesive to flow out. The mould is made from steel.

In his book, Da Silva has put the dimensions of the mould they designed. As this mould also fits in the heat press available at Bosch, the dimensions are simply copied from the book. Drawings have been made and sent to the internal workshop at Bosch Renningen, who produced the mould. The mould is treated with the Marbocote release agent. This also prevents the steel from corroding.

For the rubber sheet, different materials have been tested. Some materials could not handle the combination of the temperature and pressure and started to deform. A material that seems to work well are silicone sheets of 50° Shore A hardness from MVQ silicones GmbH. Two thicknesses, 2 mm and 4 mm, are used.

The original design prescribes a square silicone frame with sides of at least 15 mm wide. This results in square sheets of bulk material. In order to skip the step of cutting the dog bone shapes out of these sheets, also some sheets with a dog bone cavity cut out of it are made. If working correctly, this will result in bulk dog bones straight from the mould.

4.1.3 Method and Results

The mould is place on a scale. The right amount of adhesive is measured out and spread over the cavity of the silicone frame. For curing, the time and temperature according to the data sheet of the adhesive is followed. The advised pressure is 2 MPa over the combined surface of the adhesive and silicone frame. In this case, this means a pressure of 35.1 kN.

The first iterations resulted in funny shaped samples because the silicone frame was pushed out in the space between the sides and the top lid. After a new top lid with a tighter tolerance was designed, the samples were much more accurate.

The square shapes have a constant thickness, without any air bubbles. Also, the dog bones did not have any air bubbles. The accuracy was quite well, but there was still a skew in the width of around half a millimetre. This mainly caused by the accuracy of the silicone frame. It is hard to cut out the shapes with a higher accuracy as half a millimetre. A better way to cut the silicone sheet should be investigated. This might make it possible to produce dog bones directly with a high enough accuracy.



Figure 4.3: Two bulk dog bones. The top one is 2 mm thick and the bottom one is 4 mm thick. The discolouring of the bottom sample due to the exothermic reaction during curing can be clearly seen.



(a) Bottom plate for one large bulk sample

(b) Mould set up for three bulk samples

(c) Mould set up for six bulk samples

Figure 4.4: Different configurations of the second bulk mould

The dog bone with a thickness of 4 mm did de-colour a bit compared to the 2 mm. This effect can be seen in Figure 4.3. This is probably because of a too high temperature inside the adhesive, because the curing reaction of an epoxy adhesive is exothermic. Due to the larger thickness, the heat cannot escape as easily out of the adhesive. If this also effects the strength of the adhesive, these thick samples cannot be made with this adhesive.

4.1.4 Second Design

A second iteration of the design has been made by an external company. This design is optimized to use the complete space of the heat press. Different lids and edges can be placed to make one large adhesive plate, or to divide the space in 2, 3 or 6 smaller areas, see Figure 4.4. The smallest area is large enough to produce a small dog bone. This mould is made from aluminium to save weight and thus make it easier to handle the mould. Unfortunately, there was no time to test this mould.

4.2 Matlab tool

In a paper by Weißgraeber et al. [12], a computational efficient method of determining the shear and peel stress distribution inside an adhesive layer is presented. As an attachment, a Matlab script is provided that determines these stresses after manually inputting the geometry, section forces and material properties. This happens within a few hundreds of a second, so the results can be updated almost instantly. The goal of this project is to make a simple to use Matlab app with graphical user interface around this model that anyone without any programming experience can use. This tool can be used to help designing adhesive joints, as changes in the input are almost immediately displayed, making it easy to calculate different configurations.

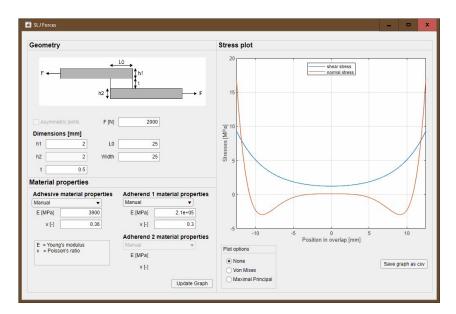


Figure 4.5: Screen shot of the user interface of the program.

4.2.1 Idea

For the first iteration, the program is made so it can be used to determine the stresses of a symmetric single lap joint only. Symmetric means that both adherends are identical. Besides this, isotropic material properties of the adherends is assumed. The model proposed in the paper by Weißgraeber et al. [12] is also capable to model asymmetric and non-isotropic adherends, but this requires another model to determine the section forces. As no easy to implement model could be found, this feature is omitted for now.

The user inputs the geometry of the single lap joint, material properties of both the adherends and adhesive and the loading on the joint. Commonly used materials can be selected from drop-down menus. The program produces a plot with the shear and normal stresses inside the adhesive over the overlap length. Additionally, the Von Mises or Maximal Principal forces can be displayed. The data can also be exported as a csv-file to be used in another program. A screen shot of the program can be seen in Figure 4.5.

4.2.2 Execution

The program is designed in Matlab App Designer. This program makes it possible to design the GUI by simply dragging and dropping different elements to the right place. This makes it quite easy to design the layout of the app, although the program has its limitations and was not completely stable with Matlab 2017b.

Every time an input is changed, the stresses are recalculated. To do so, first the inputs are read and stored in appropriate variables. Warnings will be displayed if the adhesive layer thickness is too similar to the adherend thickness or if the stiffnesses of the adhesive and adherend are similar. In this case, some assumptions made for the model do not hold any more which might lead to inaccurate results.

Using the input, the material stiffness matrices of both adherends are derived. These matrices are similar to the ABD matrices used in classical lamination theory.

After this, the section forces are determined. These section forces are dependent on the elongation and bending of the adherends outside of the overlap area. For any joint, the forces can be derived using a FEM analysis. However, for multiple adhesive joints, like the single lap joint, there are models that effectively determine these forces. For symmetric single lap joint, a model developed by Goland and Reissner is often used because of its accuracy and simplicity [8]. The downside is that this model only works for symmetric bonds. Other models that can work with non-symmetric joints and anisotropic materials were also compared [16, 17], but those need to be solved iteratively, making the program slower to work with and thereby defeating the purpose of this program. For now, it is chosen not to implement such model. The program is prepared to handle nonsymmetric configurations, only one line of code needs to be changed to enable this.



Figure 4.6: Cavity bubbles having formed after cooling down.

The Goland-Reissner model outputs the normal and shear stresses and moments on the ends of each adherend. These forces, combined with the material stiffness matrices, can be used as an input for the model by Weißgraeber et al. [12]. The model outputs the shear and normal forces in the adhesive over the overlap length. From this, the Von Mises and maximum and minimum principal stresses are calculated and plotted.

The export option shows a pop-up safe window where the path and file name can be changed. The file is saved as a csv-file using the *writetable* command in Matlab.

4.2.3 Possible Extensions

As the program is very basic, the code is commented extensively to make it easier for other people to change the code. As already discussed, being possible to model asymmetric single lap joints or anisotropic adherend material, like composites, would make this program much more versatile. For this, an alternative for the Goland and Reissner model must be found.

Besides single lap joints, there are also other models that determine the section forces in other situations. The model by Weißgraeber et al. [12] has been confirmed to work on glued on reinforcement patches, T-joints and double lap joints. By implementing multiple models to determine the section forces, this program can be used on many kinds of joints.

Furthermore, one major disadvantage of this program is that is runs within Matlab. It is also possible to package this app such it can be run on computers without Matlab license, but this will install a stripped-down version of Matlab in the background, making it a large and slow program to run. Ideally, this app should be able to run without Matlab, but this probably means the app should be re-coded in another programming language like Phyton.

4.3 Silicone Adhesive Cavitation Experiments

Besides epoxy adhesives, also some tests with a ductile silicone adhesive are performed. In this case, this is also a thermally curable, one component adhesive. Curing temperatures lay in between 100 $^{\circ}$ C to 150 $^{\circ}$ C. When cooling down after curing, the adhesive wants to thermally contract. If the adhesive layer is not able to contract, so-called cavitation bubbles appear as can be seen in Figure 4.6. The mechanisms behind the formation of these bubbles is not well understood. Therefore, some experiments in this direction are executed. The two largest experiments will be discussed in this report.

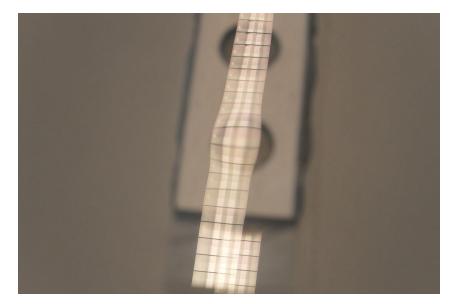


Figure 4.7: The deformation of the glass surface can be seen in the distortion in the reflection.

4.3.1 Internal pressure

The thermal contraction of the adhesive causes an internal pressure. In order to have an indication how strong this pressure is, an experiment is developed. In this, an aluminium sample is sandwiched between two microscope slides. In the aluminium sample,2 12 mm holes are drilled. When cooling down, the contracting adhesive pulls the glass inwards. This can be seen in Figure 4.7. By measuring the deflection of the glass, an indication of the internal pressure can be calculated.

Assuming a fixed boundary, the following equation can be used:

$$q = -\frac{64y_c D}{a^4} \tag{4.1}$$

Here, q is the internal pressure in MPa, y_c the deflection in the middle of the hole in mm, a the diameter of the hole in mm and D an material depended parameter in N mm. For soda-lime glass, this number is 6130 N mm.

The deflection is measured with a stylus profilometer, as optical methods are not possible due to the transparent glass. The deflection is in between 0.027 mm to 0.031 mm. Assuming that the glass is rigidly supported at the boundary of the hole, this would indicate a pressure of around 4.37 MPa. However, a small deviation in the diameter of the hole results in a large difference in the pressure as the diameter is raised to the power 4. This makes the measurements not that accurate.

4.3.2 dT Experiments

The second test is designed to determine the evolvement of the bubbles after a certain temperature drop. As the adhesive cools down after curing, the thermal contraction increases with the temperature drop. Therefore, the bubbles will grow with degreasing temperatures. The goal is to produce a video which displays the samples and the temperature, so the evolvement of the bubbles can be seen and compared to the temperature drop.

The tests will be done in a climate chamber. This is in fact an oven with build in air conditioning so it can accurately control the cooling rate. First, the samples are cured for 2 hours at 130 °C. After this, the oven is cooled down with $0.5 \,^{\circ}\text{C}\,\text{min}^{-1}$, so the temperature reaches room temperature after 3.5 hours. This slow rate is chosen to ensure a constant temperature throughout the samples.

In an access hole on the side of the climate chamber, a plug with a glass window has been made. Through this, a camera can be positioned to look inside the chamber. A frame is build using Bosch Rexroth aluminium profiles to be able to position the samples in front of the camera. This frame can be seen in Figure 4.8. Using LED-strips, a light has been made to illuminate the samples to make the bubbles more visible. A temperature logger measures both the air and the sample temperature.

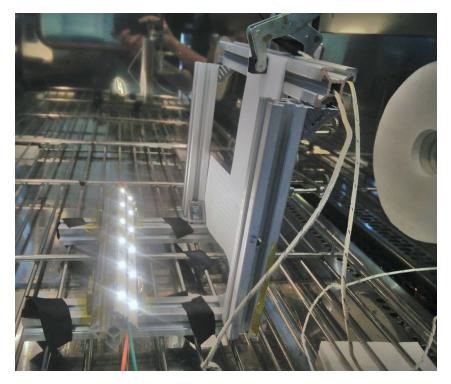
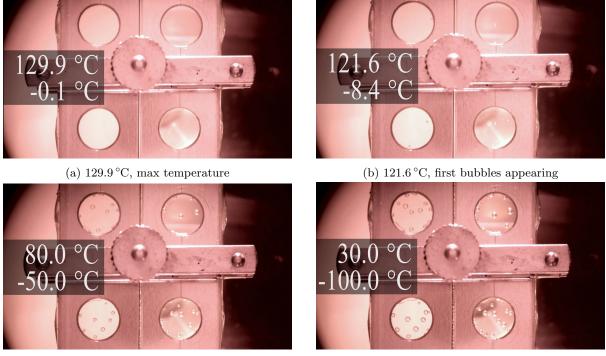


Figure 4.8: The frame inside the climate chamber. The samples are hung from the horizontal beam behind the white PTFE sheet. The camera looks through the hole on t he left.

The sample is prepared in the silicon room in the clean room and transferred to the climate chamber. A full cycle takes about 6 hours, excluding preparation and cleaning, so this process does result in long working days. In total three of these experiments were done before the results were satisfactory.

The camera was set up to take a picture every minute. After the test, a video was composed using Adobe AfterEffects. The temperature was put in AfterEffects every 5 °C using key frames and linearly interpolated for each frame in between the key frames by using a simple script. At the start, extra key frames were added to increase the accuracy.

The first bubbles formed after cooling down 6 $^{\circ}$ C. With decreasing temperature, more bubbles are forming, and existing bubbles grow larger. This effect can be seen in Figure 4.9.



(c) 80° C

(d) 30 °C

Figure 4.9: Results of the third test in the climate chamber. The top temperature is the actual temperature of the samples, the bottom one the dT with respect to 130 °C.

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Appendix A

Robert Bosch GmbH

Many consumers know Bosch from their power tools like cordless drills or appliances like fridges. But although this is an important part within Bosch, it only accounts for a quarter of the total sales. Within Bosch, Mobility Solutions is the largest department. Bosch makes many parts for vehicles like sensors, engine parts, motor control units, displays and also provides many services. Besides this, Bosch has also departments in Industrial Technology and Energy and Building Technology. Furthermore, Bosch focuses on being a leading IoT company. In 2018, Bosch had a total sales of 77.9 billion Euros and had 410 000 employees, from which 69 500 people work on Research and Development [1].

Bosch is founded in 1886 by Robert Bosch in Stuttgart, focussing on innovative car parts. Today, many locations are still located in and around Stuttgart. In 2015, a new campus was opened in Renningen that joined R&D departments around Stuttgart on one campus. The idea is that people with different backgrounds can work together and inspire each other. The site employs 1700 employees, out of which 500 are interns or PhD students. Their goal is not directly to develop products, but to do more fundamental research with other departments within Bosch that do not have the knowledge or facilities themselves.

The CR/APP department is one of the departments located on the campus. They focus mainly on modelling the behaviour of plastics and adhesives. They also help with the material choice and the (re-)design of plastic parts. Typical subjects are to design new plastics made from recycled materials, modelling of the effect of fibre orientations on material properties within injection moulded parts and the conceptual design of adhesive joints and the production system around it. There are around 70 people working inside the department, out of which around 20 are interns or (PhD) students. There is a close collaboration between colleges and an open and informal working climate.



Figure A.1: Aerial picture of the campus in Renningen. Copyright Robert Bosch GmbH.

Appendix B

Reflection

Looking back to the past half year, I am happy that I have made the choice to work at Bosch in Renningen. I specifically liked the working environment and colleges. Everyone was enthusiastic and worked hard, but there is also room for some good conversations at the coffee machine.

Furthermore, the open working environment made it easy for me to ask question or discuss problems with the appropriate people. Having many fellow interns was also nice to have as we went to lunch together and had regular dinners in and around Stuttgart. There are regular meetings with students from other Bosch locations around Stuttgart, with which I for example joined the Oktoberfest.

I found it interesting to learn more in depth how to work with adhesives and plastics. Also learning the basics of fracture mechanics, something that is not taught at my university, was really interesting. It felt special to have the possibility to make many revisions of a concept and thus have the possibility to learn how certain things that seems to work well in theory, might end up working differently in practice.

The campus also has a lot of facilities and workshops. If there was something that needed to be fixed fast, there were places where I could do some gluing, soldering or drilling. On top of this, there is a lot of testing equipment available, making it easy to do different experiments.

I found out that, although almost all people speak English, there still was some language barrier for me. Although my German did improve quite a bit, I was still not able to speak it fluently. However, I am certain that with some more time and practise, I will be able to bring it up to negotiation level. For future work, I think a smaller company would fit me more, as this will offer more freedom in working topics. This would also make it possible to work on more diverse topics, something I would really like.

But these are just small points. In all, I really liked the experience to work for Bosch in Germany. I learned a lot during this internship and really had a great time with my colleagues and fellow students!