POLYCARBONATE-TO-POLYCARBONATE SINGLE LAP JOINTS WITH POLYURETHANE FILM ADHESIVE

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Abstract
This paper discusses the mechanical performance of autoclave-bonded polycarbonate single lap joints (SLJs) using polyurethane film adhesive. Joint static load transfer capacity (LTC) data is analyzed for test samples after they have been heat cycled in an environmental chamber at either ambient low humidity or a high level of relative humidity at 85 %. The LTC data and failure mode of environmentally-cycled samples are compared to those for baseline test samples that are as they are after autoclave bonding. LTC data is generated by tensile-shear testing using Material Testing System (MTS).

Heat-only cycled SLJ have exhibited 23 % increase in their LTC as compared to their baseline LTC, however, the LTC of test samples that have been heat-cycled at high relative humidity has been reduced by 48 % from the baseline value of LTC. Detailed data analysis and failure mode discussion are provided.

Introduction
Adhesively bonded joints have become one of the popular methods as joining and fastening methods for many different combinations of materials. Adhesive is widely used in various structural and mechanical applications due to their relative strength-to-weight ratio which leads to weight reduction as well as the energy savings. Joints are often the weakest link in a design, therefore in-depth analysis with experimental validation of the joint performance and reliability become crucial especially when the joint adherends and adhesive are exposed to heat and moisture.

Awaja and et. al. [1] and Baldan [2] reviewed research articles relating to the adhesion of polymers which has been performed by numerous researchers. They summarized the adhesion phenomena, systems, mechanisms, surface characterization, as well as theories including mechanical interlocking modeling, adsorption theory, diffusion theory, chemical bonding theory and others. Quini and Marinucci [3] studied polyurethane liquid adhesive that is used in automotive composite joints. They performed a comparative investigation between the usage of adhesive and mechanical fasteners and found that use of adhesive with composite materials is more efficient than the other.

Hu and et. al. [4] carried out a study of the strength prediction of bonded joints under cyclic temperature loading whose profile fluctuates between -30 °C and 80 °C for 2 hours using cohesive zone model. Steel and aluminum were selected as adherends, and one brittle adhesive, and one ductile kind were used as adhesive. Later, the single lap joints were tested for their load-displacement relationship to compare, and Scanning electron microscopy (SEM) was also utilized to investigate the fracture surfaces. The environmental degradation factor was inserted to ABAQUS modeling, and it effectively predicted the degradation process that was 2.7 % and 5.5 % for steel and aluminum,
respectively.

Sakai and Nassar [5] studied the failure mode of adhesively bonded composite single lap joints under static tensile shear loading, after the samples have been subjected to cyclic heat at high relative humidity. In this paper, lightweight multi-material combinations that include Composite/Aluminum, Composite/Magnesium, and Composite/Composite are investigated, and changes in static load transfer capacity are investigated considering the failure analysis of fracture surfaces using both Scanning Electron Microscope (SEM), Energy Dispersive Spectrometry (EDS), Optical Metallography, as well as the Finite Element Analysis (FEA) data.

This paper not only discusses the mechanical performance of SLJs also investigates a failure prediction model when joint is cohesively failed by utilizing Hart-Smith model.

**Experimental Methods**

**Materials and Joint Preparation**

Polycarbonate (PC) and film polyurethane adhesive (PE 399) is used for the joint coupons for each single lap test. The material properties and its recommended curing process is summarized in Table I and II. The prepared joints are carefully placed in autoclave as shown in Figure 1, and Figure 2 is used as an actual bonding procedure in autoclave for the single lap polycarbonate-to-polycarbonate joints. Figure 3 shows the accrual joint curing process profile performed in autoclave. Total of three replicas for each environmental condition are prepared.

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**Table I: Adherend and adhesive material properties [6, 7]**

<table>
<thead>
<tr>
<th>Material Properties</th>
<th>Ultimate Strength (MPa)</th>
<th>Modulus of Elasticity, E (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polycarbonate</td>
<td>70</td>
<td>2300</td>
</tr>
<tr>
<td>PE 399</td>
<td>45</td>
<td>na</td>
</tr>
</tbody>
</table>

**Table II: Suggested Laminating Parameters by Manufacturer**

<table>
<thead>
<tr>
<th>Suggested Laminating Parameters by Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultimate Temperature</td>
</tr>
<tr>
<td>110 °C – 130 °C</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>------------------</td>
</tr>
<tr>
<td>Ultimate Pressure</td>
</tr>
<tr>
<td>Soaking Time</td>
</tr>
</tbody>
</table>

Figure 1: Schematic of a test SLJ.
Figure 2. Sample preparation for single lap joints in autoclave.

Figure 3. PE 399 polyurethane curing process profile in Autoclave

Experimental Procedure and Test Setup

Environmental Testing

Three different environmental conditions are investigated for their effect on the static performance of SLJ; namely, ambient environmental condition as a baseline reference, cyclic heat only loading, and cyclic heat at 85 % relative humidity as shown in Table III
and figure 4 on film adhesive SLJs.

Table III: Environmental Testing Conditions

<table>
<thead>
<tr>
<th>Environmental Condition 1</th>
<th>Environmental Condition 2</th>
<th>Environmental Condition 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ambient Environment</td>
<td>Cyclic Temperature Only</td>
<td>Cyclic Temperature + 85% RH</td>
</tr>
</tbody>
</table>

(a) Environmental Condition 2
(b) Environmental Condition 4

Figure 4. Cyclic temperature and constant relative humidity profiles (two cycles).
Static Load Transfer Capacity Testing

All the test joints are securely gripped vertically on MTS system to investigate their load transfer capacity (LTC). A quasi-static axial load is applied to the joint at a constant displacement speed of 1.27 mm/min per ASTM D1002 [8]. LTC tests were performed within twenty-four hours from the completion of an environmental test. Schematic of tensile shear test is shown in Figure 5.

Characterization of Polyurethane PE 399 by Dynamic Mechanical Analyzer (DMA)

Polyurethane samples used in the test bonded SLJs that are cured by autoclave, are characterized by Dynamic Mechanical Analyzer (DMA) using the Film-Tension fixture (Fig. 7). A polyurethane specimen is securely gripped on a fixed clamp as shown in Figure 8 and movable clamp on a DMA film-tension fixture, and a constant displacement rate is applied on a specimen to obtain stress-strain behavior of a polyurethane material.

Figure 5. Schematic of tensile shear test.
Figure 7. Dynamic Mechanical Analyzer (DMA)

Figure 8. DMA test setup
Hart-Smith Modeling [9, 10]

Closed form solution for the shear stress \( \tau \) and peel stress \( \sigma \) in single lap joints is given by Hart-Smith, for the bonded single lap model as follows,

\[
\tau = A_1 \cosh(2\lambda' x) + A_2
\]

\[
\sigma = A_3 \cosh(\chi x) \sin(\chi x) + A_4 \sinh(\chi x) \cos(\chi x)
\]

Here the constants \( A_1, A_2, A_3, A_4 \) are given by

\[
A_1 = \frac{G_a}{t_a Et} \left( \frac{P}{b} + \frac{6(1 - v^2)M}{t} \right) \frac{1}{2 \lambda' \sinh(2 \lambda' c)}
\]

\[
A_2 = \frac{1}{2c} \left( \frac{P}{b} - 2 \frac{A_2}{2 \lambda'} \sinh(2 \lambda' c) \right)
\]

\[
A = \frac{E_a M [\sin(\chi c) - \cos(\chi c)]}{t_a D_a \chi^2 e(\chi c)}
\]

\[
B = \frac{E_a M [\sin(\chi c) + \cos(\chi c)]}{t_a D_a \chi^2 e(\chi c)}
\]

Parameters \( \lambda', M, D, \) and \( \chi^4 \) are given as follows

\[
\lambda' = \sqrt{\left( \frac{1 + 3(1 - v^2)}{4} \right) \frac{2G_a}{t_a Et}}
\]

\[
M = \frac{P}{b} \frac{(t + t_a)/2}{1 + \xi c + \frac{1}{6} \xi^2 c^2}
\]

\[
\xi = \frac{P}{b D}
\]
\[ D = \frac{E_{PC}t_{PC}^3}{12(1 - \nu^2)} \]  

(1f)

\[ \chi^4 = \frac{E}{2D_a t_a} \]  

(2a)

Where \( P \) is the tensile-shear load and \( b \) is the width of adherend and adhesive. \( E_a \) and \( E \) are respectively the Young's Moduli for the adhesive and adherends' materials; \( G_a \) and \( G \) are the respective shear moduli; \( \nu_a \) and \( \nu \) are the respective Poisson ratios. \( t_a \) and \( t \) are the thicknesses of the adhesive layer and the adherends; and \( c \) is the half of the bondline length that extends on both sides after the origin \((-c \leq x \leq c)\).

The Hart-Smith solution for peel and shear stresses (Eqs. 1 and 2) assumes linear elastic behavior of all materials; it also assumes that both adherends are identical in terms of materials.

Results and Discussions

In this section, adhesively bonded polycarbonate-to-polycarbonate using film adhesive test data are presented and discussed. The average quasi-static load-elongation data of the SLJs followed by each environmental effect are collected and presented. The 1-\( \sigma \) varies from 0.03 % to 25.00 % from three samples of each test. Figure 9 shows the photographed images of the overlap area of the bonded SLJs before and after environmental testing prior to the LTC testing. It is observed that the number of bubbles as an indication of delamination of bonded layers. This phenomenon occurs when there is an increase level of the relative humidity due to moisture diffusion into polyurethane interlayer from cyclic thermal loading. The gas trapped inside of the bubbles is carbon dioxide \([11]\) because of the rearrangement of chemical formulation.

The instantaneous side view Images are captured (Fig. 9) while being under tensile-shear loading to measure their peel strength of the SLJs. The information is later correlated and compared to the Hart-Smith model for the joint failure prediction. Each load-elongation curve are shown in Figure 11 with annotation where peel begins and joint failure occurs. The failure modes because of LTC are tabulated in Table IV along with LTC data.

Environmental Effect on Failure Mode of SLJs

As shown in a summarized LTC data and its failure mode in Figures 10, 11 and Table IV, a failure mode characteristic of a bonded SLJ without environmental effect was different from all others as polycarbonate adherend began to fail before a joint did. This phenomenon explains that adhesion strength between two adherends overcome the
tensile yield strength of polycarbonate material. The polycarbonate adherend started to stretch after reaching its yield point at 413 seconds into LTC testing, and bonded area was at 3.5 degrees of an angle. By contrast, the failure mode of the bonded SLJ was shifted to the interfacial failure with 3 mm wide cohesive failure around the perimeter of the bonded area when they had been exposed to constant temperature profile for three days at 80 °C.

Figure 9 and 10 show the inspection photos of bonded area before and after environmental loading and the side view of the bonded are of SLJs when one adherend either started to fail or peel. The test result demonstrated that more bubbles observed from the interlayer, smaller angle of the bonded area and smaller LTC required for the first phase of the joint failure begin.

Figure 11 presents the magnified images of cohesively failed SLJs near edges of the bonded area. It is clearly shown that the failure modes to be more likely to be cohesive failure when bubbles form within interlayer in contrast to interfacial failure without bubbles.

Figures 12 through 14 show a comparison of elongation and maximum LTC for each environmental condition. The SLJ data with exposure of both cyclic heat and high relative humidity show by far the least value of LTC yet the longest elongation by 41 % and 22 %, respectively. This decrease in LTC is due to absorption of moisture into a polyurethane adhesive chemical structure yet elongation increased due to hyper elasticity as becoming a rubber-like material.

![Figure 9](image9.png)

**Figure 9. Photos of the PC overlap area comparison** (a) Baseline image before environmental effect, (b) image after an exposure to constant temperature profile, (c) image after effect of cyclic heat only, and (d) image after effect of cyclic heat and RH 85%.
(a) (No Environmental Effect) after 413 seconds (LTC at 8750 N, Peel Strength at 535.2 N, 10.53 N/mm)

(b) (Constant Heat Effect) after 380 seconds (LTC at 7750 N, Peel Strength at 474.0 N, 9.33 N/mm)

(c) (Cyclic Heat Effect) after 265 seconds (LTC at 7200N, Peel Strength at 251.4 N, 4.95 N/mm)

(d) (Cyclic Heat and RH 85 % Effect) after 180 seconds (LTC at 3000N, Peel Strength at 94.28 N, 1.856 N/mm)

*Figure 10. Images of a bonded joint when separation begins*
Figure 11(a) Detailed images of a fractured surface of a SLJ after tensile-shear testing (no environmental exposure) by microscope and magnification at x30

Figure 11(b) Detailed images of a fractured surface of a SLJ with cyclic heat only exposure after tensile-shear testing by microscope and magnification at x30

Figure 11(c) Detailed images of fractured surface of a SLJ with cyclic heat at 85 % relative humidity exposure after tensile-shear testing by microscope and magnification at x30
Figure 12 Load-Elongation curves

Figure 13 Maximum LTC Comparison data
Stress Distribution using Hart-Smith model and DMA Data

Utilizing Hart-Smith model and material properties obtained from DMA (Fig. 15), the curves of peel strength per unit width over the overlap length by Hart-Smith and experiments are shown in Figure 16. It shows the Hart-Smith model is predicting actual peel strength values to be are 50 % and 37 % higher for the baseline SLJ at maximum LTC and SLJ that was subjected to constant heat loading at the beginning of peel, respectively. However, the actual experimental data is 29 % and 70 % lower than that of predicted values for the SLJs that had been exposed to cyclic temperature and cyclic temperature with RH 85%, respectively.
Figure 15 (a) Stress Strain Baseline DMA Data for PE 399

Figure 15 (b) Stress Strain DMA Data for PE 399 with Cyclic Heat Effect
Figure 15 (c) Stress Strain DMA Data for PE 399 with Cyclic Heat and RH 85 % Effect

Figure 16 Stress Distribution over the Overlap Length when Peel Begins for the SLJ with Cyclic Heat and High Relative Humidity
Summary and Next Steps

This experimental study investigates the effect of cyclic heat and humidity, as well as the various joining methods on the static load transfer capacity of polycarbonate-to-polycarbonate single lap joints using polyurethane film adhesive. Investigated joining method includes bonding-only using autoclave process. Cyclic heating between 20\(^\circ\) C and 80\(^\circ\) C in coupled with two levels of relative humidity; namely, ambient level with 20 % RH and high level of 85 %. The conclusion of this study is summarized as follows;

• The young modulus of polyurethane with effect of cyclic heat and high relative humidity is only one fifth of that of baseline and a quarter of that with cyclic heat effect
• The overlap area shows most amount of bubbles with effect of cyclic heat and high relative humidity that results with cohesive failure after tensile-shear testing
• 40 % LTC reduction with effect of both cyclic heat and high relative humidity at 85 % as compared to the baseline value
• SLJs with both cyclic heat and high relative humidity effect have the highest elongation that is 22 % higher than that of baseline and 37.5 % higher than that with cyclic heat effect only
• No change in LTC between the SLJs with either baseline or cyclic heat effect

Next step as in continuation of this study is as observation and failure analysis are presented, further explanation of cohesively failed SLJ is necessary. As seen by utilizing Hart-Smith model, the shear failure begins at 1.19 MPa at the end of overlapped area. This value should be investigated in depth as in comparison with both actual material and experimental data.
References


