Project no.: ***CfP08-AIR-03-02-831882***

Project acronym: **SealedwithoUTaKiss - *SWAK***

Project title:***Non-destructive testing (NDT) of bonded assemblies***

Instrument: ***Cleansky 2 JTI***

Set of samples and structures to be tested

Start date of project: **01 April 2019** Duration: **36 Months**
Deliverable Lead Partner: **GMI**
Contributions made by: **TWI, BRU**

Revision [Draft, Intermediate, **Final**]

Dissemination Level		
PU	Public	X
PP	Restricted to other programme participants (including the Commission Services)	
RE	Restricted to a group specified by the consortium (including the Commission Services)	
CO	Confidential, only for members of the consortium (including the Commission Services)	

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1. Introduction

As detailed in SealedwithoUTaKiss (SWAK) Description of Work (DoW), **WP2** attempts to provide use cases composed of a representative set of coupons and few assembled parts, while proposing innovative surface preparation and control. More specifically:

- Within Task 2.1 GMI will manufacture 7 to 10 different representative samples and coupons fortesting for different sandwich structures as expected in topic. Beside reference samples (definition of surface state which is clean and ready to bond), contaminants (and the combination of contaminants) that are most relevant for aeronautical environment will be identified: residues of contaminants like silicone-based release agents in the bulk material of CFRP components, especially in case of bonded composite repairs. Relevant concentration levels of the chosen contaminants in real aerospace applications will be defined. Different contamination and degradation scenarios will be examined and introduced to the samples to test eligibility and the versatility of the techniques.

Definition of kissing bonds:

Kissing Bonds or “Zero volume de-bonds” can be defined as a type of interfacial defect in which two surfaces within the structure are in intimate contact with no bonding existing between the two surfaces. A similar defect to this is a weak-bond, where there is inadequate bonding between two surfaces. Such defects can occur within monolithic, sandwich, bonded or repaired composites, and can be introduced during manufacture, or as a result of damage or inadequate repair.

Possible criteria of kissing bonds:

- Bonds which fail at less than 20% of their nominal strength (lap shear test)
- Mode of failure must be adhesive in type (i.e. purely at the adherend/adhesive interface)
- Unable to detect using conventional NDT methods (ultrasonic inspection)

The outcome of the study is reported in deliverable ‘D2.1Set of samples and structures to be tested’ and will be further exploited within the following WPs.

2. Materials used

This project focuses on bonded structures made of composite materials that often found in aviation industry. For this purpose, all test samples were fabricated using aeronautical grade materials. The test samples were cut out from composite panels that were provided by Dassault Aviation. All composite panels are consisted of twenty (20) plies of the prepreg HexPly M21/IMA, which is a toughened epoxy resin system supplied with unidirectional carbon fibers. The matrix and fiber properties are presented in figure 1 and table 1. The stacking sequence is illustrated in figure 2.

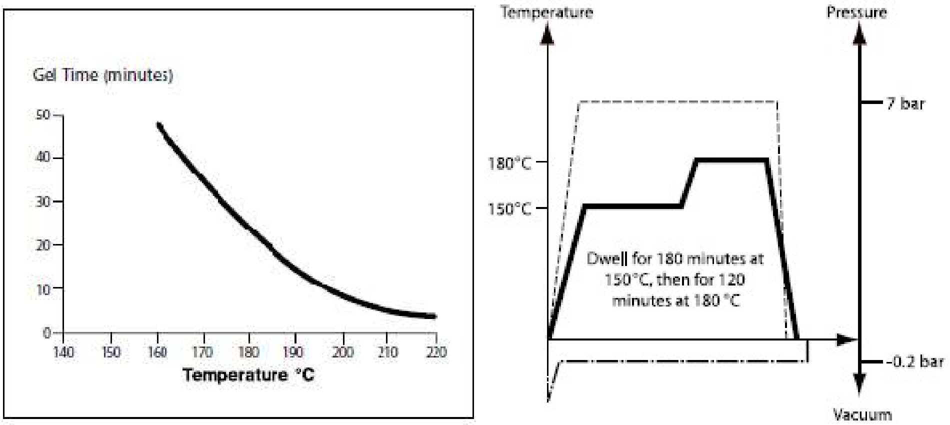


Figure 1. Resin matrix gel time and typical autoclave cure for thickness 15-48 mm

1	M21EV/IMA	0°	
2	M21EV/IMA	+45°	
3	M21EV/IMA	0°	
4	M21EV/IMA	-45°	
5	M21EV/IMA	0°	
6	M21EV/IMA	90°	
7	M21EV/IMA	0°	
8	M21EV/IMA	+45°	
9	M21EV/IMA	0°	
10	M21EV/IMA	-45°	
11	M21EV/IMA	-45°	
12	M21EV/IMA	0°	
13	M21EV/IMA	+45°	
14	M21EV/IMA	0°	
15	M21EV/IMA	90°	
16	M21EV/IMA	0°	
17	M21EV/IMA	-45°	
18	M21EV/IMA	0°	
19	M21EV/IMA	+45°	
20	M21EV/IMA	0°	

Nbr. Of plies	Orientation
10	0°
2	90°
4	+45°
4	-45°

Figure 2. The stacking sequence inside the composite the panels.

Table 1. IMA fiber typical properties

Typical Fiber Properties	SI Units
Tensile Strength (12K)	6,067 MPa
Tensile Modulus (Chord 6000-1000)	297 GPa
Ultimate Elongation at Failure (12K)	1.8%
Density	1.79 g/cm^3
Weight/Length (12K)	0.445 g/m
Approximate Yield (12K)	2.25 m/g
Tow Cross-Sectional Area (12K)	0.25 mm^2
Filament Diameter	5.1 microns
Carbon Content	95.0%
Twist	Never Twisted

3. Preparation of first set of trial test samples

3.1Cutting and surface preparation

At the first stage trial test samples were manufactured in order to establish a reliable method for creating "Kissing Bonds". The initial experiments involve mechanical testing on single lap shear joints. The trial test samples were cut out from the composite panels according to the ISO 4587 standard, as illustrated in figure 3. **In an attempt to prevent damaging the contaminated bond line, the bonding procedure was performed after the cutting of individual joints.**

All composite panels have the same thickness of about 4-4.2 mm. The cutting was done using a table saw equipped with a diamond disk. In a second step the edges of the samples were sanded using dry sand paper of 300 and 600 grit. All samples were cleaned thoroughly using Diestone cleaner. Totally sixty (60) joints and fifty (50) tabs were prepared as illustrated in figure 4.

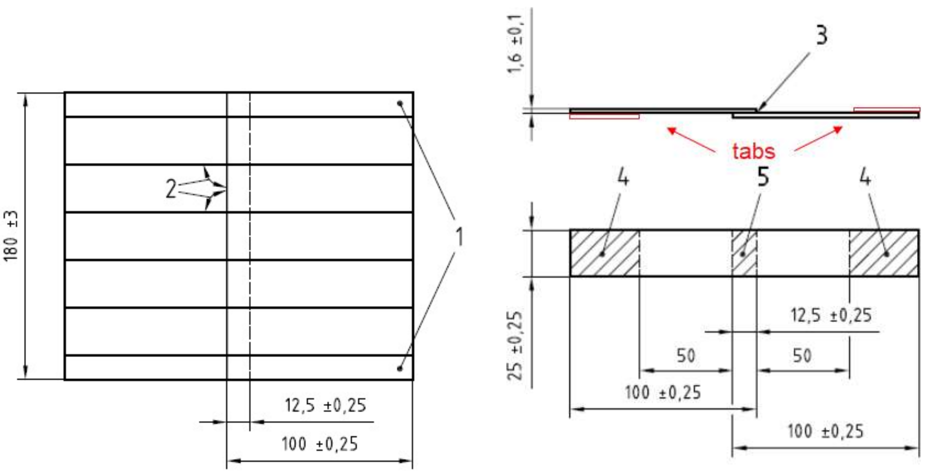


Figure 3. Geometry of the samples as described in ISO 4587 standard

Figure 4. The joints (60) and tabs (50) that were cut from the composite panel

3.2 Adhesive modification/contamination

In all experiments the bonding is done using aeronautical grade adhesive film FM 300M. Besides reference samples four different methods of contamination/modification were tested and evaluated such as:

A. Pre-Cured Adhesive

The basic idea is to pre cure the adhesive film in lower temperature for shorter time than the original cure cycle. This treatment is expected to downgrade the bonding reaction since a significant percentage will been already polymerized. The adhesive film is placed between Teflon plates and cured, as illustrated in figure 5. The pre curing took place at 130 ^{o}C for 30 min while the recommended cure cycle is 177 ^{o}C for 60 min.

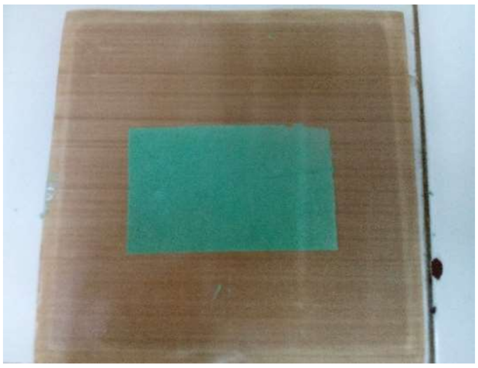


Figure 5. The adhesive film is placed between Teflon plates for pre cure

B. Graphite Powder

The graphite powder is applied on the adhesive film (one side), as illustrated in figure 6. The graphite particles prevent the adhesive to bond to the composite, downgrading the quality of the bond line. At the same time the graphite particles will be difficult to be detected due to fact that they are similar to the adherent joint.

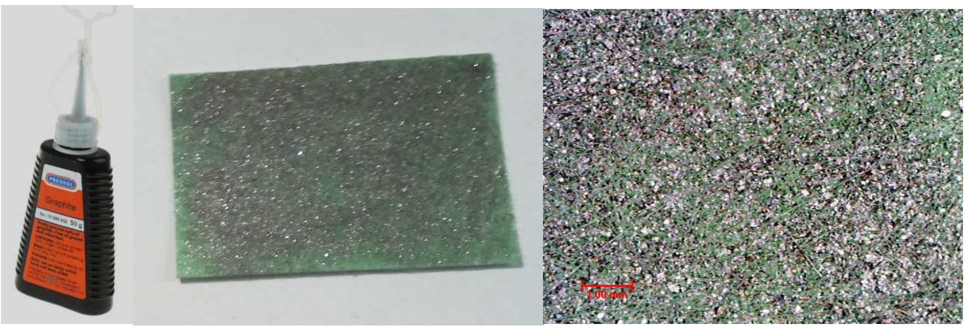


Figure 6. The graphite powder is applied on the adhesive film

C. PTFE Lubricant

In this method PTFE lubricant is applied on the surface of the joint around the bonding area, as illustrated in figure 7. Before bonding, the lubricant is left to dry for 24 hours. It is assumed that the PTFE (Teflon) particles will create a thin layer preventing the adhesive to bond to the adherent joint.

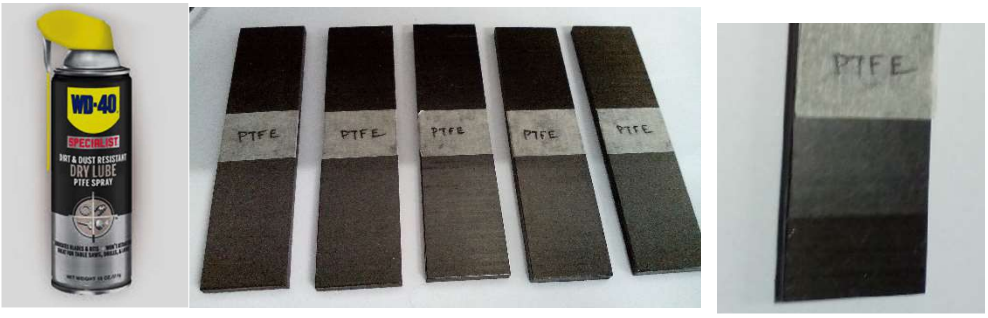


Figure 7. The PTFE lubricant is applied on the surface of the joint around the bonding area

D. Release Agent

In the same manner Release Agent (SemipermMonofilm by R&G) is applied on the surface of the joint around the bonding area, as illustrated in figure 8. Before bonding, the release agent is left to dry for 24 hours. This product is silicone-free and suited for the purpose of processing of thermosets like polyester, epoxy and phenolic resins. It should be noted that this release agent is not suited for processing temperatures above 140 ^{o}C. During the curing of the adhesive film (177 ^{o}C) the release agent will dissolve and it will not prevent the bonding as intended by the manufacturer.

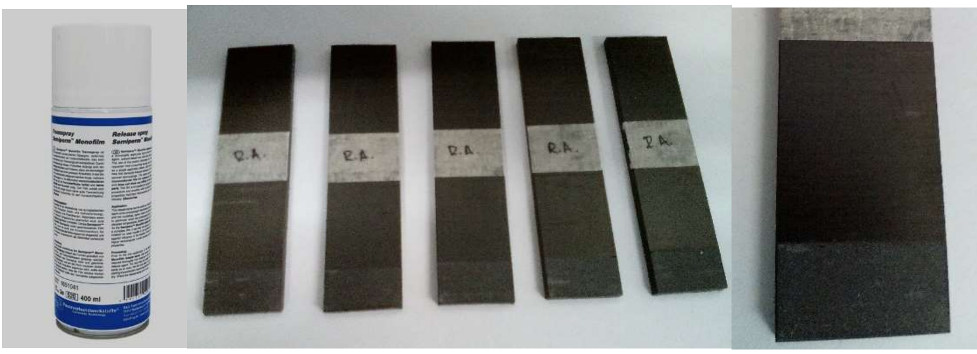


Figure 8. The Release Agent is applied on the surface of the joint around the bonding area

3.3. Bonding procedure

The joints and tabs were bonded together using the hot bonder ANITA NG9201 and a heating blanket (50x50 cm). The recommended cure cycle as stated in the data sheet is 177 ^{o}C for 60 min. The bonding procedure is illustrated in figure 9 and each step is listed below:

1. Ten single lap shear joints were prepared at the same time.
2. The adhesive film is cut to the appropriate dimensions.
3. The joints and tabs are secured using a heat resistant adhesive tape (Kapton)
4. The samples are placed inside a vacuum bag and covered by consumable fabrics and a heat conductive silicon (Mosites)
5. The heating blanket is placed above the samples and negative pressure is applied by the use of a vacuum pump (-0.8 bar).
6. The cure cycle is set to 180 ^{o}C for 60 min with a 3 ^{o}C/min rise.

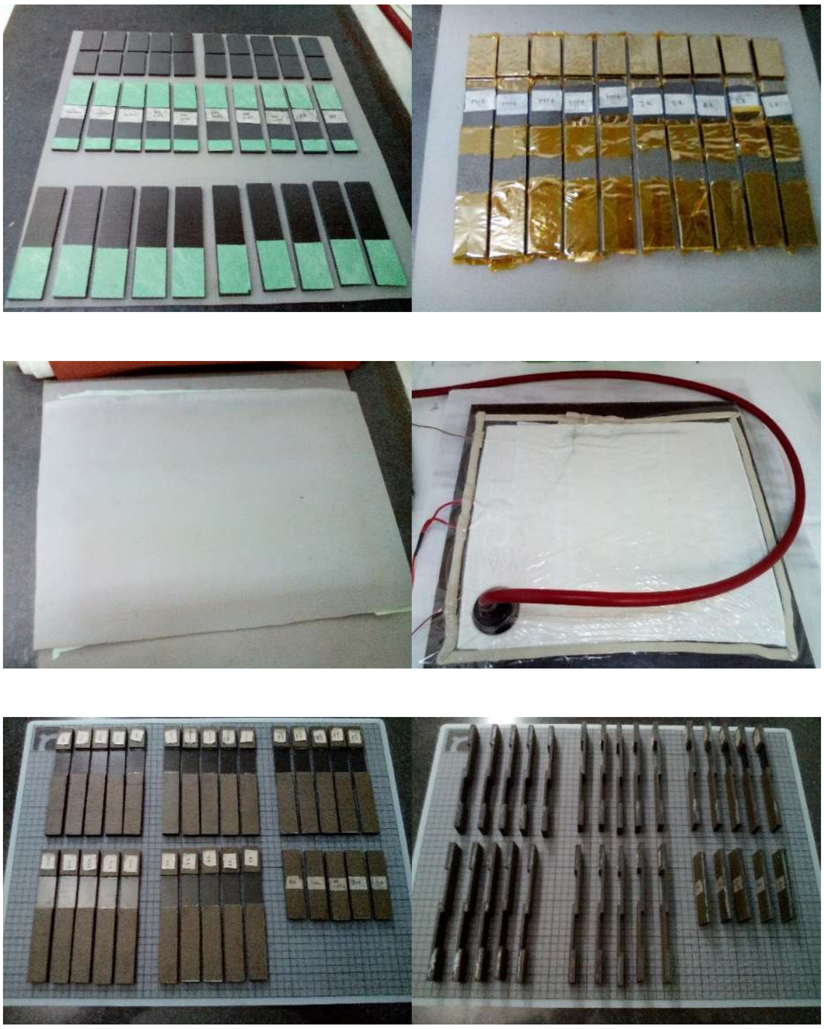


Figure 9. The bonding procedure of lap shear test samples

3.4 Ultrasonic Inspection of the bond line

After the completion of the bonding procedure the bond line is inspected for defects using ELISA ultrasonic inspection devise (A-Scan). In particular the inspection focuses in cases of de-bonding and high porosity, but also the signal from the "healthy" bond is compared to the other cases for any differences. The ultrasonic signal is captured and saved in image format. For every sample, captures are taken above the single joint (point A) and above the bond line (point B), as illustrated in figure 11. It is expected that the signal will be weaker above the bond line due to the attenuation caused by the bond. The results are presented in the following table 2, the inspection parameters are listed below:

- Transducer type: Delay line
- Inspection frequency: 5MHz
- Crystal diameter: 10mm
- Sound Velocity: 2710-2750 m/s

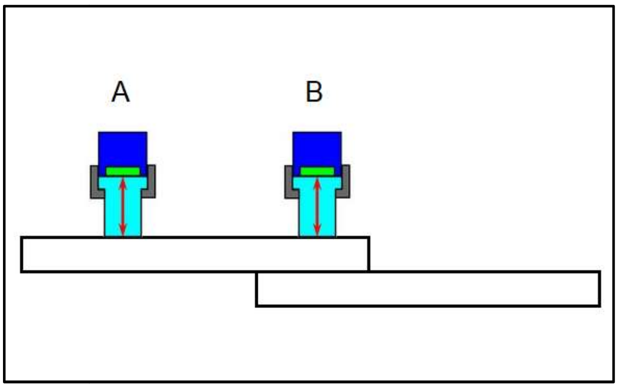


Figure 11. Schematic of the inspection on the lap shear test samples

The ultrasonic inspection didn't reveal any significant defects in the bond line of the samples. But especially in the case of the Pre cured adhesive it is obvious that the signal is much weaker than the reference.

Table 2. Comparative results from the ultrasonic inspection

<table>
<tr><td colspan="2">Reference</td></tr>
<tr><td>
</td><td>
</td></tr>
<tr><td>Single joint (Point A)</td><td>Bond line (Point B)</td></tr>
<tr><td colspan="2">Pre cured adhesive</td></tr>
<tr><td>
</td><td>
</td></tr>
<tr><td>Single joint (Point A)</td><td>Bond line (Point B)</td></tr>
<tr><td colspan="2">Graphite powder</td></tr>
<tr><td>
</td><td>
</td></tr>
<tr><td>Single joint (Point A)</td><td>Bond line (Point B)</td></tr>
<tr><td colspan="2">PTFE Lubricant</td></tr>
<tr><td>
</td><td>
</td></tr>
<tr><td>Single joint (Point A)</td><td>Bond line (Point B)</td></tr>
<tr><td colspan="2">Release Agent</td></tr>
<tr><td>
</td><td>
</td></tr>
<tr><td>Single joint (Point A)</td><td>Bond line (Point B)</td></tr>
</table>

3.5 Mechanical testing (lap shear test)

The mechanical testing (lap shear test) was performed in TWI using an Instron tensile machine. The results from the Reference samples as well as contaminated samples are presented in figures 12-17. The images from the fractured test samples are presented in Annex.

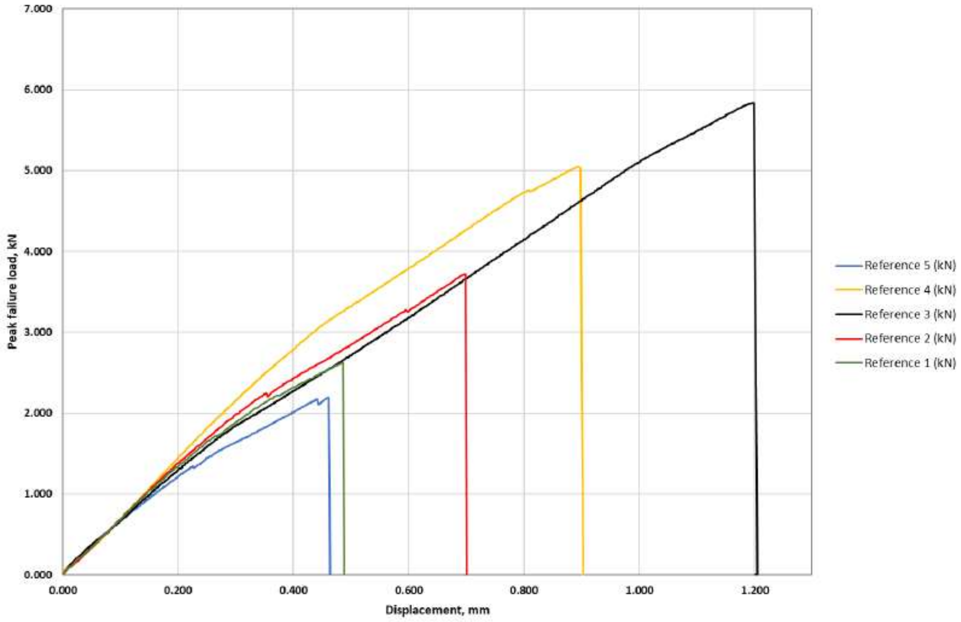


Figure 12. Results from the mechanical testing on the reference samples

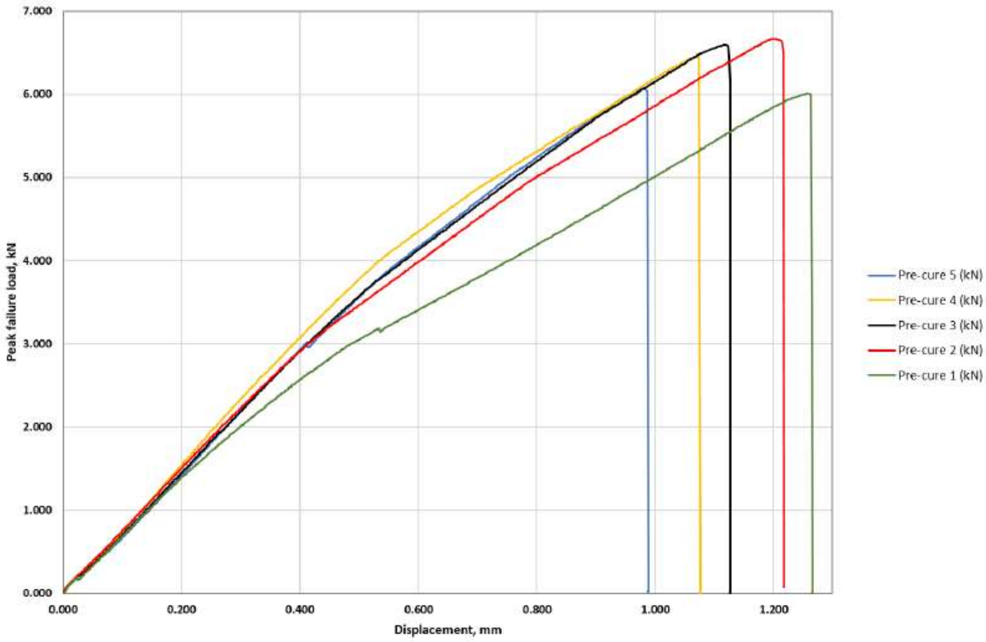


Figure 13. Results from the mechanical testing onpre cured adhesive samples

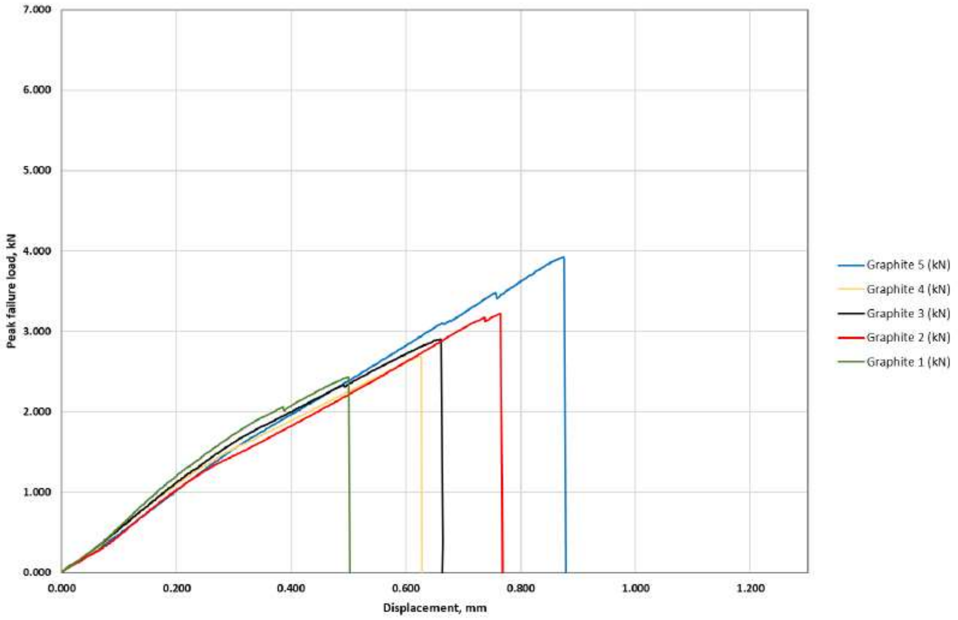


Figure 14. Results from the mechanical testing on graphite powder contaminated samples

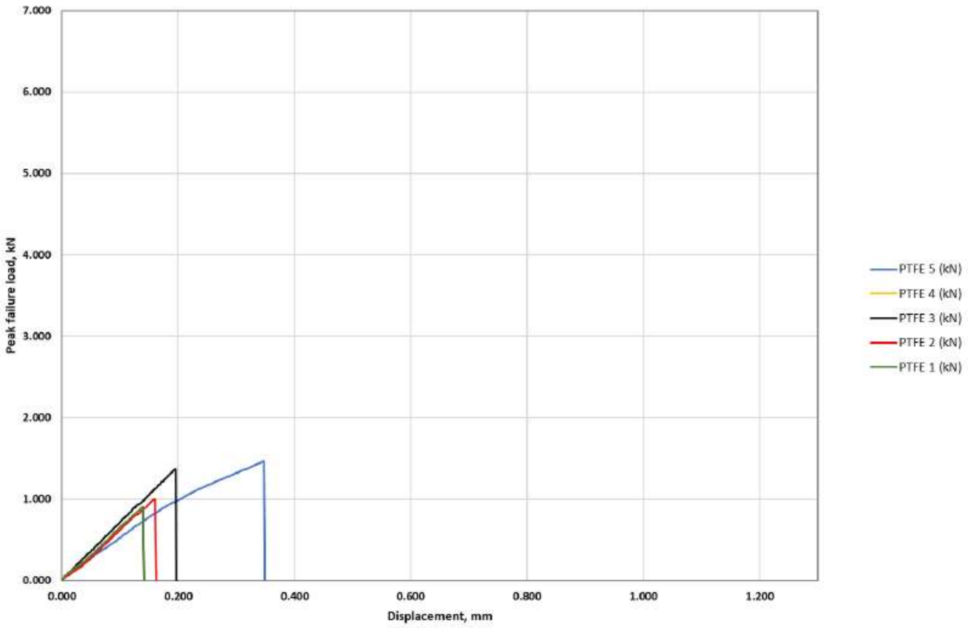


Figure 15. Results from the mechanical testing on PTFE lubricantcontaminated samples

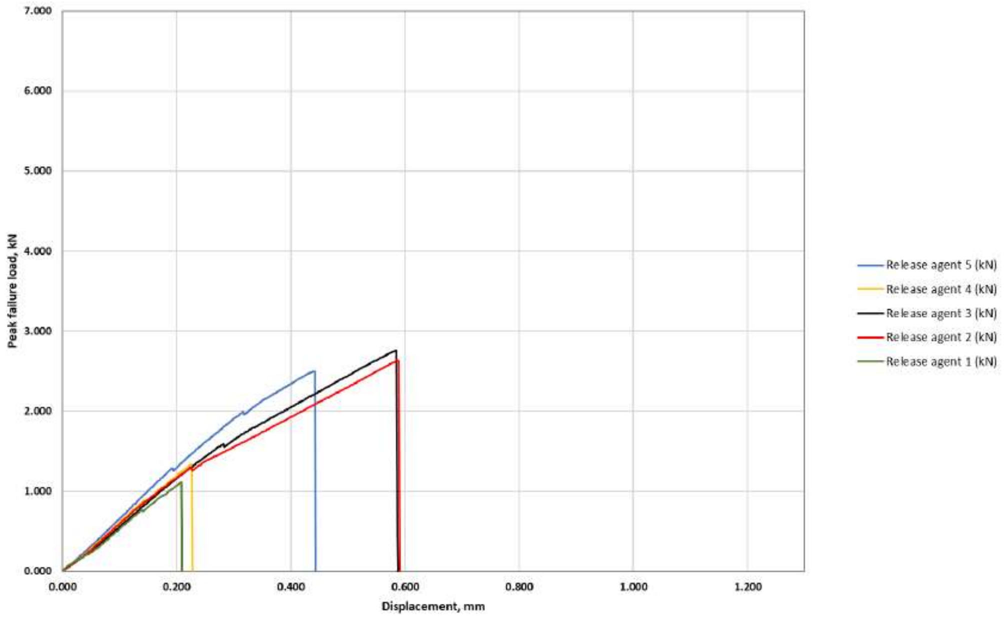
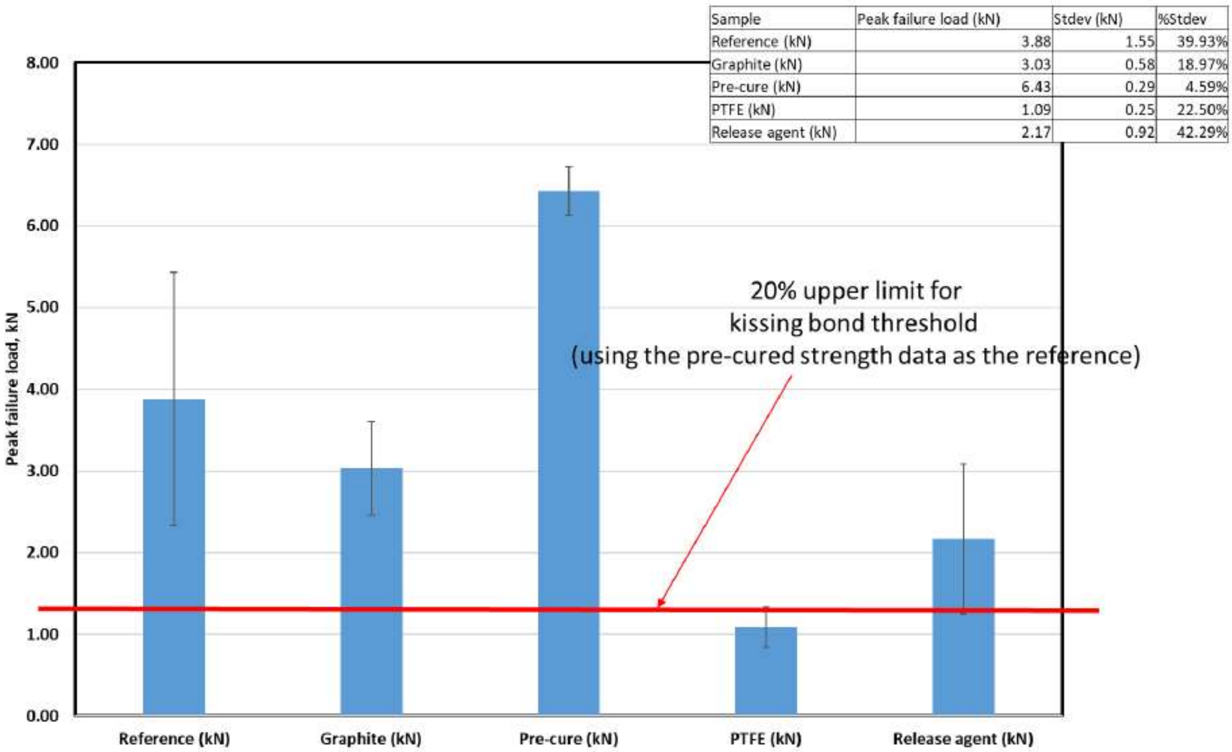


Figure 16. Results from the mechanical testing on Release agentcontaminated samples



Sample	Peak failure load (kN)	Stdev (kN)	%Stdev
Reference (kN)	3.88	1.55	39.93%
Graphite (kN)	3.03	0.58	18.97%
Pre-cure (kN)	6.43	0.29	4.59%
PTFE (kN)	1.09	0.25	22.50%
Release agent (kN)	2.17	0.92	42.29%

Figure 17. Comparative results from the mechanical testing

3.6 Comments on the first set of test samples

Reference samples:

- Significant variability and a relatively low failure strength.
- Mixed modes of failure seen eg R2 – R4 = cohesive and R1, R5 significant adhesion failure.
- Cannot be considered as reliable reference system.

Graphite powder:

- Reduced strength ~40% of strongest system.
- Slightly lower variability.
- Mixed failure mode ie some adhesion and some cohesive.

Pre cured adhesive:

- Strongest system, most consistent results, low scatter!
- Mainly substrate failure with some cohesive.

PTFE lubricant:

- Weakest system, almost all tests were below the 20% threshold to conform to kissing bond criterion.
- Results quite consistent.
- Almost total adhesion failure although some small areas of cohesive failure.

Release agent:

- Second weakest system but wide variation in results.
- Average failure strength exceeds 20% threshold.
- Mixed failure mode on some samples ie adhesion/cohesive for RA2, 3 and 5 but RA1 and RA4 almost complete adhesion failure.

The results for the reference specimens are unsafe and the experiments will have to be repeated in order to obtain some reliable data.Only the PTFE contamination passed the kissing bond strength threshold and mostly passed the failure surface type ie all/mostly adhesion in type. The experimental procedure should be performed again in a different set of samples.

4. Second set of trial test samples

4.1 Surface preparation and contamination

The experiments involve mechanical testing on single lap shear joints. Smaller panels were cut out from the composite panels provided by Dassault Aviation. The cutting was done using a table saw equipped with a diamond disk. The overlap area was abraded using sandpaper of 120 grit. On the Reference samples both sides were abraded while on contaminated samples only one side was abraded. All samples were cleaned thoroughly using Diestone cleaner.
Additionally, hybrid samples were prepared such that the contamination is applied in bands enabling each specimen to have both a contaminated and a non-contaminated bond area, as illustrated in figure 18.

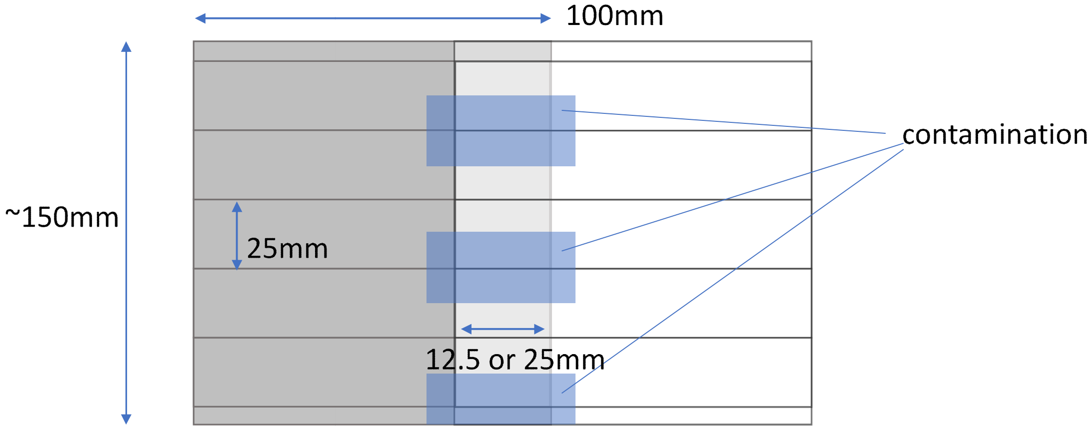


Figure 18. Schematic of Hybrid test samples

A. PTFE Lubricant

In this method PTFE lubricant is applied on the surface of the joint around the bonding area, as illustrated in figure 19. Before bonding, the lubricant is left to dry for 24 hours. It is assumed that the PTFE (Teflon) particles will create a thin layer preventing the adhesive to bond to the adherent joint.

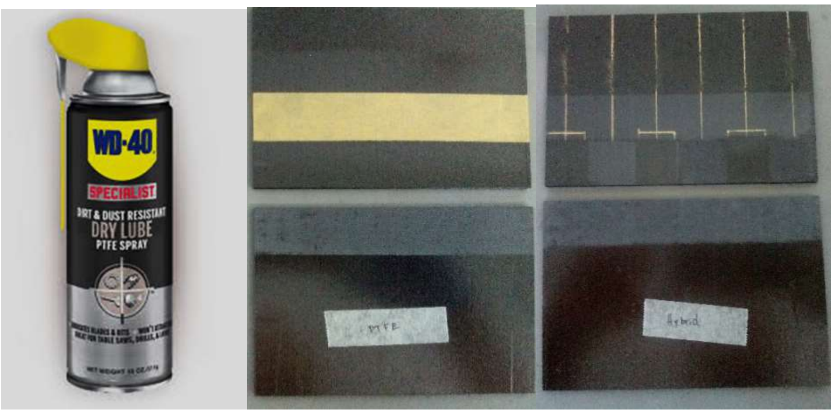


Figure 19. The PTFE lubricant is applied on the surface of the joint around the bonding area

B. Lithium grease (white grease)

In the same manner Lithium grease is applied on the surface of the joint around the bonding area, as illustrated in figure 20. Before bonding, the grease is left to dry for 24 hours. It should be noted that this the grease during the curing of the adhesive film (177 °C) will dissolve in the adhesive film enabling partial bonding.

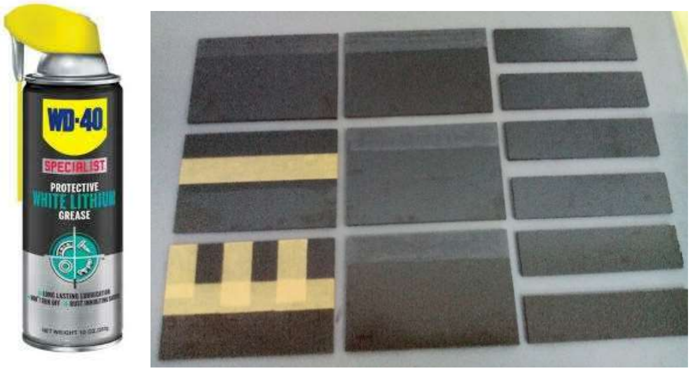


Figure 20. The Lithium grease is applied on the surface of the joint around the bonding area

4.2 Bonding procedure

The panels were bonded together using the hot bonder ANITA NG9201 and a heating blanket (50x50 cm). The recommended cure cycle as stated in the data sheet is 177 °C for 60 min. The bonding procedure is illustrated in figure 21. In all experiments the bonding is done using aeronautical grade adhesive film FM 300M. An aluminum tape is used to secure the plates during the bonding and to prevent the adhesive from leaking. This type of adhesive tape performs betters compared to Blue tape, Kapton tape and regular masking tape. In the final stage tabs were bonded using Acrylic adhesive (3M Scotch-Weld 8810).

1. The samples are placed inside a vacuum bag and covered by consumable fabrics and a heat conductive silicon (Mosites).
2. The heating blanket is placed above the samples and negative pressure is applied using a vacuum pump (-0.8 bar).
3. The cure cycle is set to 180 °C for 70 min with a 3 °C/min rise.

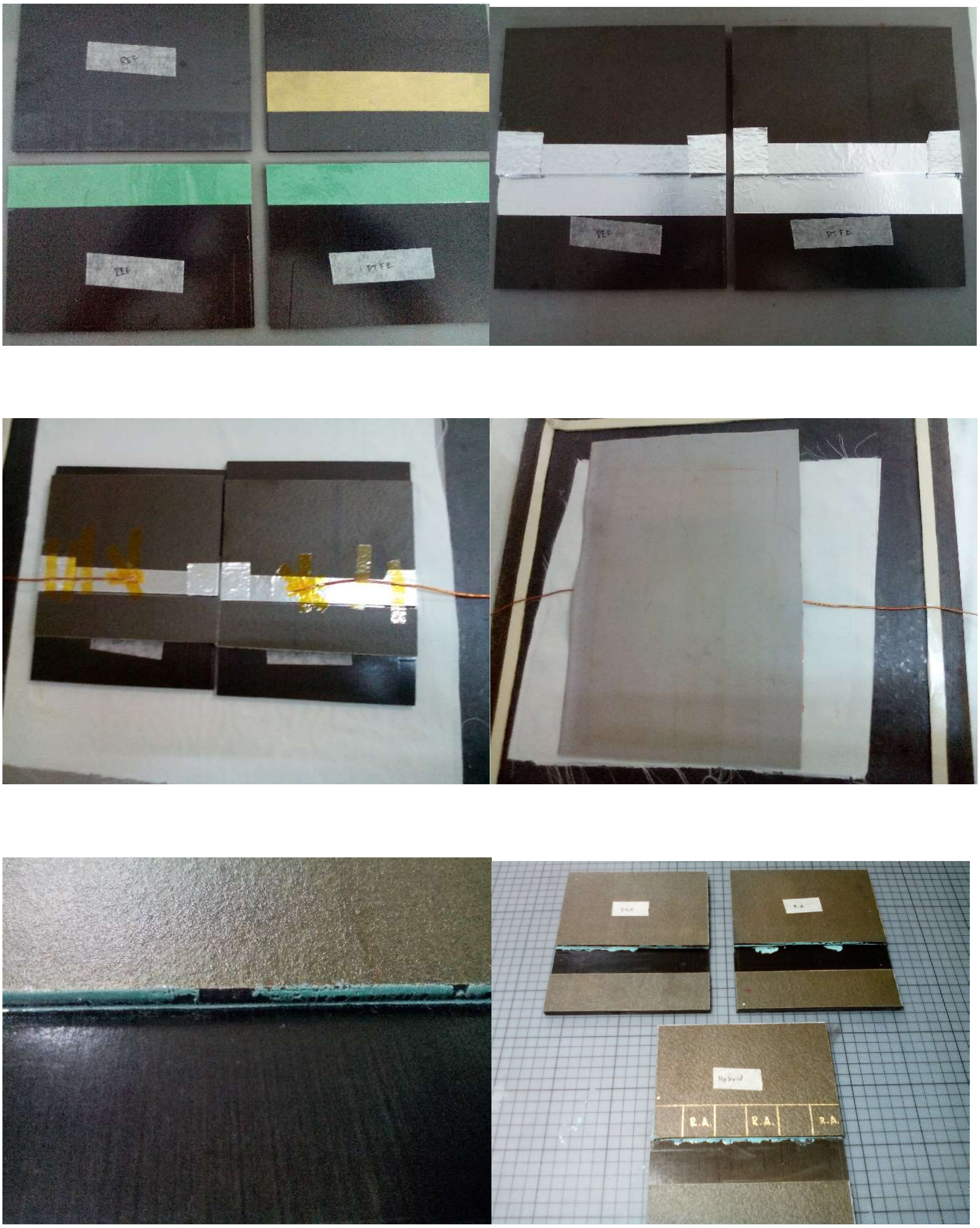


Figure 21. The bonding procedure of lap shear test samples

4.3 Ultrasonic Inspection of the bond line

The Ultrasonic inspection (C-scan) was performed by the TWI team. The results from the Reference samples as well as contaminated samples are presented in figures 22-23.

PTFE lubricant:

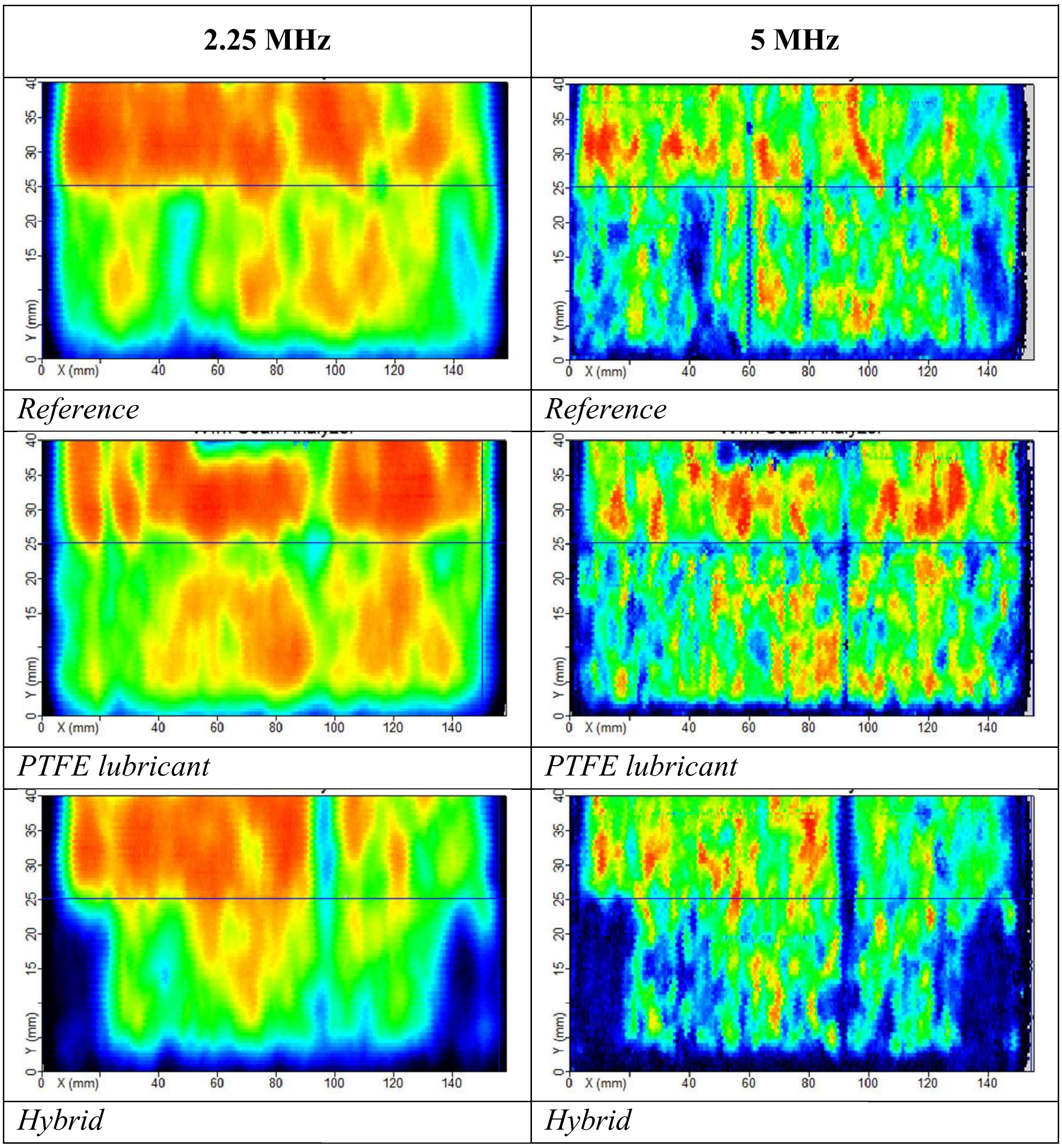


Figure 22. Images taken from C-scan inspection representing the signal from the first back wall echo

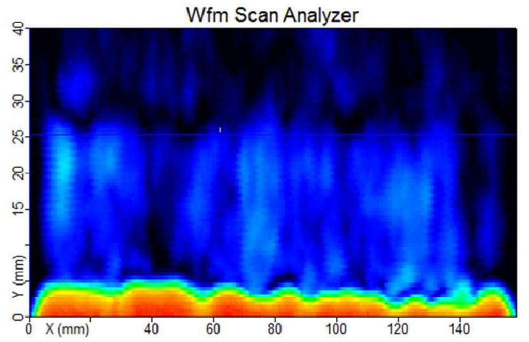
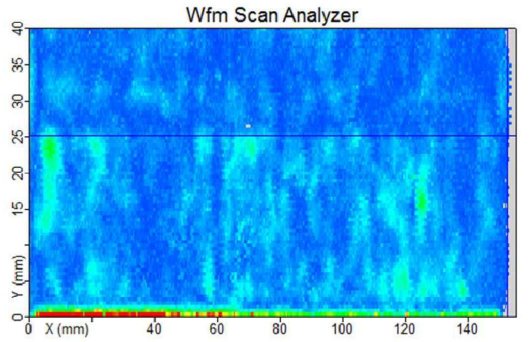
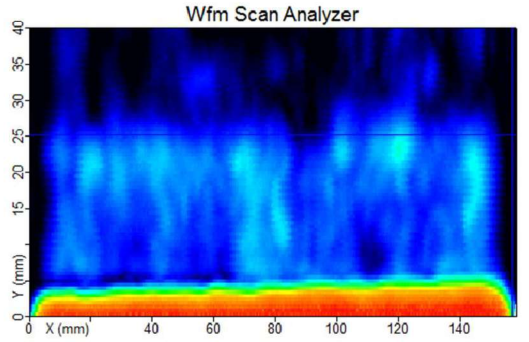
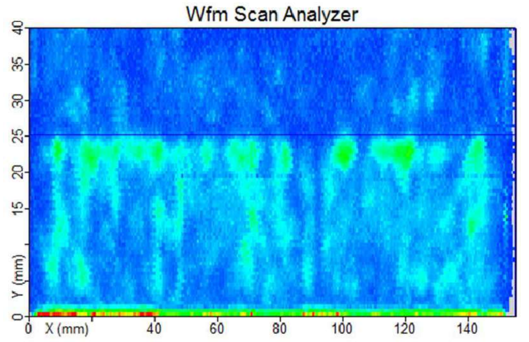
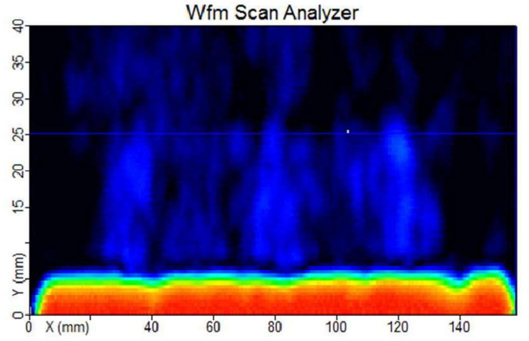
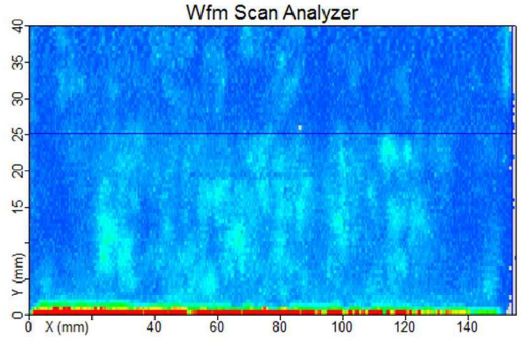
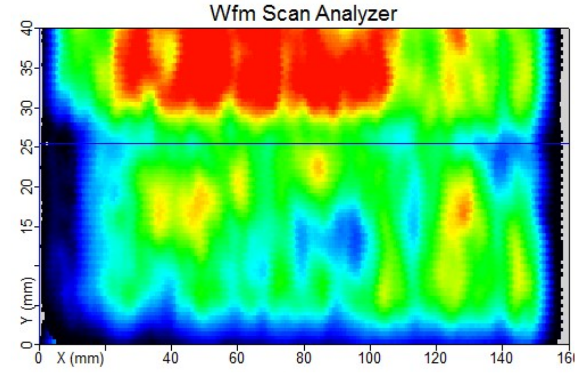
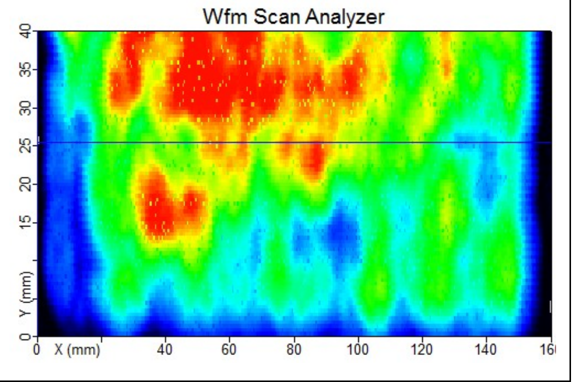
2.25 MHz	5 MHz
	
Reference	Reference
	
PTFE lubricant	PTFE lubricant
	
Hybrid	Hybrid

Figure 23. Images taken from C-scan inspection representing the signal from the second back wall echo

White Grease:

2.25 MHz	5 MHz
	
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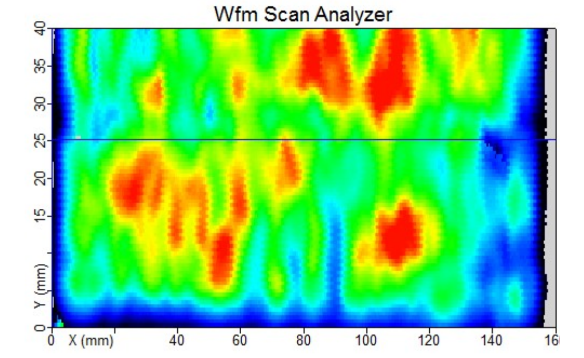
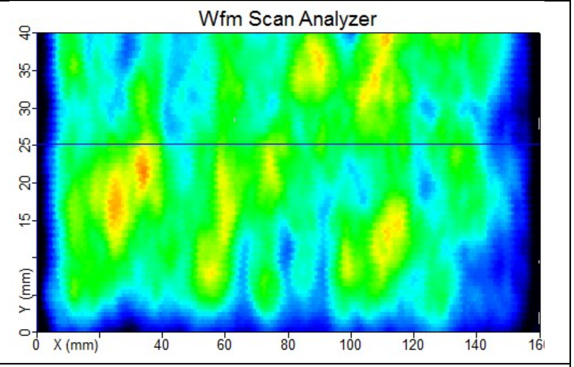
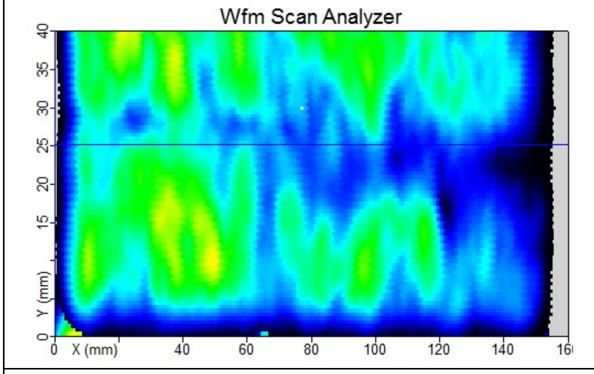
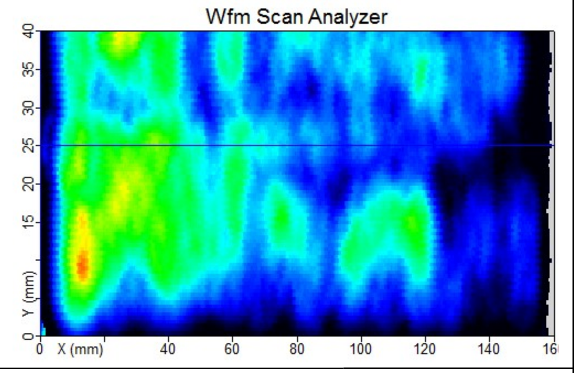
	
White grease	_White grease_
	
Hybrid	_Hybrid_

Figure 24. Images taken from C-scan inspection representing the signal from the first back wall echo

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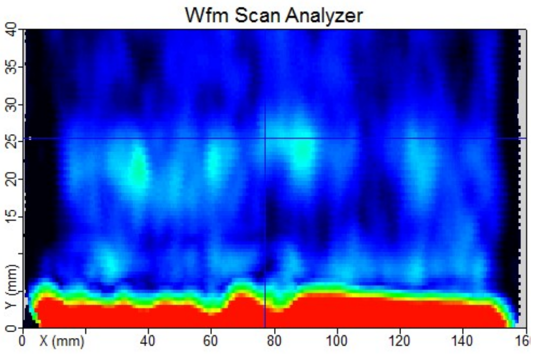
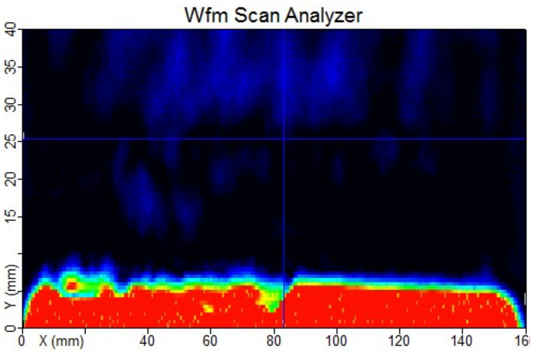
<tr><td>
</td><td>
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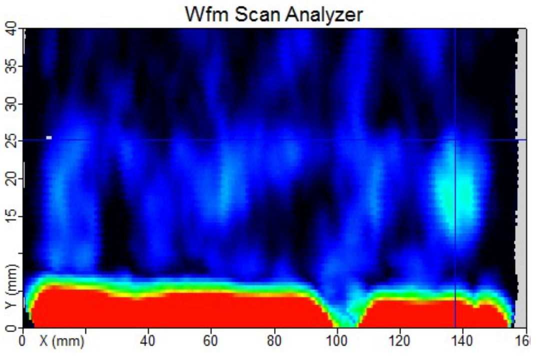
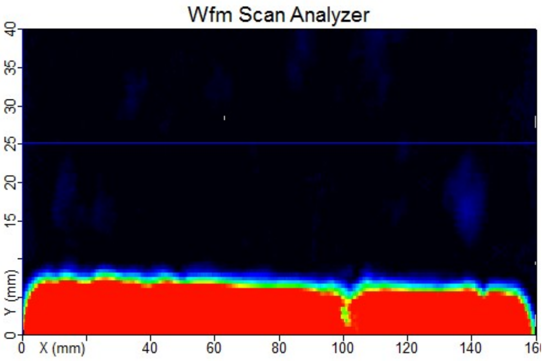
<tr><td>
</td><td>
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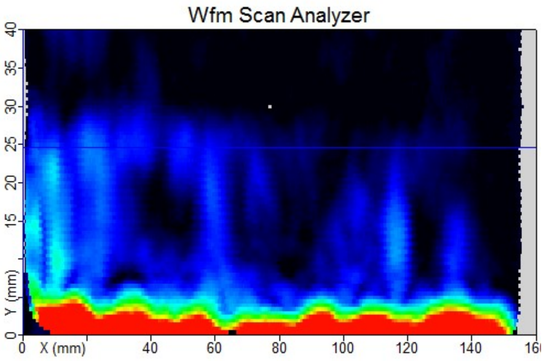
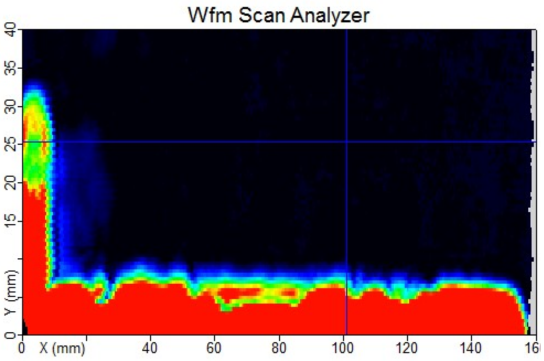
<tr><td>White grease</td><td>White grease</td></tr>
<tr><td>
</td><td>
</td></tr>
<tr><td>Hybrid</td><td>Hybrid</td></tr>
</table>

Figure 25. Images taken from C-scan inspection representing the signal from the second back wall echo

4.4 Mechanical testing (lap shear test)

The mechanical testing (lap shear test) was performed in TWI using an Instron tensile machine. The results from the Reference samples as well as contaminated samples are presented in figures 26-31. The failure mode of samples contaminated with White Grease is illustrated in figure 34.

PTFE lubricant:

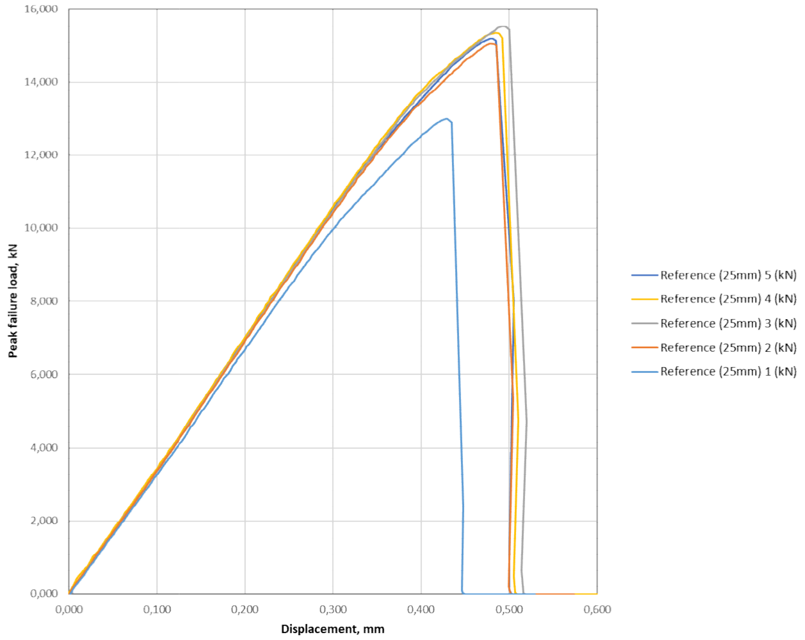


Figure 26. Results from the mechanical testing on the reference samples

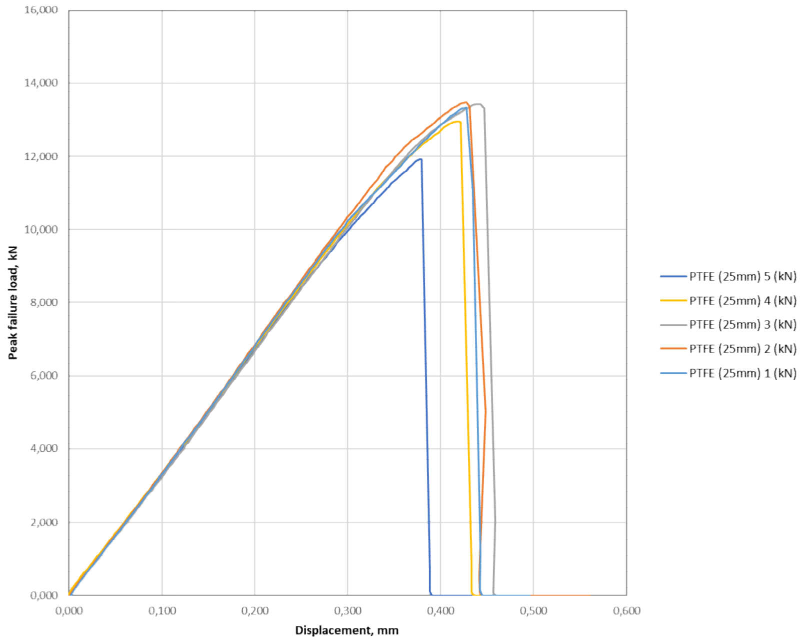


Figure 27. Results from the mechanical testing on the PTFE lubricant contaminated samples

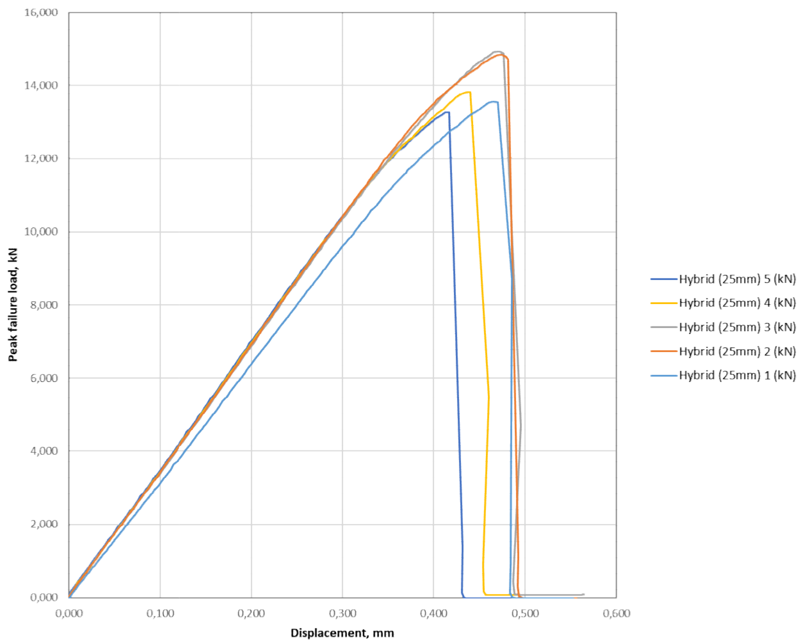


Figure 28. Results from the mechanical testing onthe Hybrid (PTFE lubricant) samples

White Grease:

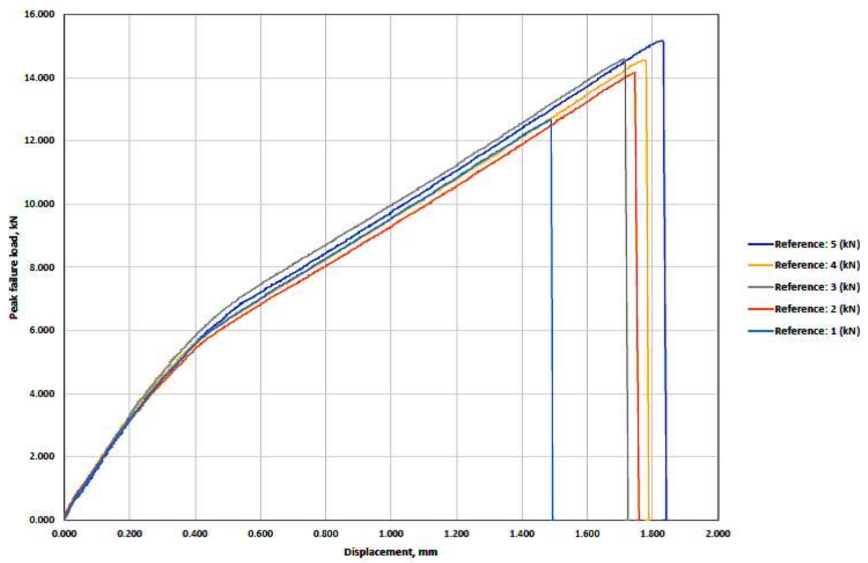


Figure 29. Results from the mechanical testing on the reference samples

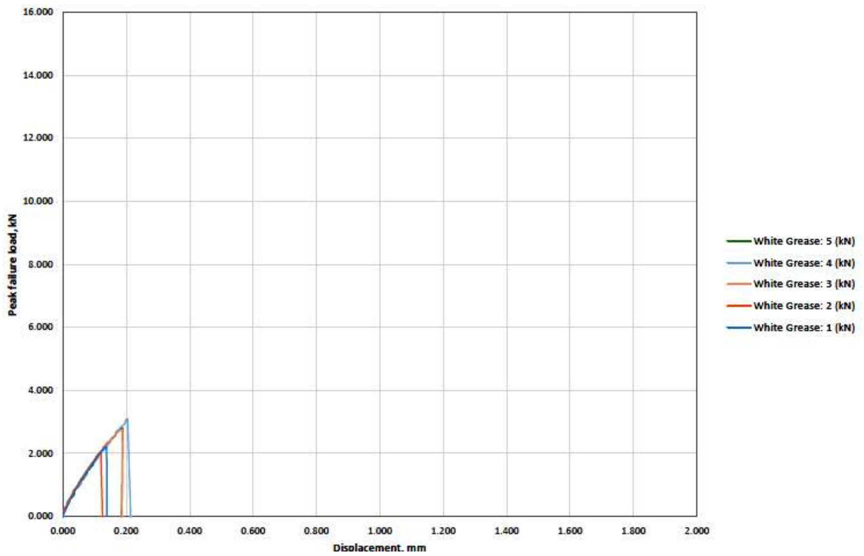


Figure 30. Results from the mechanical testing on White grease contaminated samples

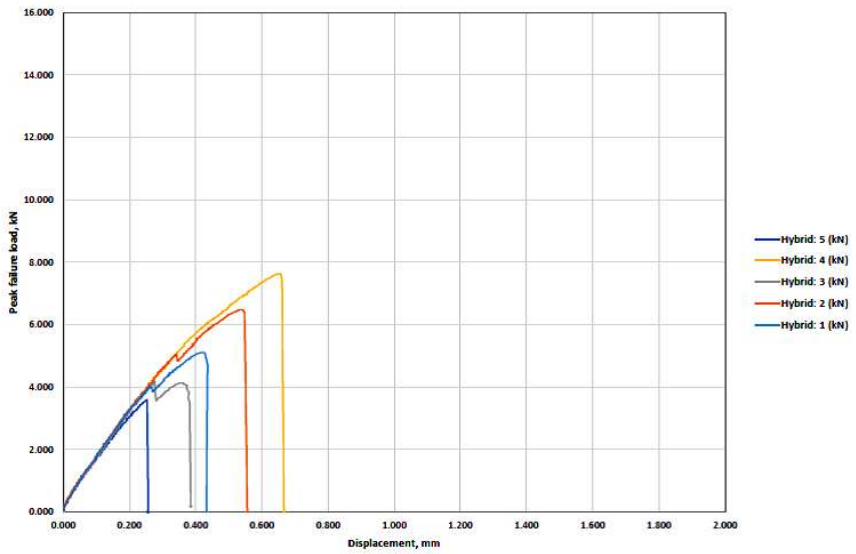


Figure 31. Results from the mechanical testing onthe Hybrid (White grease) samples

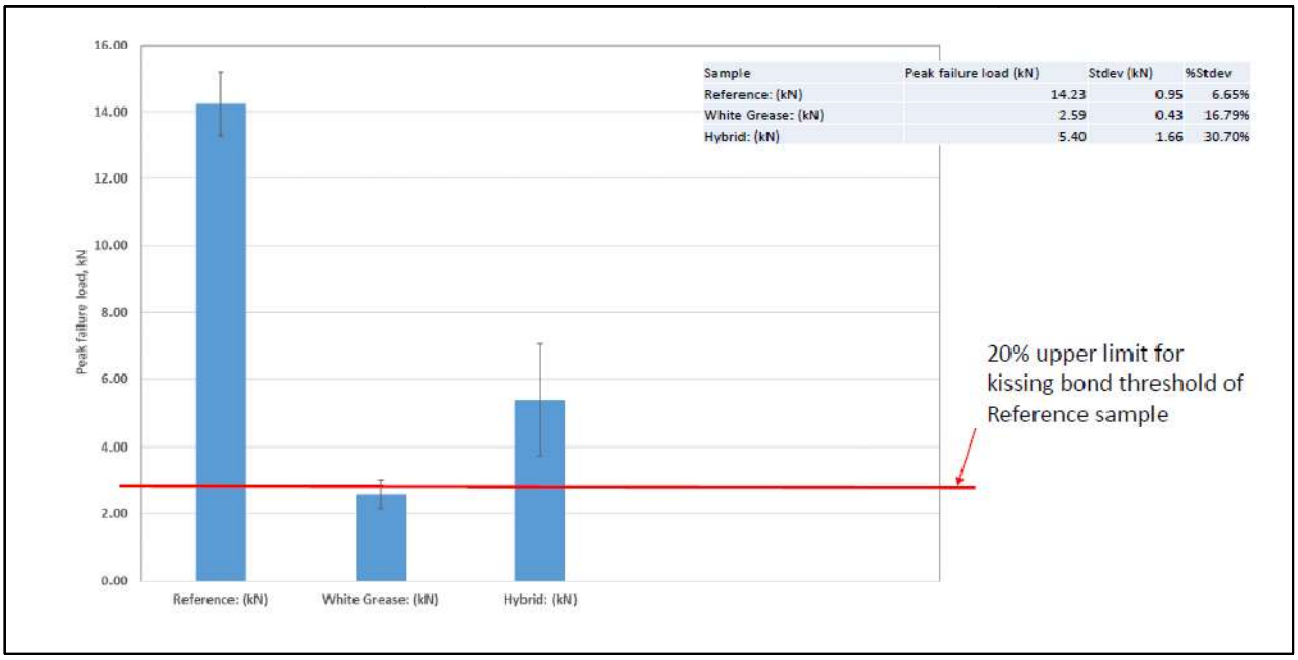


Figure 32. Comparative results regarding the failure load (White Grease contamination)

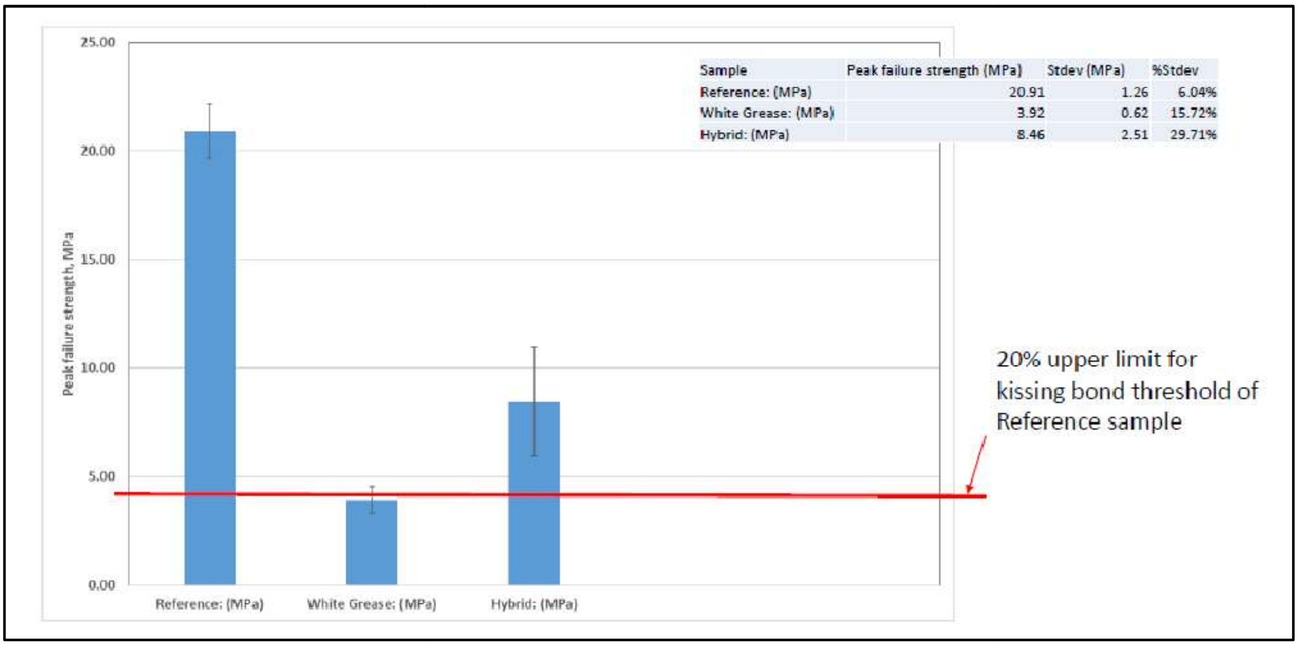
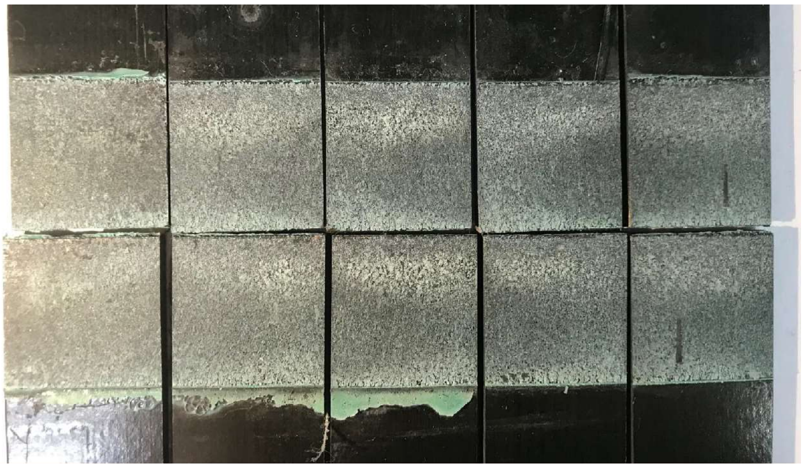
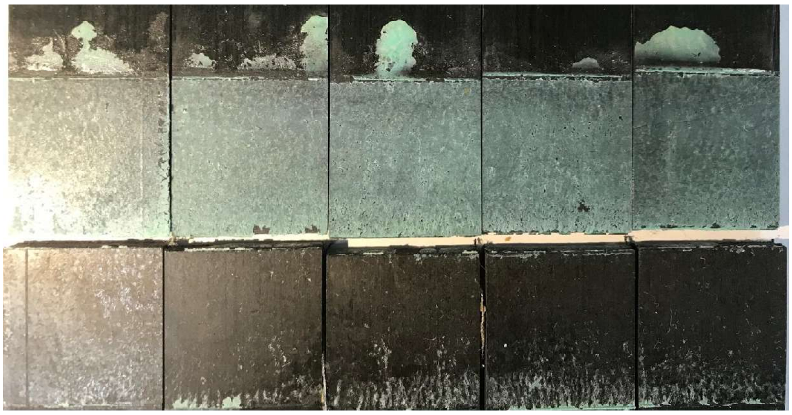


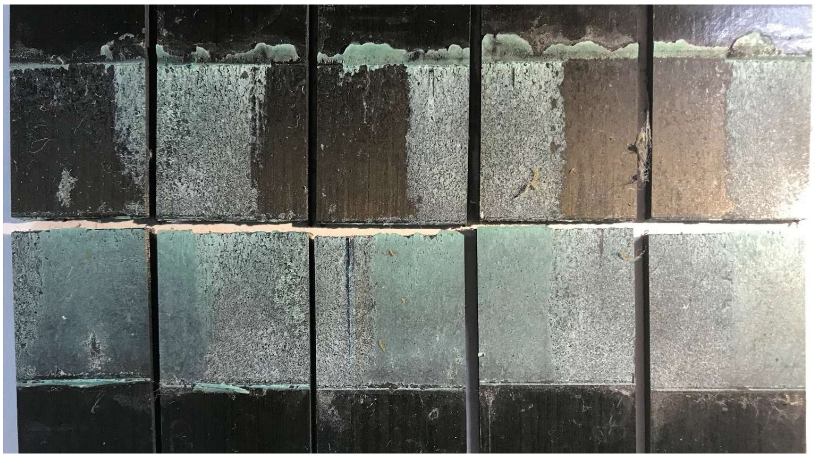
Figure 33. Comparative results regarding the failure strength (White Grease contamination)



_a)_



_b)_



_c)_

Figure 34. The failure mode of samples contaminated with White grease a) Reference b) Contaminated c) Hybrid

4.5 Comments on the second set of test samples

PTFE lubricant contamination:

- For Reference samples, failure surface is what is required i.e. full cohesive and no fibre failure.
- For PTFE contaminated samples, the failure load did not occur low enough when compared to the reference samples to replicate kissing bonds.
- Lack of fibre failure is due to top layer fibres being parallel to load direction.
- For PTFE contaminated samples failure is a mixture of cohesive and adhesion.
- Adhesion failure is predominantly seen in the end regions of the joint.
- Presence of PTFE is having a difference on failure mode but not sufficient to influence failure load value in a significant way.
- Some visible evidence of differing failure mode between two area (clean vs contaminated) on the Hybrid samples.
- Fibre failure indicative of very high levels of stress in these regions.

White Grease contamination:

- The Reference samples show high strength and consistency and the failure mode is cohesive
- Contaminated samples with White grease show <20% failure load compared to reference samples.
- Contaminated samples with White grease show consistent adhesion failure at the grease interface.
- Hybrid samples contaminated with White grease show ~30%failure load compared to reference samples.
- Hybrid samples contaminated with White show large scatter which is typical of variable greased surface area present
- Hybrid samples contaminated with White showclear mixed mode failure –cohesive and adhesion failure

5. Remarks and final Conclusions

Within Task 2.1several methods were tested and evaluated regarding the ability of recreating artificial "Kissing Bonds". The criteria on which this study is based are summarized as follows:

- ✓ Bonds which fail at less than 20% of their nominal strength (lap shear test)
- ✓ Mode of failure must be adhesive in type (i.e. purely at the adherend/adhesive interface)
- ✓ Unable to detect using conventional NDT methods (ultrasonic)

Among the all methods tested, only the use of "White Grease" as contamination could meet those criteria.

- Single lap shear tests on contaminated samples with White Grease showed failure at less than 20% compared to Reference samples.
- The use of White Grease causes adhesion failure in a reliable and repeatable manner.
- The NDT data from the hybrid joints does not appear to reveal the presence of the treated areas although the mode of failure and the low failure value (<20%) of the fully treated joints would suggest that the defect is 'invisible' to that type of NDT and can therefore be termed a 'kissing bond' for that process.
- Joints, where regions of treated areas have been produced adjacent to well bonded areas, will enable direct comparison studies to be made to explore the efficacy of various NDT techniques.

6. Annex: Images from fractured test samples

6.1 First set of trial test samples

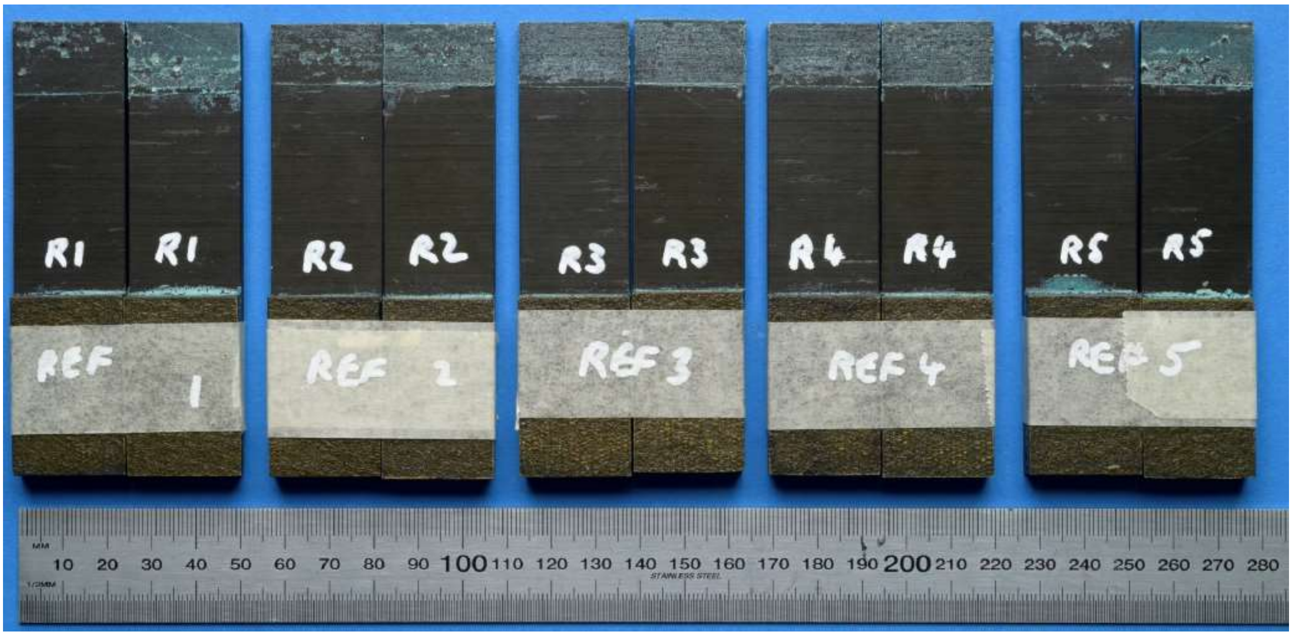


Image 6.1. Failure mode of Reference test samples

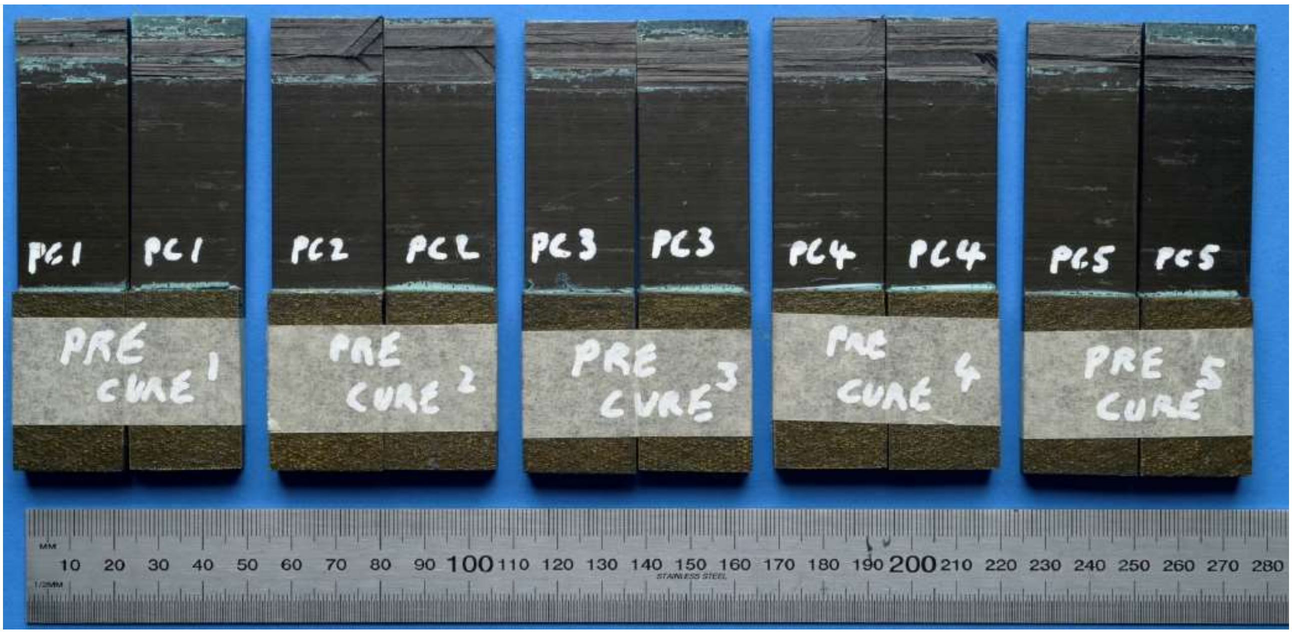


Image 6.2. Failure mode of Pre cured adhesive test samples

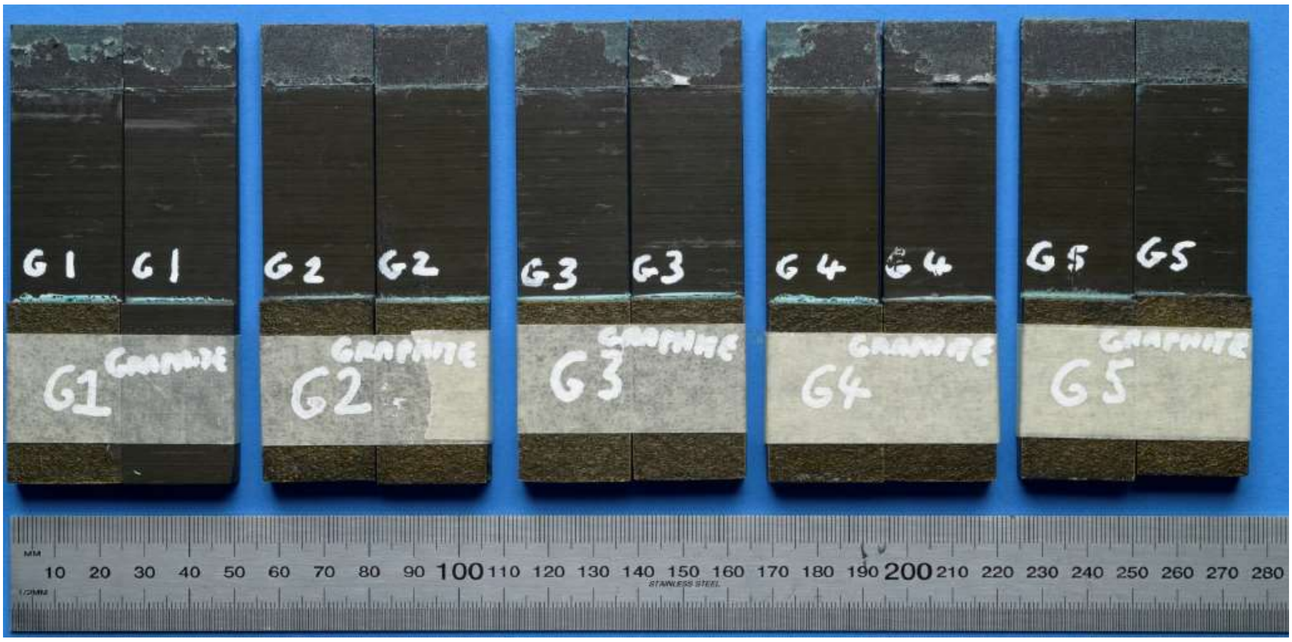


Image 6.3. Failure mode of Graphite powder contamination test samples

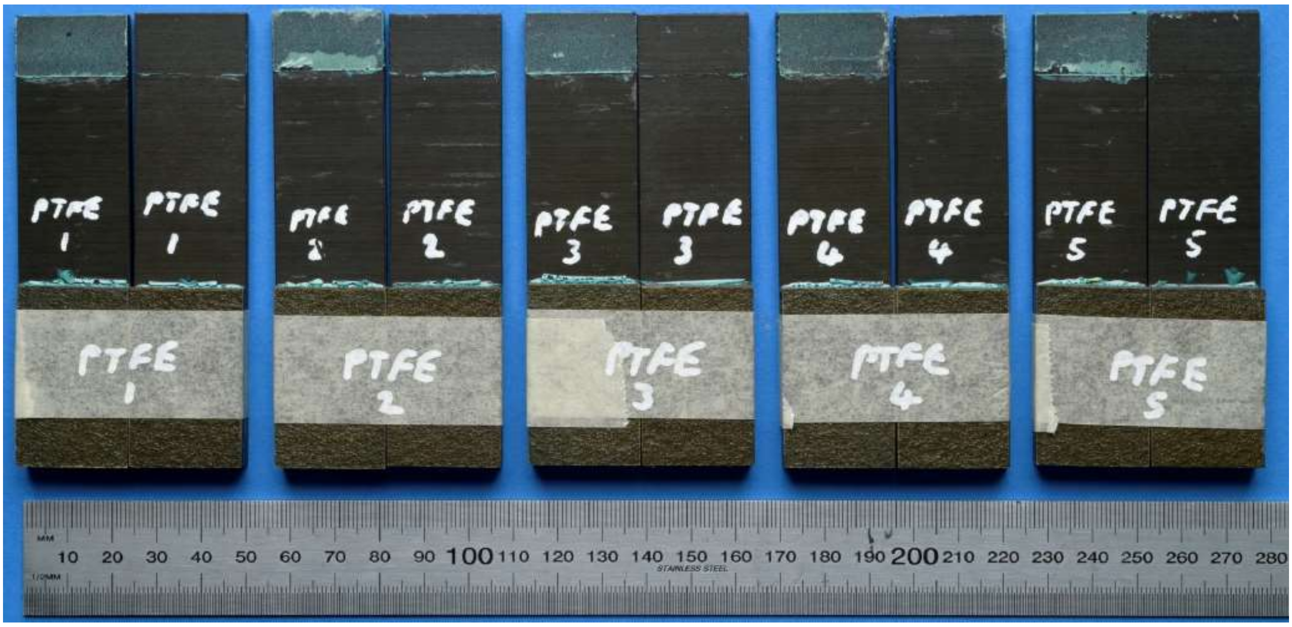


Image 6.4. Failure mode of PTFE lubricant contamination test samples

Image 6.5. Failure mode of Release agent contamination test samples

6.2 Second set of trial test samples

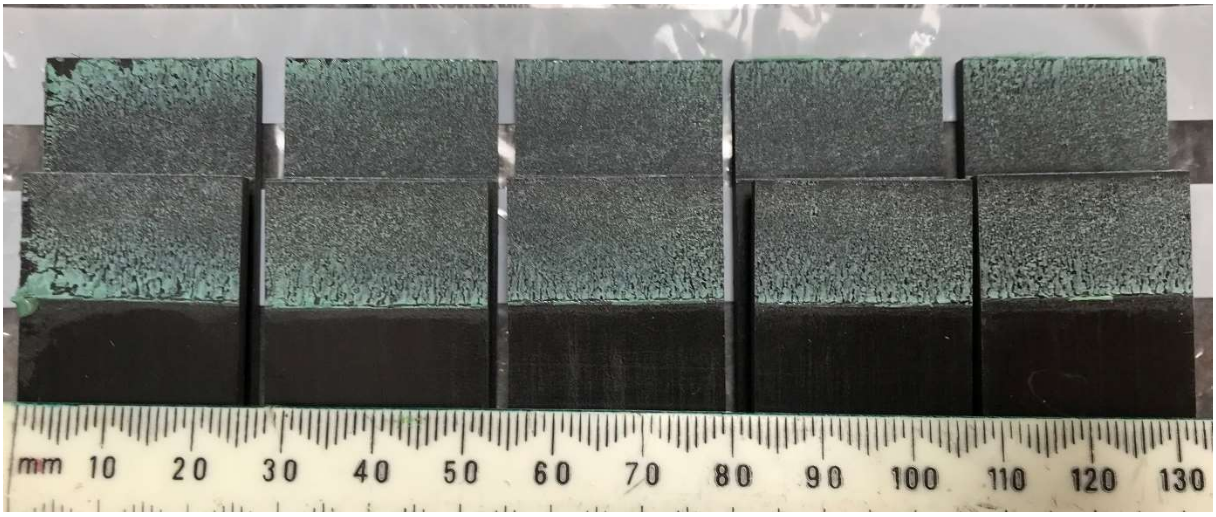


Image 6.6. Failure mode of Reference test samples

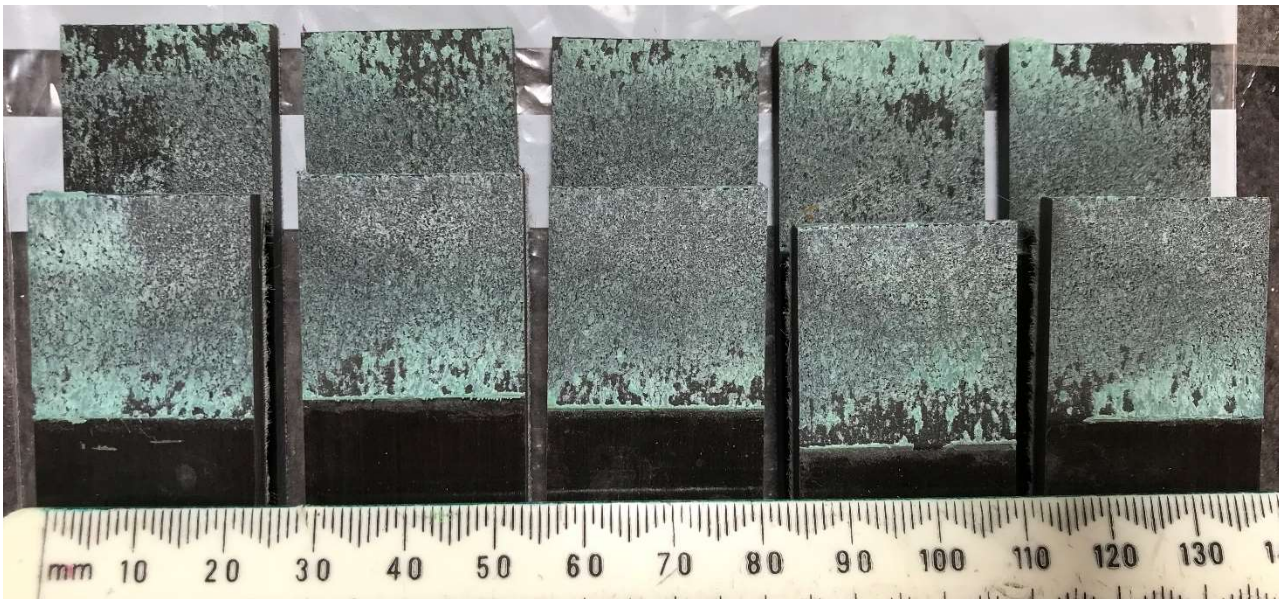


Image 6.7. Failure mode of PTFE lubricant contamination test samples

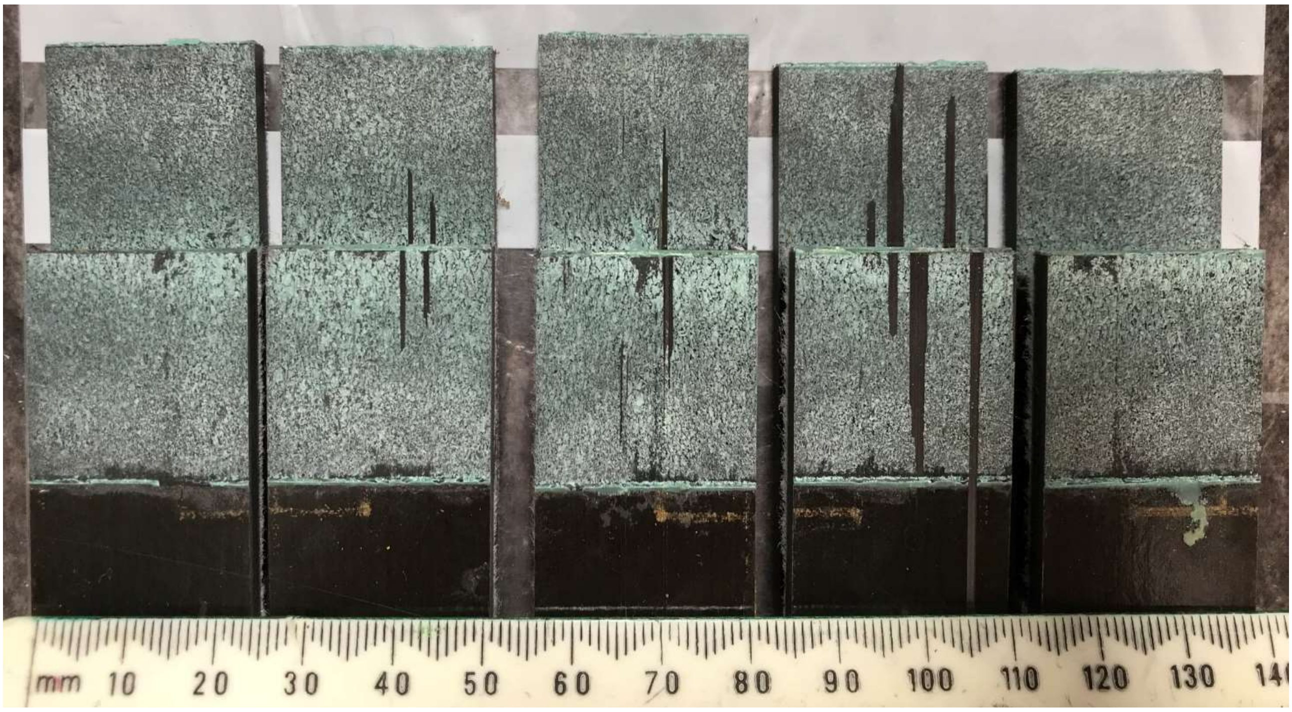


Image 6.8. Failure mode of Hybrid (PTFE lubricant contamination) test samples