Report LR-423

DURABILITY OF ARALL

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ABSTRACT

In this paper the present developments in the ARALL durability program are described. The moisture absorption as well as the influence of moisture on the mechanical properties are examined. Apart from standard durability aspects, the fibre/adhesive adhesion and its role in the ARALL fracture mechanisms are discussed. The first results show a high environmental resistance of both fibres and adhesive.
<table>
<thead>
<tr>
<th>CONTENTS</th>
<th>page</th>
</tr>
</thead>
<tbody>
<tr>
<td>ABSTRACT</td>
<td>1</td>
</tr>
<tr>
<td>INTRODUCTION</td>
<td>3</td>
</tr>
<tr>
<td>1. MOISTURE ABSORPTION</td>
<td>4</td>
</tr>
<tr>
<td>2. INFLUENCE OF MOISTURE ON THE INTERLAMINAR SHEAR STRENGTH</td>
<td>5</td>
</tr>
<tr>
<td>3. SUSTAINED LOAD TESTING</td>
<td>6</td>
</tr>
<tr>
<td>4. BONDLINE CORROSION</td>
<td>6</td>
</tr>
<tr>
<td>5. ADHESION</td>
<td>7</td>
</tr>
<tr>
<td>6. RELAXATION DUE TO TEMPERATURE AND/OR MOISTURE</td>
<td>9</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENT</td>
<td>10</td>
</tr>
<tr>
<td>REFERENCES</td>
<td>10</td>
</tr>
<tr>
<td>TABLES</td>
<td>11</td>
</tr>
<tr>
<td>FIGURES</td>
<td>13</td>
</tr>
</tbody>
</table>
INTRODUCTION

A new family of structural composite materials is now being developed at the Department of Aerospace Engineering of the Delft University of Technology in cooperation with the Fokker Aircraft Company, the Dutch National Aerospace Laboratory (NLR), ENKA (producer of aramid fibres), 3M (producer of adhesives) and ALCOA (producer of aluminium alloys). The new family of materials is called ARALL (Aramid Aluminium Laminate) and is built up as laminated sheet material with (fig. 1):

1) thin high strength aluminium alloy sheets
2) strong unidirectional aramid fibres
3) epoxy metal adhesive system

ARALL was developed principally to obtain a material with a superior fatigue strength. This property is primarily obtained by a restraint on fatigue crack opening as the result of the presence of uncracked fibres in the wake of the crack (ref. 1). Especially when a favourable residual stress system is introduced in ARALL (the result of a prestrain process after curing), the material becomes almost fatigue insensitive (fig. 2 and 3).

ARALL is also a very strong material. Some typical properties are shown below:

<table>
<thead>
<tr>
<th></th>
<th>7H32 prestrained</th>
<th>2H42 as cured</th>
<th>7075-T6 monolithic</th>
<th>2024-T3 monolithic</th>
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<tr>
<td><strong>tensile strength</strong> (MPa/ksi)</td>
<td>735 (107)</td>
<td>590 (86)</td>
<td>560 (81)</td>
<td>470 (68)</td>
</tr>
<tr>
<td><strong>0.2% yield stress</strong> (ksi)</td>
<td>635 (92)</td>
<td>380 (55)</td>
<td>480 (70)</td>
<td>360 (52)</td>
</tr>
<tr>
<td><strong>Young’s modulus</strong> (ksi)</td>
<td>69000(10005)</td>
<td>70000(10150)</td>
<td>72000(10440)</td>
<td>72000(10440)</td>
</tr>
<tr>
<td><strong>proportional limit in compression</strong> (ksi)</td>
<td>355 (52)</td>
<td>255 (37)</td>
<td>480 (70)</td>
<td>270 (39)</td>
</tr>
<tr>
<td><strong>elongation</strong> (%)</td>
<td>1.9</td>
<td>2.4</td>
<td>11</td>
<td>17</td>
</tr>
<tr>
<td><strong>density</strong> (10^3 kg/m^3)</td>
<td>2.35</td>
<td>2.44</td>
<td>2.8</td>
<td>2.8</td>
</tr>
</tbody>
</table>

In the table, the following code is used to define the ARALL configuration:

A B C D
- the first symbol defines the aluminium alloy
  7    7075 T6
  2    2024 T3

- the second symbol defines the aramid fibre
  H: high modulus fibre
  I: intermediate modulus fibre

- the third symbol defines the thickness of the individual aluminium sheets (10^{-1} mm)

- the fourth symbol defines the number of aluminium layers

In spite of the use of structural fibres in ARALL, nearly all advantages of metals over pure composites are preserved such as plasticity, formability, easy machining and impact strength but with the lower density and superior fatigue behaviour common to composites (ref 2).

In this paper, some durability aspects are discussed including moisture absorption, bondline corrosion and the influence of moisture and temperature on some mechanical properties. In this context, the fibre/adhesive adhesion is also considered. A survey of the durability test programmes running at the Delft University is shown in table 1. All specimens used in the test series are fully pretreated according Fokker specifications which includes:
- degreasing
- pickling
- chromic anodising
- priming

1. MOISTURE ABSORPTION

According to Fick's second law (non steady state condition), the diffusion of moisture can be described by:

\[
\frac{\delta c}{\delta t} = D_x \frac{\delta^2 c}{\delta x^2} + D_y \frac{\delta^2 c}{\delta y^2} + D_z \frac{\delta^2 c}{\delta z^2}
\]  

(1)

In the case of ARALL (aluminium sheets on the outer parts), equation 1 can be reduced to:

\[
\frac{\delta c}{\delta t} = D_y \frac{\delta^2 c}{\delta y^2} + D_z \frac{\delta^2 c}{\delta z^2}
\]

Because of the presence of unidirectional, moisture absorbing fibres in the bondline, a limitation to a onedimensional approach is not allowed.

The unknown diffusion coefficients \(D_x\) (diffusion coefficient in the fibre direction; \(D_z\): diffusion coefficient perpendicular to the fibre direction) can be experimentally obtained by means of weight gain tests. For these experiments, elongated I.L.S.-specimens, submerged in distilled water are used (fig 4). Some results of the weight gain tests are shown in fig 5. Because of the two-dimensional approach, for every test temperature two different specimen configurations are necessary. A survey of the diffusion coefficients as obtained from the tests is shown in table 2. It is noteworthy that the diffusion perpendicular to the fibre direction is about one third of the diffusion in fibre direction. Using the Arrhenius equation, the diffusion coefficients can be found for arbitrary temperatures (fig 6).
With adequate boundary conditions, equation 2 can be solved:

\[
\frac{c_{\infty} - c}{c_{\infty} - c_0} = \frac{16}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \sin \left(\frac{(2n+1)\pi y}{L}\right) \sin(2n+1) \frac{\pi z}{D} \cdot e^{-\left(\frac{(2n+1)^2}{b^2}\right) \cdot t}.
\]

or written as moisture contents:

\[
\frac{M_{\infty} - M}{M_{\infty} - M_0} = \frac{64}{\pi^4} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^4} \cdot e^{-\left(\frac{(2n+1)^2}{b^2}\right) \cdot t}.
\]

The concentration distribution in a specimen as a function of time and temperature can be shown in the so-called moisture profiles using equation 3 (fig 7).

The saturation period as a function of temperature can be found using equation 5 (ref 3):

\[
t = \frac{1}{\pi^2} \left(\frac{D_y}{\ell^2} + \frac{D_z}{b^2}\right)^{-1} \left(0.420 + \ln \left(1 - \frac{M}{M_{\infty}}\right)\right)
\]

Some typical results are shown in table 2.

Concluding, because of its configuration (aluminium sheets on the outer parts), the moisture pick-up of ARALL is very small as compared to aramid reinforced or even carbon reinforced composites. These composite materials have large unprotected areas open for moisture absorption. For ARALL, moisture absorption is limited to the edges of the material.

2. INFLUENCE OF MOISTURE ON THE INTERLAMINAR SHEAR STRENGTH

Two different test series have been executed:

1) Exposure to salt water (65°C) or to saltspray (35°C) followed by three point bending tests after specified exposure periods

2) Distilled water exposure of I.L.S.-specimens up to specified moisture content levels followed by a three point bending test

The equation to calculate the I.L.S.-strength of ARALL is corrected for (ref 4):

- moment of inertia of a non-homogeneous cross-section
- maximum shear stress not in the plane of symmetry

\[
I.L.S. = \frac{3P_{\text{max}} (t_1^2 - t_3^2)}{8b ((t_1^3 - t_2^3 + t_3^3) + \frac{E_{ar}}{E_{al}} (t_2^3 - t_3^3))}
\]

All results are the average of at least six specimens.

To avoid edge effects in the fracture mechanism, the length of the I.L.S. specimen is increased to 50 mm (2.5 times the standard I.L.S.-specimen length).

The results of the first test series are shown in fig 8. The I.L.S.-strength of the specimens exposed at 65°C is somewhat higher, probably because of
plasticizing effects. The overall environmental influence on the I.L.S.-strength is small up to 8 weeks of exposure for both temperature levels.

The results of the second test series are shown in fig 9. There is a small decrease in I.L.S.-strength with increasing moisture content; the stiffness is hardly influenced.

Apart from these experiments the influence of several pretreatments (Fokker specifications) on ARALL is examined using the three point bending test. It is well possible that, because of bonding processes, ARALL sheets have to be pretreated during which moisture absorption is possible. The results are shown in fig 10. The I.L.S.-strength is not influenced even if the duration of every step in the pretreatment process is doubled (ref 4).

3. SUSTAINED LOAD TESTING

A test series is started to obtain a sustained load curve for ARALL. A possible curve is shown in fig 11. The curve is obtained by plotting the time to failure of specimens at different constant load levels. The variables in the test series and the first results are shown in table 3. Because of the specimen configuration (in the centre of the specimen the metal sheets are interrupted whereas the fibres are continuous; see fig 11), this test is mainly an indication for the sustained load capacity of the aramid fibres in different environments but, at the central edges, also the adhesive system is severely exposed (environment + shear stress peaks).

The first results show that both fibres and adhesive system seem to have an extreme environmental resistance for this kind of loading condition. The few failed specimens ($80\% P_{ult}$; 25 and 65°C) showed a fibre-dominated fracture and three typical delamination zones (fig 12).

4. BONDLINE CORROSION

Bondline corrosion is a typical real service type of fracture, characterized by debonding at the aluminium/adhesive interface followed by severe corrosion processes on the aluminium. In this test series Bell-peel specimens are exposed to two different environments:
- salt spray exposure (35°C, continuous)
- outdoor exposure

As a reference, two different pure adhesive systems (AF 163-2 and Redux 775) have been added to the test series.

The results of the salt spray exposure are shown in fig 13 (peel strength) and fig 14 (peeled surface). The low peel strength of ARALL is remarkable and will be discussed in chapter 5 (ADHESION). There is, for the three systems, a continuous decrease in peel strength with increasing exposure period. The peeled surface do not show any bondline corrosion up to 15 months of salt spray exposure. For ARALL (least influenced by the exposure) it is most probably the result of the dominant role of the aramid fibres in the moisture absorption process whereby the moisture is fixed in the fibres and not conducted towards the aluminium/adhesive interface.

In spite of full pretreatments, severe corrosion was encountered on the outside surfaces of the aluminium adherends. But then the fibre/adhesive
interface acts as a corrosion stopper and no through-the-thickness corrosion was observed for all specimens (fig 15).

Only the first results are available for the outdoor exposure (1 year). This exposure period does not influence the peelstrength and no bondline corrosion is observed.

Apart from this test series, ARALL peel specimens were exposed to hydraulic oil used in aircraft systems (tradename SKYDROL). The first results (up to 12 weeks of exposure) did not show any influence of the environment on the peelstrength or peeled surface.

5. ADHESION

For laminated structures, the interfaces mostly have a determining influence on the behaviour of the material, especially under environmental conditions. For ARALL, 2 different interfaces should be considered:

1. aluminium/adhesive interface: much theoretical and experimental work is performed in the past to obtain acceptable adhesion between aluminium and adhesive system. This work led to the use of anodising and primer systems to improve the adhesion at this interface.

2. aramid/adhesive interface: because of the relatively slow breakthrough of aramid as a structural material, less knowledge is obtained about this interface and about methods (sizing a.o.) to improve the adhesion at this interface.

Although interfacial debonding can occur under different loading conditions, it is convenient to study the adhesion under a mode 1 loading (loading perpendicular to the fibre direction) which includes three well-known test methods:

1. Bell-peel test
2. Wedge-edge test
3. W.T.D.C.B. test

In these tests, fracture is primarily caused by tensile stress and not by shear stresses. Some results obtained with each test method will be briefly discussed.

1. Bell-peel test: In addition to the bondline corrosion tests (chapter 4) a series of experiments was performed to investigate the fracture behaviour of ARALL in this type of mode 1 loading. In an early stage it was already clear that the peelstrength of ARALL was low compared to "pure" adhesive systems (e.g. fig 13). This is also shown in fig 16 in which the peelstrength is plotted as a function of the volume fraction on the fibres. In fig 16 three different fracture behaviours are indicated (ref 5):

1. ARALL in its original composition; lowering of the fibre volume fraction gives only a small increase in peelstrength

2. ARALL in an adjusted condition (the fibres are locally situated outside the centre of the prepreg by adding an extra adhesive film); the peelstrength is doubled because of the existence of a second crack front.
3. at a very low volume fraction of the fibre, ARALL behaves as a pure adhesive with cohesive crack growth in the resin and a peel-strength comparable with pure adhesives. In this condition (fibre volume fraction beneath 15%) ARALL looses its superior fatigue behaviour and is therefore not of interest in the current applications.

Close examination of the fractured surfaces showed that insufficient adhesion between the aramid fibre and adhesive (fig 17) is the determining factor for the low peel-strength of ARALL. The fibre/adhesive interface is for this loading condition definitely the weakest link.

The peeltwist however is not a reliable quantitative test method for comparing different systems because of the considerable plastic deformation of the thin adherend. The amount of fracture energy, consumed by plastic deformation of the thin adherend is different for all adhesive systems (plastic deformation is a function of the brittleness of the adhesive and the strain to failure of the adhesive perpendicular to the fibre direction) and therefore the energy really available for crack growth cannot be easily determined.

2. Wedge-edge test: This test method is initially developed by Bethune at the Boeing company and was originally used as a qualitative test for pretreatments. Although the weakest link is different for ARALL, this test is also very useful to investigate the fibre/adhesive adhesion, but again on a qualitative base only. In these experiments, the strain energy release rate G is used as a parameter instead of the crack length a. G is calculated as:

\[ G = \frac{d^2 E h^3}{16} \cdot \frac{(3(a+0.6 h)^2 + h^2)}{((a+0.6 h)^3 + ah^2)^2} \]

In fig. 18, the results of ARALL are again compared with a pure adhesive. The initial threshold energy for crack growth is definitely lower than for the adhesive and also the decrease of the threshold value due to environmental exposure (saltspray) is larger for ARALL than for the adhesive. Also the time to reach G\textsubscript{isc} is significantly longer (ref 6). Examination of the cracked surfaces showed that the fibre/adhesive interface is the weakest link both in initial and environmental condition. The energy necessary for crack growth at the fibre/adhesive interface is therefore significantly lower than the necessary energy for cohesive crack growth in the adhesive.

Prestraining ARALL has also a significant effect on the G-level (decreasing G with increasing prestrain level; fig 19). There is no difference in environmental influence between specimens with different prestrain levels.

Although the disadvantage of the peeltwist (varying plastic deformation) is not present, a fracture mechanics approach is extremely difficult. The strain energy release rate G is a function of the compliance of the specimen (or better; a function of the change of compliance with crack length; \( G = f(dC/da) \)). For the wedge-edge specimen this function is unknown and cannot be obtained by experiments (unloading and reloading at arbitrary cracklengths is not possible). Because of the specimen configuration, dC/da will be negative (which explains the existence of the threshold G-level) but no detailed descrip-
tion of the function is available.


For this specimen the following relation can be derived:

\[ G = \frac{P^2}{2hd} \cdot \frac{dc}{da} \]

In this case \( \frac{1}{2h} \cdot \frac{dc}{da} \) is constant and \( G \) is only a function of the applied load.

A test series is performed including 3 different adhesive systems (AF 163-2, aramid + AF 163-2 and carbon + AF 163-2), different displacement rates and loading conditions (static + fatigue).

As could be expected for the ARALL specimens (aramid + AF 163-2), fracture occurs at the fibre/adhesive interface which is in agreement with the other test methods. Some results are shown in fig. 20. The energy necessary for crack growth of ARALL is considerably lower than for the carbon reinforced and pure adhesive system. It might be surprising that in a typical fracture-toughness test like the W.T.D.C.B.-test carbon reinforcement shows to be superior compared to aramid. One has to consider that toughness in this test is mostly depending on the adhesive system and interfacial adhesion. The fibres themselves are not loaded to failure and therefore the brittle character of the carbon fibre is not present (its superior adhesion to the adhesive system leads to the higher values). It is obvious that in an in-plane fracture toughness test, aramid is far superior to carbon because of its higher strain to failure. Also in an in-plane toughness test, extreme interfacial adhesion will result in an undesired brittle fracture mechanism. It is therefore not desirable to look for maximum adhesion but a sort of optimum has to be found.

In fig 20; there is a significant rate dependency for the reinforced adhesives but not consistent (increasing \( G \) for aramid; decreasing \( G \) for carbon with increasing displacement rate). A few specimens are tested, submerged in distilled water. The energy for crack growth is increased for the pure adhesive (plasticizing effects) but decreases for the fibre reinforced adhesives probably because of disturbance of interfacial bonds.

6. RELAXATION DUE TO TEMPERATURE AND/OR MOISTURE

To obtain the superior fatigue behaviour of ARALL a prestrain process after the curing cycle is necessary to create the favourable internal stress distribution (tension in the fibres; compression in the aluminium). It is obvious that this stress distribution should remain during the life time of the material.

To examine possible relaxation processes a test series is started in which specimens are exposed to a whole range of temperatures, environments and external loading conditions.

Preliminary results are shown in fig 21. No significant relaxation is encountered for all test temperatures, environments and external loading conditions up to 12 weeks of exposure.
ACKNOWLEDGEMENT

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6 - Verbruggen M.L.C.E., Report, Delft University of Technology, Department of Aerospace Engineering, February 1983
<table>
<thead>
<tr>
<th>PROJECT</th>
<th>TYPE OF SPECIMEN</th>
<th>PROGRESS</th>
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<tbody>
<tr>
<td>diffusion properties of ARALL</td>
<td>elongated I.L.S.-specimen</td>
<td>ISO9000.A.: finished</td>
</tr>
<tr>
<td>bondline corrosion of ARALL</td>
<td>Bell-peel specimen</td>
<td>other R.N.: in progress</td>
</tr>
<tr>
<td>sustained load testing</td>
<td>delamination specimen</td>
<td>saltspray: finished</td>
</tr>
<tr>
<td>environmental influences on the I.L.S.-properties</td>
<td>RAAB-specimen</td>
<td>outdoor: in progress</td>
</tr>
<tr>
<td>relaxation of internal stresses</td>
<td>thick adherend specimen</td>
<td>in progress</td>
</tr>
<tr>
<td>due to temperature and environment</td>
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<td>finished</td>
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<tr>
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<td>relaxation specimen</td>
<td>in progress</td>
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<tr>
<td>influence of SKYDROL on the overall durability properties</td>
<td>stiffened panel compression specimen</td>
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<td>influence of prestrain level on the crack propagation during mode 1 loading</td>
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<td>environmental fatigue</td>
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<td>adhesion (fibre/adhesive interface)</td>
<td>wedge-edge specimen</td>
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<td>influence of environment on adhesion</td>
<td>W.T.D.C.B. general</td>
<td>in progress</td>
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</table>

Table 1: Survey of the durability test program

I.L.S.: interlaminar shear strength
W.T.D.C.B.: width tapered double cantilever beam
Table 2: Diffusion coefficients and saturation periods for different temperatures

<table>
<thead>
<tr>
<th>Temperature</th>
<th>D_{YT}</th>
<th>D_{ZT}</th>
<th>D_{YT}/D_{ZT}</th>
<th>t_{50%M1}</th>
<th>t_{99%M1}</th>
</tr>
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<tbody>
<tr>
<td>°C</td>
<td>mm²/s</td>
<td>mm²/s</td>
<td></td>
<td>days</td>
<td>days</td>
</tr>
<tr>
<td>5</td>
<td>9.576</td>
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<td>2.8</td>
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<td>50520</td>
</tr>
<tr>
<td>20</td>
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<td>1281.0</td>
<td>3.3</td>
<td>7.5</td>
<td>115</td>
</tr>
</tbody>
</table>

D_{YT}: diffusion coefficient in fibre direction.
D_{ZT}: diffusion coefficient perpendicular to the fibre direction.
M_{m}: maximum moisture content.

Table 3: First results of the sustained load test

<table>
<thead>
<tr>
<th>sustained load level</th>
<th>Pult</th>
<th>temperature environment °C</th>
<th>time to failure days</th>
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<tr>
<td>kgf</td>
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<td></td>
<td></td>
</tr>
<tr>
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<td>65</td>
<td>1</td>
</tr>
<tr>
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<td>80</td>
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<td>20</td>
<td>25</td>
<td>&gt;517</td>
</tr>
<tr>
<td>720</td>
<td>60</td>
<td>SKYDROL</td>
<td>&gt;141</td>
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Fig 1 Cross-section of ARALL sheet

Fig 2 Crack propagation rates in "standardized" ARALL sheet material
Fig. 3 Comparison of fatigue lives. 
Results of constant amplitude test on ARALL and monolithic 2024-T3 riveted joints.

Fig. 4 Built-up I.L.S.-specimen.
Fig. 5. Weight gain of ARALL specimens as a function of fibre orientation and temperature.

Fig. 6. Diffusion coefficients parallel and perpendicular to the fibre direction for arbitrary temperatures.
Fig. 7: Moisture profile of an ARALL specimen with fibres in width direction after 100 days of immersion at 60 °C. Because of symmetry, only one quarter of the specimen is shown.

Fig. 8: Influence of salt water and salt spray exposure on the I.L.S.-strength of ARALL.
Fig. 9  Influence of moisture absorption on the I.L.S. strength and stiffness

Fig. 10  Influence on the I.L.S.-strength of various pretreatments on ARALL
Fig. 11: Sustained load strength of ARALL delamination specimens.

Fig. 12: Typical delamination zone's after failure of the specimen as a result of environment and loading condition.
Fig 13 Influence of salt spray exposure on the peel strength of 3 adhesive systems.

[35°C; continuous exposure]

Fig 14 Peeled surface of ARALL specimens after different periods of salt spray exposure
Fig 15 Corrosion stopping behaviour of an adhesive layer

Fig 16 Relation between the peel strength and the volume fraction of the fibre.
Fig 17 Insufficient adhesion between aramid fibre and adhesive
photo: Enka research
magnification: 2400x

Fig 18 Influence of saltspray exposure on the energy release rate
of ARALL compared to a pure adhesive.
Fig. 19 Influence of prestrain level on the energy release rate $G_\Sigma$.

Fig. 20: $G_\Sigma$ for different adhesive systems.

- after sustained load (500 N) for 91 h.
- test performed in distilled water.
Fig. 21  Relaxation due to temperature, environment and external loading condition, exposure period: 8 weeks.