On the manufacturing and testing of dissimilar and adhesive Ti-CFRP joints

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Problem area

In Europe, research is performed in the area of hybrid laminar flow control (HLFC) on aircraft wings for drag reduction. HLFC is achieved by so-called transition delay through suction of the turbulent boundary layer by perforated wing surfaces. A promising structural solution for these perforated surfaces is the combination of a micro-drilled outer titanium surface adhesively bonded with an inner composite segmented structure.

In the EU TICOAJO project this titanium-composite adhesive joint is investigated. Failure of this joint must be investigated for certification and characterization of the adhesive properties is needed for future numerical simulation purposes. This research is relevant for Dutch industry since joining of hybrid structures will become more common.

Description of work

A generic configuration of the titanium-composite adhesive joint is studied. The metal and composite adherents have different thicknesses, while also two metallic
backing beams of different thicknesses are applied on both sides of the coupon to ensure the non-yielding of the metallic adherent. Special attention is paid to the Double Cantilever Beam (DCB) and the End Notched Flexure (ENF) specimens. Residual thermal stresses are considered in the design and post-processing of the results to account for high-temperature curing cases. In this study, the manufacturing and testing of coupons for the determination of the strain energy release rates during delamination crack growth in dissimilar (metal to composite) adhesive joints is investigated.

The research includes different technologies for the bonding of dissimilar materials. The ones investigated are co-bonding with adhesive and without, secondary bonding for thermoset composites and secondary bonding with thermoplastic material. Tests have been performed using the DCB and ENF setup and are planned for the novel moment loading setup at NLR, the so-called DCB-UBM. The advantage of this setup is that the crack length does not need to be measured, since the moment on the specimen is constant, independent of the crack length. The test results are evaluated to determine the optimal process parameters and technology for the joints.

**Results and conclusions**

The basis for the testing done was the standard DCB and ENF setup that has been modified to accommodate the asymmetry of the samples and the large thickness due to the backing material. From the test results it showed that for most types of connection the adhesive and non-adhesive joint is very strong with the pre-treatments used. The composite itself appears to be the weak point and delaminations start to form during the DCB testing. Further research will be done in the TICOAJO project to investigate these effects including the use of the moment loading setup. Also the effect of environmental conditions and fatigue on the joint behaviour will be investigated. Also these aspects are greatly complicated by the need for the backing material.

**Applicability**

The results of this research are applicable to similar metal-composite connections.
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Summary

In Europe, research is performed in the area of hybrid laminar flow control (HLFC) on aircraft wings for drag reduction. HLFC is achieved by so-called transition delay through suction of the turbulent boundary layer by perforated wing surfaces. A promising structural solution for these perforated surfaces is the combination of a micro-drilled outer titanium surface adhesively bonded with an inner composite segmented structure.

In the EU TICOAJO project this titanium-composite adhesive joint is investigated. Failure of this joint must be investigated for certification and characterization of the adhesive properties is needed for future numerical simulation purposes. Four types of connection are investigated, see Table 1.

<table>
<thead>
<tr>
<th>Union type</th>
<th>Description</th>
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<tbody>
<tr>
<td>1</td>
<td>Secondary bonding using FM94 adhesive</td>
</tr>
<tr>
<td>2</td>
<td>Co-bonding with adhesive FM300</td>
</tr>
<tr>
<td>3</td>
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The composite itself appears to be the weak point and delaminations start to form during the DCB testing. Further research will be done in the TICOAJO project to investigate these effects including the use of the moment loading setup. Also the effect of environmental conditions and fatigue on the joint behaviour will be investigated. Also these aspects are greatly complicated by the need for the backing material.
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## Abbreviations

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<tr>
<th>ACRONYM</th>
<th>DESCRIPTION</th>
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<tbody>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>CFRP</td>
<td>Carbon Fibre Reinforced Plastic</td>
</tr>
<tr>
<td>CTD</td>
<td>Cold Dry conditions</td>
</tr>
<tr>
<td>DCB</td>
<td>Double Cantilever Beam test</td>
</tr>
<tr>
<td>DCB-UBM</td>
<td>Double Cantilever Beam test using the Moment loading setup</td>
</tr>
<tr>
<td>ENF</td>
<td>End Notch Fracture test</td>
</tr>
<tr>
<td>HLFC</td>
<td>Hybrid Laminar Flow Control</td>
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<tr>
<td>HTW</td>
<td>Hot Wet conditions</td>
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<tr>
<td>NLR</td>
<td>Netherlands Aerospace Centre</td>
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<tr>
<td>RT</td>
<td>Room Temperature conditions</td>
</tr>
<tr>
<td>VARTM</td>
<td>Vacuum Assisted Resin Transfer Moulding</td>
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</table>
ON THE MANUFACTURING AND TESTING OF DISSIMILAR ADHESIVE TI-CFRP JOINTS

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Keywords: adhesive joint, composite, titanium, manufacturing

Abstract
In this study, the manufacturing and testing of coupons for the determination of the strain energy release rates during delamination crack growth in dissimilar (metal to composite) adhesive joints is investigated. It is part of the TicoAjo EU project and the results will be used for an application in aerospace wing design using Hybrid Laminar Flow Control (HLFC). A generic joint configuration is studied; the metal and composite adherents have different thicknesses, while also two metallic backing beams of different thicknesses were applied in both sides of the coupon to ensure the non-yielding of the metallic adherent. Special attention was paid to the Double Cantilever Beam (DCB) and the End Notched Flexure (ENF) specimens. Residual thermal stresses are considered in the design and post-processing of the results to account for high-temperature curing cases.

The research includes different technologies for the bonding of dissimilar materials. The ones investigated are co-bonding with adhesive and without, secondary bonding for thermoset composites and secondary bonding with thermoplastic material. Tests have been performed using the DCB and ENF setup and are planned for the novel moment loading setup at NLR, the so-called DCB-UBM. The advantage of this setup is that the crack length does not need to be measured, since the moment on the specimen is constant, independent of the crack length. The test results will be evaluated to determine the optimal process parameters and technology for the joints.
1 Introduction

In Europe the nearby state-of-the-art Hybrid Laminar Flow Control (HLFC) is achieved by extracting the (turbulent) boundary layer by perforated suction surfaces [2]. First trials using micro-perforated titanium suction surfaces are being performed at leading edge sections of wings and tail air foils. A promising structural solution is the combination of a micro-drilled outer titanium surface adhesively bonded with an inner composite (segmented) structure, as can be observed in Figure 2. The purpose of applying HLFC technology on wing, empennage or nacelle is to reduce drag and consequently fuel-burn reduction.

![Figure 2. Model of the bonded thin titanium sheet (yellow) over the CFRP structure designed for Hybrid Laminar Flow Control (HLFC)](image)

The critical topic dealt within this research is exactly this titanium-composite adhesive joint. Strength of this joint must be investigated for certification and characterization of the adhesive properties is needed for future numerical simulation purposes. In the TicoAjo project cost effective industrial manufacturing processes of titanium-composite joints are investigated using pre-treatment studies. The hybrid material joints are extensively evaluated by testing under static, fatigue loads and at high strain rates by the partners NLR, TU Delft [1] and University of Patras. The environmental influences; CTD, ambient and HTW are investigated experimentally.

The adhesive properties are characterized and - this test data is used for future numerical simulation purposes in order to predict damage growth behavior on an aircraft sub-component level. This sub-component is a stiffened aircraft panel which will be experimentally verified to assess the validity of this prediction. The test-data and validated method obtained through TicoAjo will enable prediction of failure to aid the design and certification process.

In the research four different types of joints are investigated, hereafter called the union types. The first union type is based on a secondary bonding between the CFRP and the titanium. The FM94 adhesive is used for this type. The second union type uses co-bonding of the CFRP and titanium with a FM300 adhesive. The third union type is also co-bonding, however without an adhesive, hence the connecting is achieved by the RTM6 resin of the composite. The fourth union type is a thermoplastic material and secondary bonding using FM94 adhesive. The union types are summarized in the table below.
Table 2. **Union types**

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From these four union types the most optimal pre-treatment of the titanium provided by TU Delft and optimal union type is determined by means of experimental testing.
# Manufacturing

For the evaluation of the union types the coupons are designed together with the University of Patras and manufactured following the ASTM standard as close as possible. However, due to the low thickness and yield stress of the titanium, additional backing material for the DCB and ENF tests are needed. Otherwise the titanium would yield during the DCB and ENF tests and the fracture toughness measured will not be accurate. The coupons were manufactured according to the following material specifications. An overview of the coupon layers can be observed in figure 2 and indicated below.

- Titanium Grade 2 (CP40), 0.8 mm thickness
- CFRP adherent from HexFlow RTM 6 epoxy and HexForce G0926 fabrics. With a layup of [0/45]s
- Aluminium 2024 T3 backing plates (5.0 mm)
- Adhesives
  - Adhesive FM 94K, carrier: knit, areal weight 293 gsm (0.06 psf), nominal thickness 0.25 mm. Cure at 120 °C
  - Adhesive FM 300M, carrier: mat, areal weight 150 gsm (0.03 psf), nominal thickness 0.13 mm Cure with RTM 6 at 180 °C
  - Bonding aluminium with 3M Scotch-Weld 9323 B/A at RT after specimen machining 0.2 mm thickness
- Upilex-25S foil for starter crack (0.025 mm thickness). Both sides with Fre-Kote

The titanium sheets are pre-treated using cleaning, grit-blasting and UV/Ozone treated and for secondary bonding the CFRP plates are cleaned, grit-blasted and UV/Ozone treated. The hybrid plates are manufactured using the Vacuum Assisted Resin Transfer Moulding VARTM process. The aluminium backing is bonded onto the composite and titanium afterwards using the 3M Scotch Weld adhesive.

![Image of the coupon composite with the backing material, adhesives, titanium and composite. The blue layer is the Upilex foil. (Right) the VARTM process. (bottom image) Finished Ti-CFRP coupon without the backing plates where the curvature is clearly visible. The titanium is on the upper side, the CFRP on the lower side. The titanium has a larger coefficient of thermal expansion and shrinks more during cooldown.](image)
From initial calculations on the hybrid joint some thermal stresses were expected. After consolidation the thermal stress and consequent distortion of the plate was fairly obvious, see figure 2. This thermal stress is caused by the delta in the coefficient of thermal expansion of CFRP and titanium. Titanium wants to shrink more during cooling. The thermal stress leads to mode II shear loading on the joint interface. So consequently for post-processing of the test-results this effect needs to be taken into account. This effect is most apparent in the union-2 and union-3 cases where the co-bonding is applied and the delta in temperature is largest. For C-scan inspection the curved plates are compressed to be as flat as possible. Also for the application of the aluminium backing the curved plates are forced towards a flat geometry. This further increases the internal stress in the plates which should be accounted for in the post-processing of the test data.

The quality of the manufacturing Ti/CFRP plates is good as was shown by C-scan data, see figure 3. These plates are used to extract the coupons from using a waterjet cutting process after which the 5 mm thick aluminium backing plates are bonded on both sides.

Figure 3: Finished plate with six individual pre-treated titanium sheets. (Right) the c-scan results of the plate shows low void content and overall good quality. The plates for all union types are used to extract the hybrid coupons from
3 Testing DCB UBM

Testing is performed following the ASTM standard as close as possible using the standard DCB setup, see figure 4. Adjustments were made to enable the hybrid material testing. The coupon size is 25 mm width and 280 mm in length. The total thickness including the backing material is around 12.4 mm. Because the adherents including the backing material are thick, the expected loads are high. Therefore the DCB loading hinges are replaced by stronger components.

![Figure 4: Schematic view of the DCB samples. For the TicoAjo tests additional backing material is used and stronger hinges are used](image)

The DCB test is performed in three steps. First the starter crack is created initiating from the Upilex foil. Next a crack growth of 10 mm is established and the coupon is unloaded. Finally the crack length is progressed to 100 mm. For the ENF testing the load is applied using a three point setup. Load is applied to the central point until the first load drops occurs.

In order to understand the behaviour of the titanium and checking of the calculation, first several tests were performed without backing material. Plastic deformation of the titanium was seen, as expected, so indeed the aluminium backing plates are needed to allow for a valid test, see figure 5.

![Figure 5: DCB test of the union 3 coupon without backing plates. The titanium sheet is on the bottom side, the CFRP on the upper side of the specimen. As expected the thin titanium sheet quickly starts to deform plastically and therefore rendering the results to determine the fracture toughness values invalid](image)
**DCB test results**

The DCB tests with aluminium backing showed consistent results for the six specimen tested. The maximum load is at 400 N after which the load starts to decrease as the crack grows, see figure 6. This load drop is mainly caused by the increase of the effective arm length from the crack growth. The fracture toughness value for mode I, $G_{Ic}$ appeared to be quite low at 123 N/m and an thermal stress inherent mode II, $G_{IIc}$ of 25 N/m. Hence there was a mode mixity of 17.1 % during these tests. These values, however have a limited validity since during the testing it became clear that a debonding occurred at the interface together with a delamination in the CFRP, hence adherent failure together with adhesive bond failure. By this, two cracks started growing in the specimen and therefore making the fracture toughness calculation invalid. A similar behaviour occurred at all the specimen.

This indicates that although the starter crack with the Upilex foil is on the interface between the titanium and the CFRP, the interlaminar bonding strength in the composite is lower. This result is obtained with the optimized and best possible pre-treatment of the titanium. Although this a good indication for the adhesive bond strength, for the extraction of the fracture toughness values this possess a difficulty.

![Figure 6](image.png)

*Figure 6: (left) figure of the DCB setup with TicoAjo coupon. (right) the load – displacement curve of the six DCB coupons tested. The peaks in the beginning are the settling of the test-setup joints*

In the next step the ENF tests were performed using the pre-cracked DCB samples. These samples were already pre-cracked and in some cases also delaminations in the CFRP were present. The results of these tests were not satisfactory since a minimal load drop indicating the onset of damage growth is observed, see figure 7. First the behaviour is linear up to a load of 4 kN. Beyond 4 kN loading, the load-displacement curve becomes non-linear and a small load drop is observed. Afterwards the load increases further almost linearly. In the specimen a small crack growth could be observed which is, however very difficult to detect.
Figure 7: (left) the ENF test setup using the three point loading. The distance between the central point and starter crack is 35 mm. (right) the load – displacement curve where an atypical behaviour can be observed. This could be caused by the starter pre-crack and delaminations.

As a result of the ENF tests using the pre-cracked samples, further testing was performed on non-pre-cracked samples. The non-precracked samples were tested similar to the pre-cracked samples. A slightly different load-displacement curve was found, a small peak was found in the transition area between the two linear sections of the curve, see figure 8.

Figure 8: (left) Penetrant inspection result of ENF specimen tested just beyond the peak. Black marker indicates the end of the Upilex foil. (right) Non-precracked sample results of an ENF test.

To find out if the peak is caused by the crack initiation between the titanium and composite, a few tests were done which were stopped just after and just before the peak. The specimens were investigated with a microscope. Since the visibility of the cracks in ENF tested specimens is difficult, also penetrant inspections were done. The inspections showed that during the first linear part of the test, the Upilex starter crack is present and the crack between composite and titanium is initiated at the peak between the two linear parts of the crack. So the peak value can be used to calculate the fracture toughness in GIIc mode of the specimens. The second linear part of the load-displacement curve is most probably caused by bending of the specimens with a crack which is propagated beyond the mid loading point. Detailed analyses have to be made to prove this theory.
4 Outlook

The next steps in the project TICOAJO will focus on the durability of adhesive connection between titanium and CFRP. Also a novel moment loading test setup [3, 4] will be employed to enable a-symmetric laminates to be tested, see figure 9.

Figure 9: Moment loading cantilever beam setup. This will enable a-symmetric coupons to be testing in Mode I, Mode II and mixed modes with constant moment. The load is applied by the arms which are loaded by the steel cables.
5 Conclusions

Within the TICOAJO project the aim is to characterise the adhesive connection between titanium and composite material. Due to the limited titanium thickness in the design and the low yield stress (grade 2) and thermal stresses the characterisation using standard testing proved to be challenging. Additional backing material is needed to avoid plastic deformation and extracting correct fracture toughness values. Four different types of adhesive and joining options have been manufactured and evaluated using experimental testing.

The basis for the testing done was the standard DCB and ENF setup that has been modified to accommodate the a-symmetry of the samples and the large thickness due to the backing material. From the test results it showed that for most types of connection the adhesive and non-adhesive joint is very strong with the pre-treatments used. The composite itself appears to be the weak point and delaminations start to form during the DCB testing. Further research will be done in the TICOAJO project to investigate these effects including the use of the moment loading setup. Also the effect of environmental conditions and fatigue on the joint behaviour will be investigated. Also these aspects are greatly complicated by the need for the backing material.

Acknowledgements
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6 References


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