NATIONAL TRANSPORTATION SAFETY BOARD

Office of Research and Engineering
Materials Laboratory Division
Washington, D.C. 20594

July 1, 2003

MATERIALS LABORATORY FACTUAL REPORT Report No. 02-082

A. ACCIDENT

Place	: Belle Harbor, New York
Date	: November 12, 2001
Vehicle	: Airbus A300-600, N14053
NTSB No.	: DCA02MA001
Investigator	: Brian Murphy, AS-40

B. COMPONENTS EXAMINED

The vertical stabilizer and the rudder skin panels.

C. ACCIDENT SUMMARY

On November 12, 2001, at approximately 0917 EST, American Airlines flight 587, an Airbus A-300-600, N14053, crashed into a neighborhood in Belle Harbor, New York, several minutes after taking off from Kennedy International Airport. The airplane was on a scheduled flight to Santo Domingo, Dominican Republic. All 260 persons aboard the airplane were fatally injured, as were five on the ground.

D. DETAILS OF THE EXAMINATION

This report documents the materials testing and microstructural examination of composite materials from the vertical stabilizer and rudder. The testing and examination were completed primarily at the National Aeronautics and Space Administration's Langley Research Center (NASA Langley) in Hampton, Virginia. Some testing and microscopy were completed at the Airbus Industrie's composites technology division in Bremen, Germany. Other aspects of the examination of the subject components, such as the visual examination, nondestructive testing, and fractography, are documented in separate reports including Materials Laboratory Factual Reports 02-077, 02-078, and 02-083.

Parties to the examination were the Federal Aviation Administration (FAA), the Bureau Enquetes – Accidents (BEA), Airbus Industrie, American Airlines (AA), and the Airline Pilots Association (APA). Participants in the composite materials testing and examination included:

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Samples were selected from multiple locations on the vertical stabilizer and rudder for materials testing and microscopic examination to determine chemical composition, extent of cure, glass transition temperature (T_g), fiber and void volume fractions, and ply stacking sequence (layup). For the vertical stabilizer, testing in each area included differential scanning calorimetry (DSC) and infrared spectroscopy (IR). One area was tested using dynamic mechanical analysis (DMA) and modulated differential scanning calorimetry (MDSC). The fiber volume fraction, void volume fraction, and layup in each area were determined using microscopic examination of polished cross-sections. For the rudder, one area was tested using DMA and MDSC. Other samples were selected from the rudder for peel tests and flatwise tensile tests.

Except for the DMA and MDSC testing, NASA Langley completed the materials testing and microscopic examination requested by the Safety Board. Airbus Industrie completed the DMA and MDSC testing of one area each from the vertical stabilizer and rudder as requested by the Safety Board and monitored by the BEA. Airbus Industrie also completed the preparation and analysis of one polished cross-section for microscopic examination.

1. Construction and Materials

1.1. Vertical Stabilizer

The vertical stabilizer design is a stiffened box with removable LE fairings and TE panels. The stiffened box consists of two integrally stiffened skin panels for the left and right sides, spars for the forward and aft sides, and closure ribs at the upper and lower ends. The integral stiffeners in the skin panels consist of 24 "I"-shaped stringers that extend spanwise parallel to the aft spar, numbered from the aft to forward. Internal stiffeners for the box consist of a center spar at the lower end of the span and 16 ribs, not including the two closure ribs. The ribs are numbered from the lower end upward starting with the lower closure rib. The components of the box are riveted together, and the LE fairings and TE panels are attached with threaded fasteners.

Except for the fasteners, lightning protection strips, and TE panel support frames, the vertical stabilizer is made entirely of composite materials. The stiffened box of the vertical stabilizer is a solid carbon-fiber reinforced polymer (CFRP) laminate composed of T300 carbon fibers in a Hexcel 913[1] epoxy matrix. The laminate includes both unidirectional tape and eight-harness satin fabric layers in the construction. The zero-degree fibers of the fabric and tape layers in the composite skin panels are generally oriented parallel to the stringers and aft spar, which are at an angle of 33.3 degrees to the aft of vertical, with the exception that the zero-degree fibers associated with the front spar flange are parallel with this spar, which is at an angle of 41.5 degrees to the aft of vertical. In the spar webs, the zero-degree fiber direction is parallel to the vertical centerline. In the ribs, the zero-degree direction is either parallel to the horizontal centerline for panel ribs, or it is parallel to the longitudinal axis of truss members for truss ribs.

The curing temperature for the CFRP is specified to be 250 degrees Fahrenheit. According to Airbus material qualification data, the onset glass transition temperature ($T_{g\text{-onset}}$) should be 144 degrees Celsius in the dry condition and should be 122 degrees Celsius after exposure to a climate of 50 percent relative humidity (corresponding to a moisture content of 0.7 weight percent).[2] According to the engineering drawings, the fiber volume fraction for the CFRP is 60% ± 4%. The maximum porosity permitted in the cross-section is 2.5 percent. The layup consists of fabric and tape, with the fabric layers oriented at ±45 degrees and at 0/90 degrees relative to the zero-degree fiber direction and the tape layers with fibers all oriented parallel to the zero-degree fiber direction. The layup varies with location and will be shown for each sample location in the results section below.

[1] At the time the accident airplane was manufactured, the epoxy used in the vertical stabilizer was CIBA 913C, made by Ciba-Geigy Ltd., of Switzerland. Ciba-Geigy sold their composites business to Hexcel Corporation in 1996.
[2] For more information regarding calculation of $T_{g\text{-onset}}$ and average $T_{g\text{-onset}}$ values for this material system, see Appendix A.

1.2. Rudder

The rudder is a single-segment wedge-shaped design with removable LE fairings. The wedge consists of left and right skin panels with a single spar at the forward side. The skin panels are fastened together at the trailing edge by rivets with a metallic strip on each side. Threaded through-bolts near the trailing edge also help fasten the two skin panels. The spar is riveted to the skin panels. Pieces of the LE fairings are attached to the skin panels with threaded fasteners and to each other with threaded fasteners through metal support flanges. There are no internal stiffeners in the wedge. Closure ribs cap the upper and lower ends of the rudder.

The rudder skin panels and spar are sandwich composites. Each panel has a nomex honeycomb core and GFRP and CFRP face sheets.

2. Materials Testing and Examination

2.1. Vertical Stabilizer

Samples from the vertical stabilizer were taken from each skin panel, the forward, center, and aft spars, rib 1, and rib 3. These samples included both damaged and undamaged areas from the lower end to the upper end. Locations where samples were cut from the skin panels are indicated in figures 1 and 2, which show ultrasonic test data for the right and left skin panels, respectively. For more details regarding the ultrasonic testing and other nondestructive examination (NDE) data, see Materials Laboratory Factual Report 02-078.

The locations, sample sizes, and tests performed are listed in table 1. On the right side skin panel, four samples were cut from undamaged areas near the aft spar plus two samples were cut from damaged areas in the forward and aft lower attachment lugs. On the left side skin panel, three samples were cut from undamaged areas near the forward spar and one sample was cut from a damaged area near the left forward lug. One sample each from the forward, center, and aft spar, and from ribs 1 and 3 were cut from undamaged areas.

2.2. Rudder

Samples from the rudder were cut from the right skin panel between hinges 5 and 7. One 8-inch by 12-inch sample was cut for DMA and MDSC testing, one 11-inch by 12-inch sample was cut for climbing drum peel tests, and four 2-inch by 2-inch squares were cut for flatwise tensile tests. The locations of the samples are shown in figure 3 superimposed on the tap test data for the right skin panel. For additional information regarding the tap testing and other NDE data, see Materials Laboratory Factual Report 02-078.

Table 1. Vertical Stabilizer Test Samples

Sample Name	Sample Size (inches)	Sample Location	Tests Performed
Right Skin Panel			
RS1	2 by 2	Right skin between the aft spar and stringer 1, below and adjacent to rib 5	IR, DSC, microstructural examination
RS2	2 by 2	Right skin between aft spar and stringer 1, above and adjacent to rib 7	IR, DSC, microstructural examination
RS3	2 by 2	Right skin between aft spar and stringer 1, below and adjacent to rib 12	IR, DSC, microstructural examination
RS4	4 by 9.5	Right skin between aft spar and stringer 1, below and adjacent to rib 16	IR, DSC, microstructural examination, DMA, MDSC
RA3	1 by 1 triangle	Right aft lug aft and adjacent to the attachment hole bore	IR, DSC, microstructural examination
RF4	1 by 1 triangle	Right forward lug forward and above the attachment hole bore	IR, DSC, microstructural examination
Left Skin Panel			
LS1	2 by 2	Left skin between the forward spar and stringer 22, below and adjacent to rib 4	IR, DSC, microstructural examination
LS2	2 by 2	Left skin between the forward spar and stringer 19, below and adjacent to rib 7	IR, DSC, microstructural examination
LS3	2 by 2	Left skin between the forward spar and stringer 15, below and adjacent to rib 12	IR, DSC, microstructural examination
LF3f	1 by 1 triangle	Forward and adjacent to stringer 23, above and adjacent to the rib 1 fasteners	IR, DSC, microstructural examination
Aft Spar Web			
AS1	2 by 2	Above and adjacent to the rib 1 attach flange near the web centerline	IR, DSC, microstructural examination
Center Spar Web			
CS1	2 by 2	One inch below rib 1 to the right of the web centerline	IR, DSC, microstructural examination
Forward Spar Web			
FS1	2 by 2	Below and adjacent to rib 5 at the web centerline	IR, DSC, microstructural examination
Rib 1			
R1-1	2 by 2	Forward of the center spar	IR, DSC, microstructural examination
Rib 3			
R3-1	one-inch length	Forward left truss leg	IR, DSC, microstructural examination

3. Results

3.1. Vertical Stabilizer

3.1.1 Chemical Composition

The chemical composition of each sample shown in table 1 was assessed using IR spectroscopy, measuring total attenuated reflectance through a microscope. The results were typical for this composite material with no significant variances in the spectra for each specimen.

3.1.2. Cure and T_g

The extent of cure and the T_g of sample RS4 (from the upper end of the right skin panel) were analyzed using MDSC, DMA, and DSC. Details of the MDSC and DMA procedures and results are shown in Appendix A. Portions of sample RS4 were tested in the as-received condition and in the dry condition. The moisture content for the as-received condition was approximately 0.58 percent.

The MDSC results showed an average residual heat value of 4.5 joules per gram, which corresponded to an extent of cure greater than 97 percent. The average T_g measured was 154 degrees Celsius.

The DMA results show that in the as-received condition, the $T_{g\text{-onset}}$ measured 134 degrees Celsius, which was between the qualification values of 144 degrees Celsius for the dry condition and 122 degrees Celsius for the 50 percent relative humidity (0.7 percent moisture content) condition. The portion of sample RS4 that was tested in the dry condition had a $T_{g\text{-onset}}$ of 149 degrees Celsius.

The extent of cure and the T_g of each sample shown in table 1, including sample RS4, was assessed using DSC. No significant variance was observed in the results among all the samples. Results indicate that the extent of cure for each sample was sufficient.

3.1.3 Fiber and Void Volume Fractions

Sections of the samples were cut, then mounted and polished for microscopic observation. For specimens analyzed at NASA Langley, volume fractions were determined by computer image analysis of micrographs taken from 5 randomly selected locations on each sample, resulting in analysis of approximately 0.20 square millimeter. Results indicate that the materials were prepared to the desired fiber volume fractions with acceptable void content. No evidence of microcracking was observed. The results are shown in Table 2.

Specimens RS1b and RS1c were cut and mounted at NASA Langley and then sent to Airbus Industrie for final polishing and analysis. The fiber fraction and volume fraction were determined by computer image analysis across 104 square millimeter for sample RS1b and 145 square millimeter for sample RS1c. The results are shown in Table 2.

Table 2. Fiber and Void Volume Fractions

Section Name	Volume Fraction (percent)	
	Fiber	Void
Right Skin Panel		
RS1a	57	0.6
RS1b	47	0.6
RS1c	50	0.5
RS2	62	0.7
RS3	59	0.5
RS4	59	0.8
RA3	65	1.1
RF4	58	2.0
Left Skin Panel		
LS1	52	0.6
LS2	61	0.7
LS3	51	0.5
LF3f	55	1.4
Aft Spar Web		
AS1	52	1.5
Center Spar Web		
CS1	49	2.6
Forward Spar Web		
FS1	53	1.3
Rib 1		
R1-1	57	1.5
Rib R3		
R3-1	55	1.2

3.1.4 Layup

The layup for each sample was determined by optical microscopy of cut and polished specimens from each sample listed in table 1. Micrographs of each specimen were assembled into mosaics to determine the layups. Typical cross-sectional views for samples RS1, LS1, and R3-1 are shown in figures 4 to 6.

The cross-section for sample RS1 shown in figure 4 was cut at approximately a 45-degree angle to the zero-degree fiber. A long relatively white area interwoven with light-gray tows of fibers is a layer of ±45-degree fabric. A continuous layer appearing light gray is a zero-degree tape layer. Light gray layers with individual tows of fibers are 0/90-fabric layers. The matrix appears dark gray. Voids and surface stains appear nearly black.

Cross-sections for samples LS1 and R3-1 shown in figures 5 and 6 were cut nearly parallel to the 90-degree fiber direction. In these cases, the continuous layers of light gray are zero-degree tape layers. Light gray layers with individual tows of fibers are ±45-degree fabric. No 0/90-degree fabric layers were present in sample LS1 or R3-1. The matrix appears dark gray. Voids and surface stains appear nearly black.

Two tape materials were used in the vertical stabilizer. One consisted of a single sheet with a thickness close to that of the fabric layers. The other consisted of two sheets placed together such that the total thickness was nearly equal to that of the fabric layers. A cross-section of a single layer of the latter tape material would appear as two layers, each having a thickness nearly equal to the thickness of a tow of fibers in the fabric. An example of this is shown in the tape layers in sample R3-1 in figure 6. The four tape layers at the center appear to be eight layers, each having a thickness approximately half that of the fabric layers.

The observed layup in each sample was compared to the engineering drawings from the manufacturer. In some cases, a splice layer[3] was observed in the cross-section, such as shown in figure 5. Splice layers are not listed in the layup results unless the splice was continuous across the examined cross-section. Results of the comparisons are shown in detail in Appendix B and are summarized below.

Layup discrepancies between the sample and the drawing were observed only in sample RF4. In the outer precured half layers, one layer of ±45-degree fabric was observed in the place of one layer of 0/90-degree fabric, and one layer of 0/90-degree fabric in the place of one layer of ±45-degree fabric. These layers, drawing reference numbers 5 and 76, were located within 12 layers from each other. According to the engineering drawing, the contours for layers 5 and 76 are identical.

In the inner precured half layers for sample RF4, discrepancies were observed at two locations within a span of less than ten layers. At the first location, one extra layer of

[3] A splice is where the ends of two sheets of fabric overlap, creating a joint in the layer. In the cross-section, the joint may appear as an extra layer that does not continue across the entire cross-section.

±45-degree fabric and one extra layer of 0/90-degree fabric were present. At the second location, one layer of ±45-degree fabric and one layer of 0/90-degree fabric were missing.

In the remaining samples, RS1, RS2, RS3, RS4, RA3, LS1, LS2, LS3, LF3f, AS1, CS1, FS1, R1-1, and R3-1, no layup discrepancies were observed between the samples and the drawings.

3.2. Rudder

3.2.1. Cure and T_g

The extent of cure and the T_g of a sample from the right skin panel were analyzed using MDSC and DMA. Details of the MDSC and DMA results are shown in Appendix A. Portions of the sample were tested in the as-received condition and in the dry condition. The moisture content for the as-received condition was approximately 0.81 percent.

The MDSC results showed no residual heat, which corresponded to an extent of cure of 100 percent.

The DMA results showed that in the as-received condition, the $T_{g\text{-onset}}$ measured 82.9 degrees Celsius, which was between the qualification values of 102 degrees Celsius for the dry condition and 75 degrees Celsius for the 70 percent relative humidity / 70 degrees Celsius (0.75 to 0.90 percent moisture content) condition. The portion of the right skin sample that was tested in the dry condition had a $T_{g\text{-onset}}$ of 102.5 degrees Celsius.

3.2.2. Climbing Drum Peel Tests

The drum peel force of the outboard skin panel facesheet was measured on the right skin panel of the rudder. The peel test sample location where four specimens were cut from the skin panel is shown in figure 3. Three test specimens measured 2.95 inches wide, and one specimen measured 2.02 inches wide. The drum peel force was measured in a direction perpendicular to the spar using a drum having a two-inch inner radius and a 2.5-inch outer radius.

The average measured drum peel force for the 2.95-inch wide specimens was 33.5 pounds (149 newtons), and the drum peel force for the 2.02-inch wide specimen was 21.7 pounds (96.5 newtons).

The peel test specimens fractured both within the facesheet matrix between the innermost layer of fibers and the honeycomb and within the honeycomb core itself (core failure). For the 2.95-inch wide specimens, the average area of core failure was 67 percent. For the 2.02-inch wide specimen, the area of core failure was 22 percent.

In a report dated October 10, 1990, Airbus conducted rolling drum peeling tests on rudder skin panels containing carbon and glass fibers (such as that of the accident rudder skin panel). In these tests, the peel force was measured in a direction perpendicular to the spar for 75-millimeter (2.95-inch) wide samples using a drum having a 50 millimeter inner radius and a 62.5 millimeter outer radius. According to this report, for these skin panels, the average drum peeling force for the outboard facesheet was 222 newtons (49.9 pounds), and the average area of core failure was 38 percent.

3.2.3. Flatwise Tensile Tests

The transverse (through-thickness) tensile strength of the right skin panel was measured for four specimens from four locations on the right skin panel (see figure 3). Load was applied perpendicular to each facesheet of the sandwich composite.

All four specimens fractured and separated within the honeycomb core (core failure), although one specimen also was cracked at the facesheet-to-core interface. The average transverse tensile strength was 0.97 megapascals (140 pounds per square inch). Airbus Industrie does not have flatwise tensile test data on rudder panels. However, according to Hexcel Corporation, the transverse tensile strength for a HRH 10-1/4-1.5 nomex honeycomb is approximately 1.14 megapascals (165 pounds per square inch).

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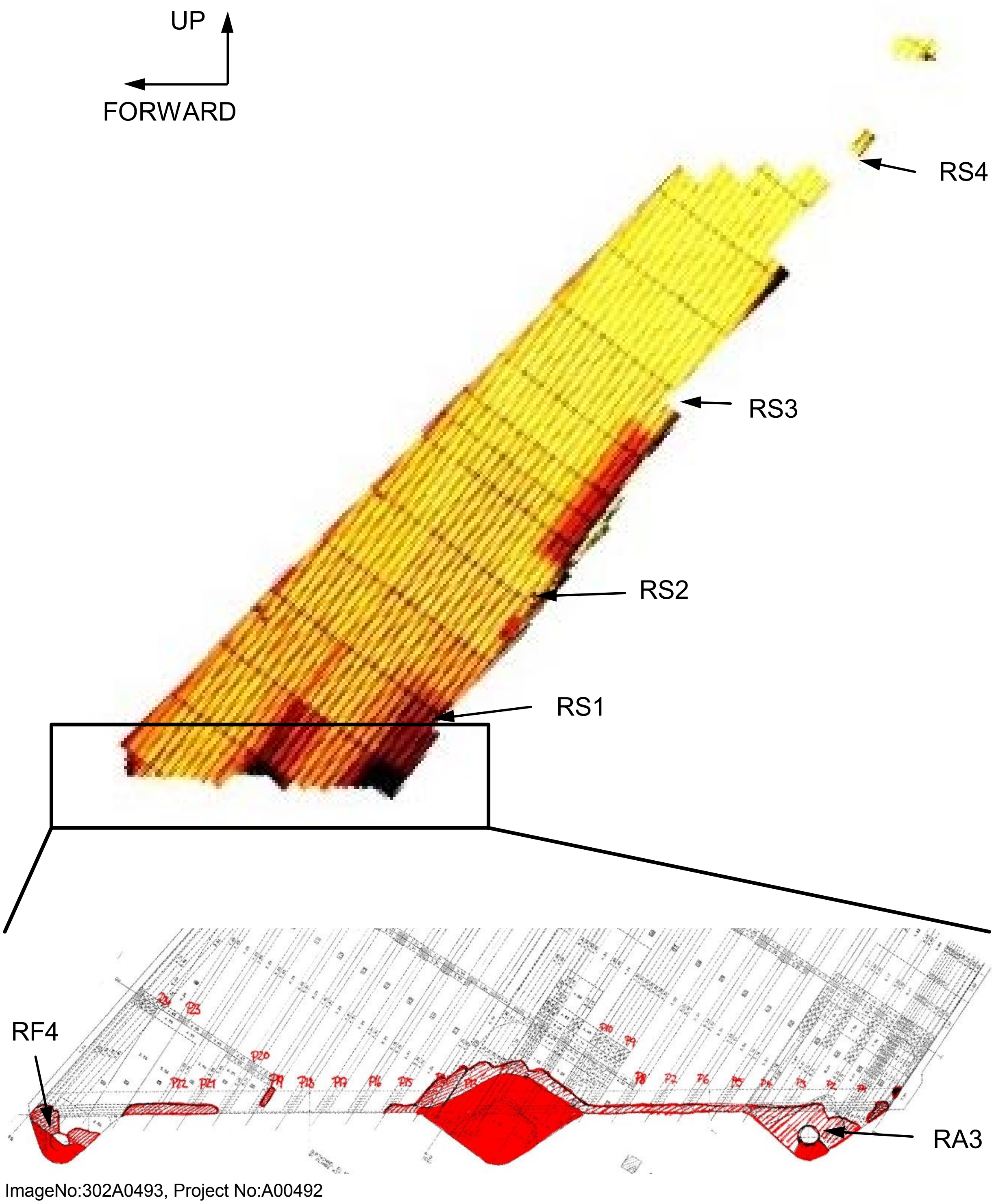

Figure 1. View of the vertical stabilizer right skin panel showing ultrasound testing results (see Materials Laboratory Factual Report 02-078). Arrows indicate locations where materials samples were cut from the skin panels.

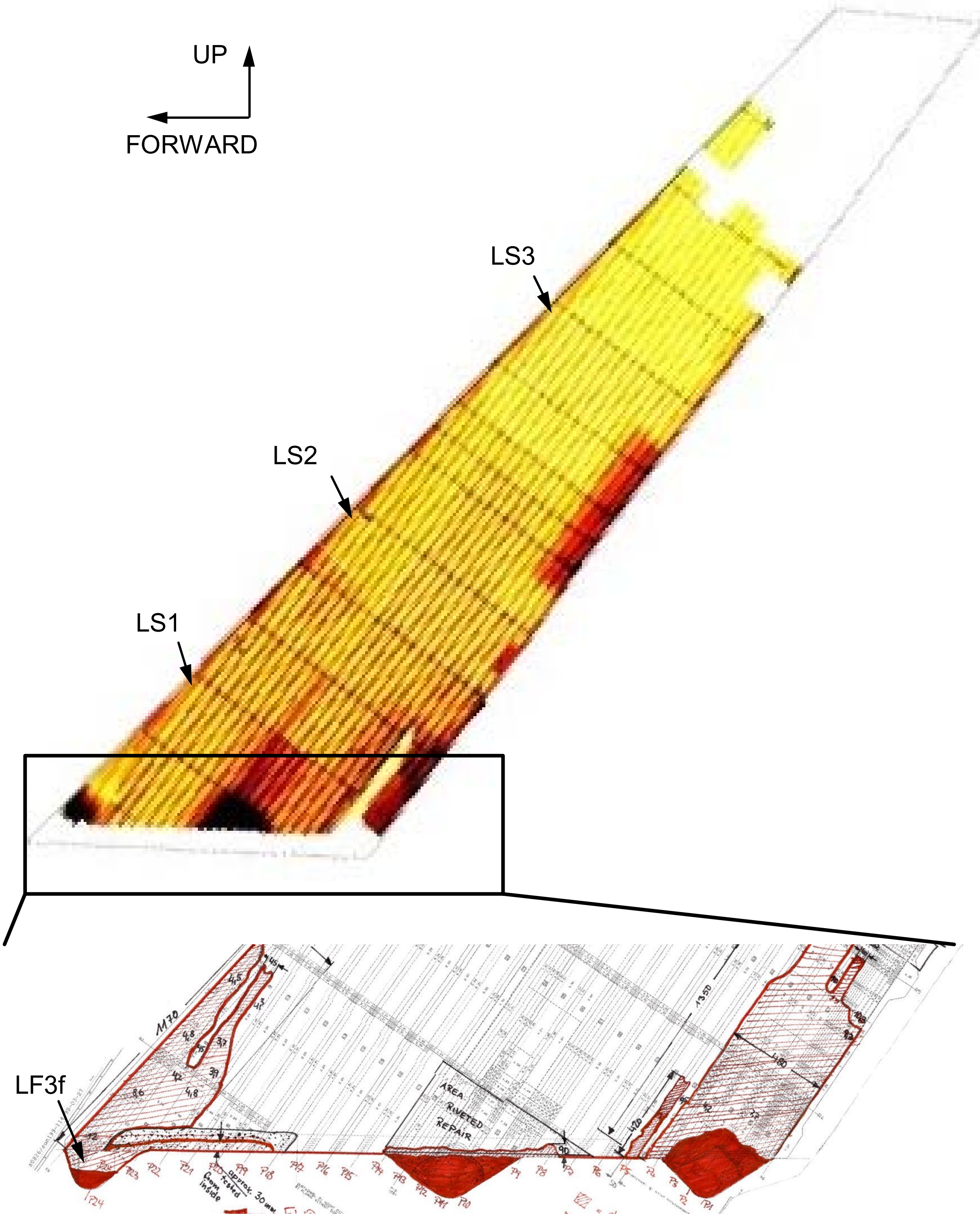

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Figure 2. View of the vertical stabilizer left skin panel showing ultrasound testing results (see Materials Laboratory Factual Report 02-078). Arrows indicate locations where materials samples were cut from the skin panels.

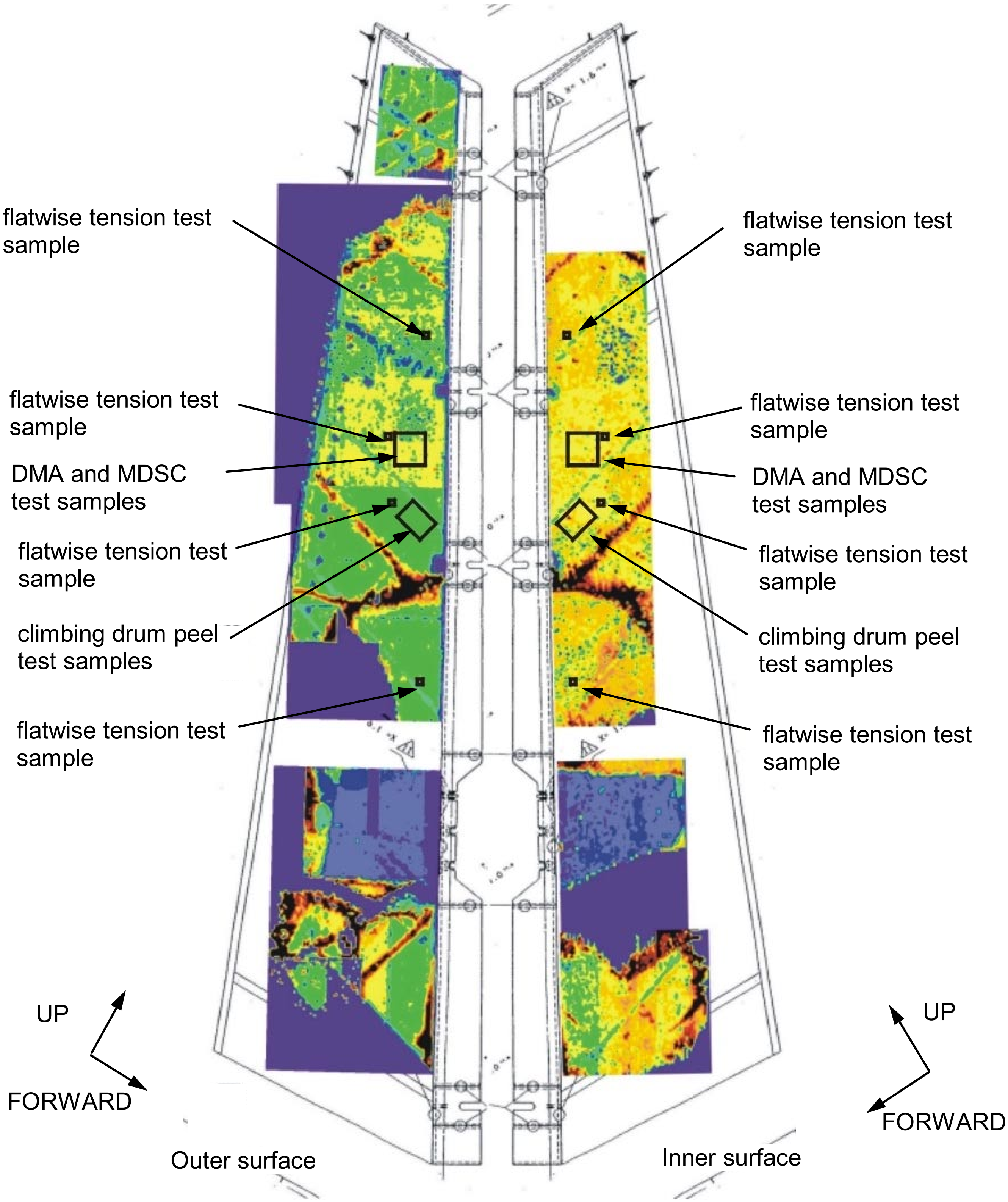

Figure 3. Overall view of the right rudder skin panel showing tap test results for the inner and outer surfaces (see Materials Laboratory Factual Report 02-078). Locations where test samples were cut from the skin are indicated on both surfaces.

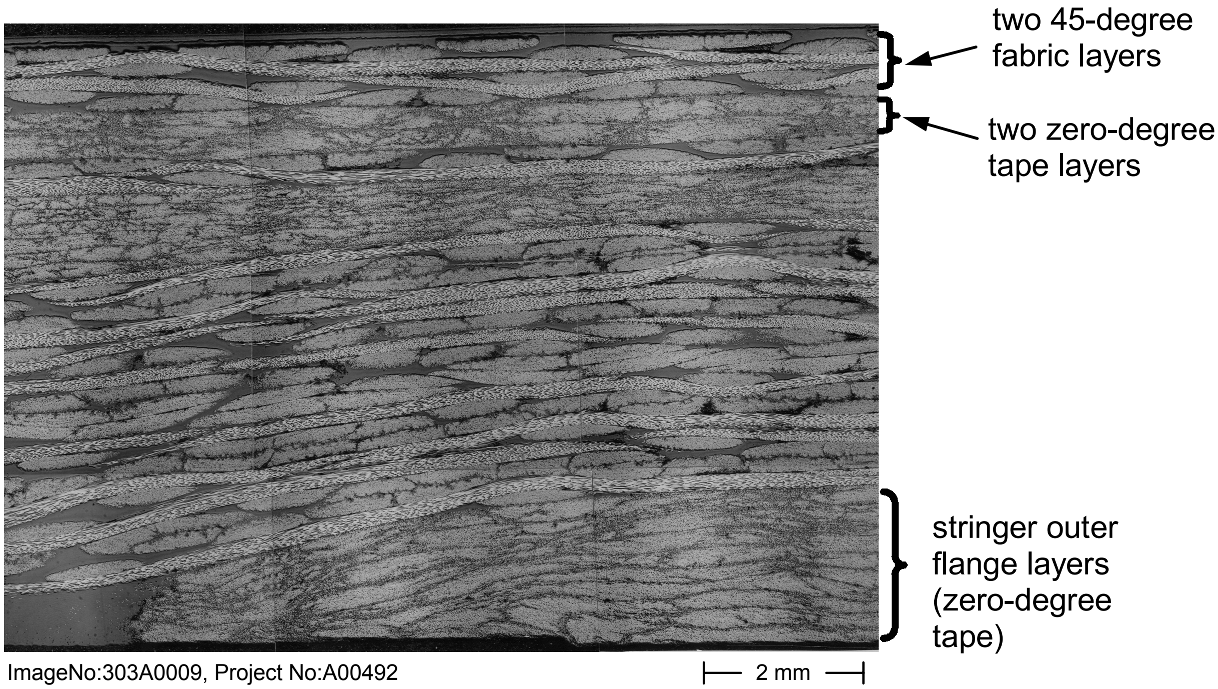

Figure 4. Cross-sectional view of sample RS1 in a plane parallel to the plus or minus 45-degree fiber direction. The outer surface is at the top of the micrograph.

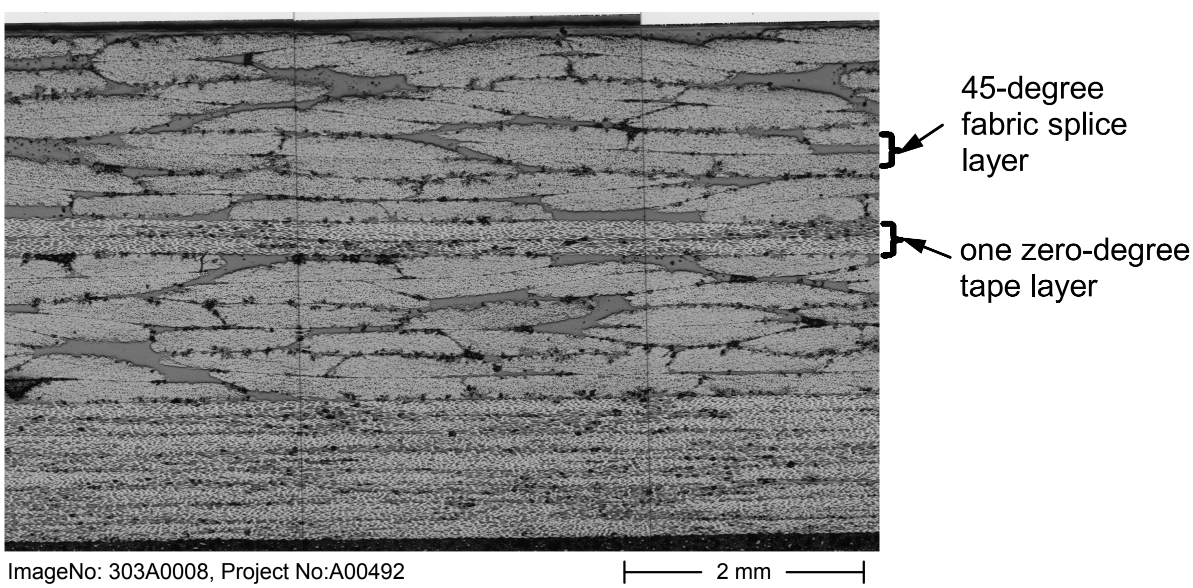

Figure 5. Cross-sectional view of sample LS1 in a plane parallel to the 90-degree fiber direction.

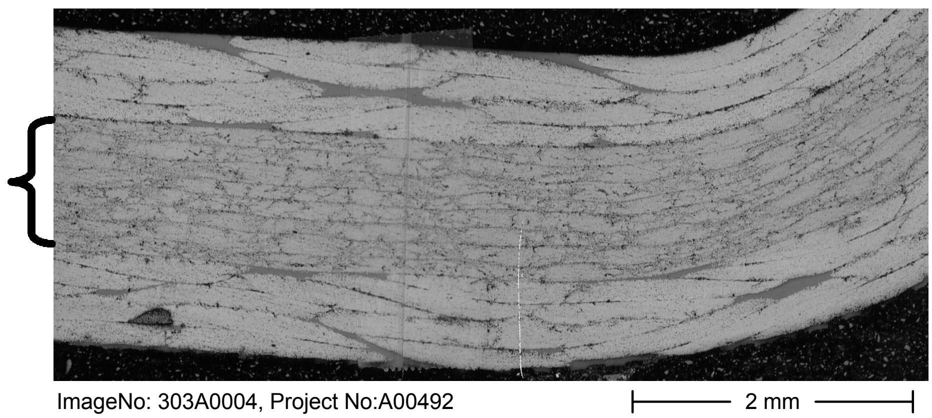

Figure 6. Cross-sectional view of the flange area on sample R3-1 in a plane parallel to the 90-degree fiber direction.

Appendix A
Airbus Technical Note TN-ESWCG-1181/02

AIRBUS	TECHNICAL NOTE	Page 1

Order - No.: Preparation: Duration:	Copy to (* = only this coversheet) See page 2

Report No.: TN – ESWCG-1181/02

Author: -------------------

Department: ESWCG

Title: Thermal Analysis by DMA and MDSC of CFRP materials Hexcel F913, F550 and AIK EHG 250 after 14 years of service

Date: 24.09.2002

Summary: T_g by DMA Analysis and DSC-Analysis by MDSC has been performed on specimens that were cut out of the composite vertical tail plane (VTP) of an Airbus A300-600. Referenced by testing during qualification (testing period of time 1984-1988) the DMA- and MDSC- tested specimen showed the same level for both wet and dry test specimens. No significant difference could be noticed. As well, the degree of cure was sufficient.

Key Words (Retrieval Terms): CFRP, DMA, MDSC, tg-onset, degree of cure

	Issue	Date	No. of page	Revised pages	Valid from/for
	1	24.09.2002	16		

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Date	24.09.2002		25.09.2002		
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	Issue 1					
	Date Prepared Checked					

1 INTRODUCTION

The investigations of this TN were made on specimens which were cut from the fin-box and rudder skins of the vertical tail plane (VTP) of MSN 420 (flight AAL587). Different resin systems were used in these parts. The fin-box is made of the resin system Hexcel F913 carbon tape and fabric and the rudder skins are made from F550 carbon fabric in combination with EHG250 fiberglass fabric. The EHG250 fiberglass fabric was used to adhere the Nomex ® honeycomb core to the CFRP skin and was co-cured with the skin.

All tested specimens were original parts of MSN 420, delivered by the BEA (Bureau d´enquetes et d´analyses, french accident investigation office). Testing was done on behalf of NTSB (National Transportation Safety Board) and witnessed by BEA.

2 EXPERIMENTAL

2.1 Dynamic Mechanical Analysis (DMA)

DMA Analysis has been performed according to AITM 1-0003, issue 2 [1].

AITM 1-0003 is a method for determination of the glass transition temperature by DMA analysis. Tests have been performed for specimens in original “as received” condition and after drying.

In the DMA Analysis the glass transition temperature T_g is defined as the temperature where the sample exhibits a dramatic change in mechanical and damping behavior with increased temperature when connected to an oscillation displacement. Three different T_g values can be determined by the measurement according to AITM 1-0003. The three different T_g values are interpreted in *Fig 1* and described below.

- $T_{g\text{-onset}}$ is defined as the temperature of extrapolated tangents drawn from points on the storage modulus curve before and after the start of the glass transition event.
- $T_{g\text{-loss}}$ is defined as the temperature where the diagram loss modulus versus temperature has its maximum.
- $T_{g\text{-peak}}$ is defined as the temperature where the diagram tan σ (damping) versus temperature has its maximum.

The $T_{g\text{-peak}}$ value is usually several degrees higher than $T_{g\text{-loss}}$ value and corresponds more closely to the transition midpoint while the $T_{g\text{-onset}}$ value more closely denotes the initial drop from the glassy state into the transition. The T_g value that is of interest differs and should be stated in the report.

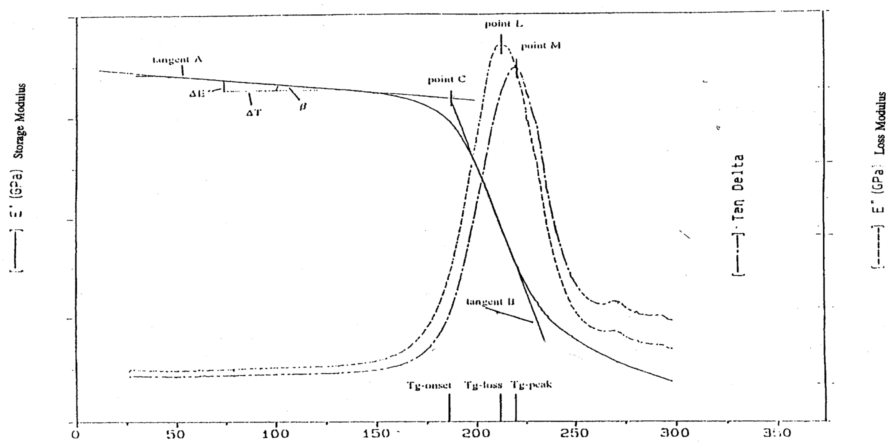

Figure 1

Fig. 1 Three different Tg values $T_{g\text{-}onset}$, $T_{g\text{-}loss}$, and $T_{g\text{-}peak}$ are interpreted in the diagram [*1*]. *The unit on the x-axis is °C.*

2.1.1 Sample preparation for DMA and Moisture Content

The DMA specimens were cut in order to have the faces parallel to the fiber direction. After machining, the test specimens were conditioned before testing according to *tables 1 and 2*. In minimum one specimen was kept in an oven chamber at a temperature of 90 °C. This so called Moisture Content Specimen indicates the moisture absorption of the component during lifetime.

2.1.2 Testing parameters

The relevant test parameters according AITM 1-0003 were used. Prior to the thickness of the DMA specimen the heating rate was sometimes reduced to 2 °C/min.

The DMA analysis were performed according to:

Instrument:	DMA 983 (Dynamic Mechanical Analyses)
	Thermal Analyst 2100, Du Pont
Method:	AITM 1-0003 A, issue 2
Frequency:	Resonant frequency
Amplitude:	0.2 mm
Rate:	normally 3 °C/min; thick specimen: 2 °C/min
Moment:	7 lbs
Nitrogen flow:	None

Note: Integrity of the test results was insured by calibration measurements and data correlation to previously performed DMA tests using the same DMA 983 equipment.

Table 1 DMA test specimens for 913

Specimen	**1** (VTP)	**2** (VTP)	**3** (VTP)	**4** (VTP)
Condition	as received	as received	as received	1) drying at 90 °C

1) After machining the test specimen was dried to a constant level prior to DMA analysis

Table 2 DMA test specimen for F550/EHG250

Specimen	**5** rudder skin	**6** rudder skin	**7** rudder skin	**8** rudder skin	**9** rudder skin	**10** rudder skin
Condition	as received	as received	as received	1) Drying at 90 °C	1) Drying at 90 °C	1) Drying at 90 °C

1) After machining the test specimen was dried to a constant level prior to DMA analysis

2.1.3 Moisture Content Specimen

Comparing figures of moisture pick-up, it is important to know to what base they are related (dry weight), so as a consequence the so-called “Moisture Content Specimens” were prepared taken account for:

- Calculation of the actual moisture content of the “as received” sample, prior to testing
- Testing of DMA-specimen in real dry condition

The moisture content specimens were dried in a 90 °C-oven. Related to the initial moisture content, the loss of weight was recorded to have the asymptote of the curve.

2.2 MDSC Analysis

Note: Calorimetric measurements were made for comparative reasons only.

The MDSC technique measures the amount of energy (or heat) absorbed or released by a material as it is either heated, cooled or maintained at a constant (isothermal) temperature. This heat flow/temperature data provides valuable information of such physical/chemical properties as:

- glass transition event
- determine the level of curing

MDSC analysis has been performed according AITM 3-0008, issue 1 [2]. The used MDSC equipment and testing parameters are the following:

Instrument:	DSC Q 100 (Modulated Differential Scanning Calorimetric), Du Pont
Sample weight	5 – 10 mg punctured out the laminate cross section
Method:	AITM 3-0008, issue 1
Rate:	10 °C/min
Nitrogen flow:	7 ml/min

Note: Integrity of the test results was insured by calibration measurements

The investigated specimens are shown in *tables 3 and 4*.

Table 3 MDSC test specimens for 913

Specimen	**11** (VTP)		**12** (VTP)		**13** (VTP)		**14** (VTP)	
Condition	as received		As received		as received		as received	
Tested for	residual heat (J/g)	Tg (°C)	residual heat (J/g)	Tg (°C)	residual heat (J/g)	Tg (°C)	residual heat (J/g)	Tg (°C)

Table 4 MDSC test specimen for F550/EHG250

Specimen	**15** rudder skin	**16** rudder skin	**17** rudder skin	**18** rudder skin	**19** rudder skin	**20** rudder skin
Condition	as received	as received	as received	1) drying at 90 °C	1) drying at 90 °C	1) drying at 90 °C

1) After machining the test specimen was dried to a constant level prior to DMA analysis

3 RESULTS

3.1 Moisture Content Specimen

3.1.1 Moisture content for 913

The “Zero Weight Specimen” were dried in an oven at 90 °C and by gravimetric measurements the loss of weight is calculated which will correspond to the loss of moisture. In t*able 5* the weight loss of 913 is shown for different times. After this model is established it will be possible to introduce a correction in the “wet” (as received) DMA values if necessary.

The moisture content in the dried specimens is viewed by ca. 0.55-0.60 % by weight*, see figure 2.*

Table 5 Moisture content (weight %) in the test specimen after re-drying for 913

Exposure date	12. Apr	Specimen Nr.4 m_0 (g)=	14.0142
Exposure time: 10.15			Weight %
Date	Time (h)	m1 (g)	M [%]
12. Apr	0	14.0142	0
12. Apr	3	13.9924	-0.16
12. Apr	5	13.9893	-0.18
15. Apr	77	13.9489	-0.47
16. Apr	101	13.9376	-0.55
18. Apr	149	13.9336	-0.58
19. Apr	173	13.933	-0.58

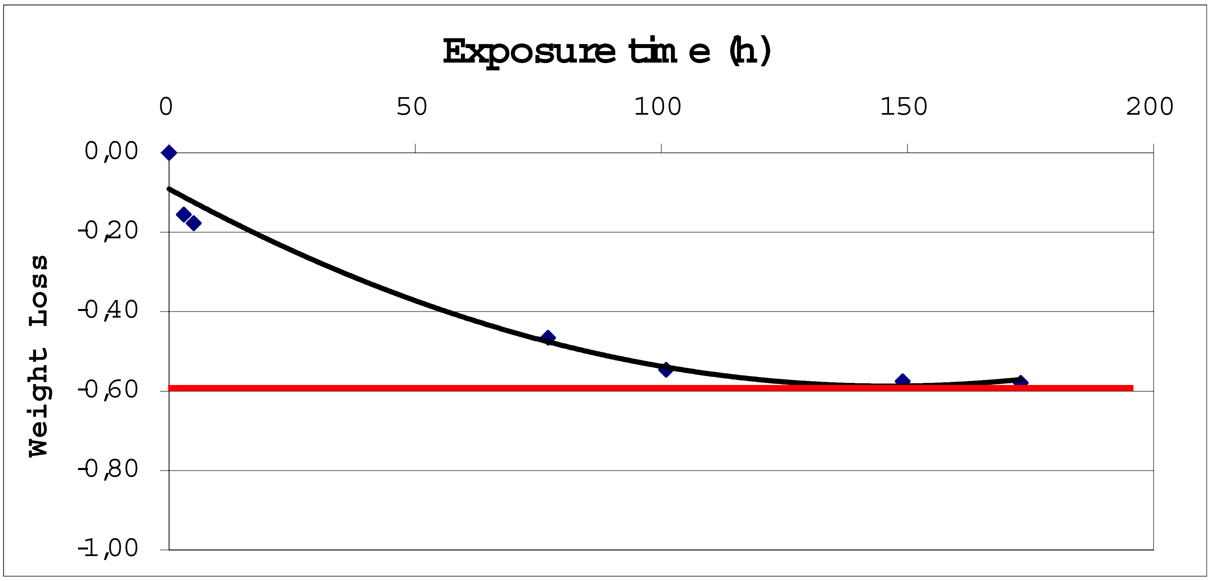

Fig. 2 Moisture content (weight %) in the test specimen after drying

3.1.2 Moisture content for F550/EHG250

The moisture content of F550/EHG250 is shown in *table 6*. After drying the weight loss is about 0.80 % by weight. The experience has shown that the moisture absorption until saturation for a climate of 70 °C/70 % RH for F550 and EHG 250 is between 0.75 and 0.9 %. Therefore the drying of the F550/EHG250 sample has reached the dried state.

Table 6 Weight loss of F550/EHG250 (rudder skin)

Specimen		Date	Xav. 1)	**18** rudder skin	**19** rudder skin	**20** rudder skin
m_0	[g]	20.08.02		0.5314	0.5441	0.5365
m_1	[g]	10.09.02		0.5270	0.5397	0.5322
weight loss	[%]		**0.81**	0.83	0.81	0.80

1) Xav.: average

3.2 DMA

3.2.1 Results for 913

The test results from the T_g analysis for the tested specimens are shown in *table 7.*

Typical DMA curves from the T_g measurement are shown in the appendix (1) as examples.

Table 7 The T_g test results for the “as-received”specimens (resin: 913)

Specimen	Xav. 1)	**1** (VTP)	**2** (VTP)	**3** (VTP)
Tg-onset [°C]	***134***	*137*	*132*	*134*
Tg-loss [°C]	***156***	*158*	*152*	*159*

Note 1) Xav.: mean value

For comparison reasons relevant values of the system 913C Fabric (see Qualification report W6/87, authorized in 1988 [3]) are summarized in *table 8* and *figure 3*.

Table 8 Moisture content (weight %) of traveler specimen exposed till saturation

Tg onset of DMA specimen exposed till saturation

Exposure in Climate % rel. Humidity	Moisture content by weight %	Tg onset (°C)		
		X average	s	n
-	0	**144**	5	33
50	0.7	**122**		4
70	1.3	111	5	11
85	1.6	102	3	11
95	2,1	96	2	11

X: mean value
s: standard deviation
n: number of tested specimen

When comparing the observed $T_{g\text{-onset}}$ value, see *table 7*, it can be noticed that the mean average value of 134 °C for the specimens in “as received condition” do not show a large difference for both the dry specimens ($T_{g\text{-onset}}$ = 144 °C) and the wet specimens after saturating at 50% relative humidity ($T_{g\text{-onset}}$ = 122 °C), see *Table 8,* which were referenced in the cited Qualification report.

For a better understanding of the observed level of 134 °C, the relationship between moisture content and $T_{g\text{-}onset}$ was introduced in *figure 3,* which shows a linear correlation between moisture content and $T_{g\text{-}onset}$.

By the indicated moisture content of about 0.55-0.60 % by weight, and after extrapolating that value in *figure 3* it is obvious that at this moisture level a theoretical $T_{g\text{-}onset}$ of about 129 °C should be reached. This extrapolated value is well comparable to the experimentally measured $T_{g\text{-}onset}$ value of 134 °C.

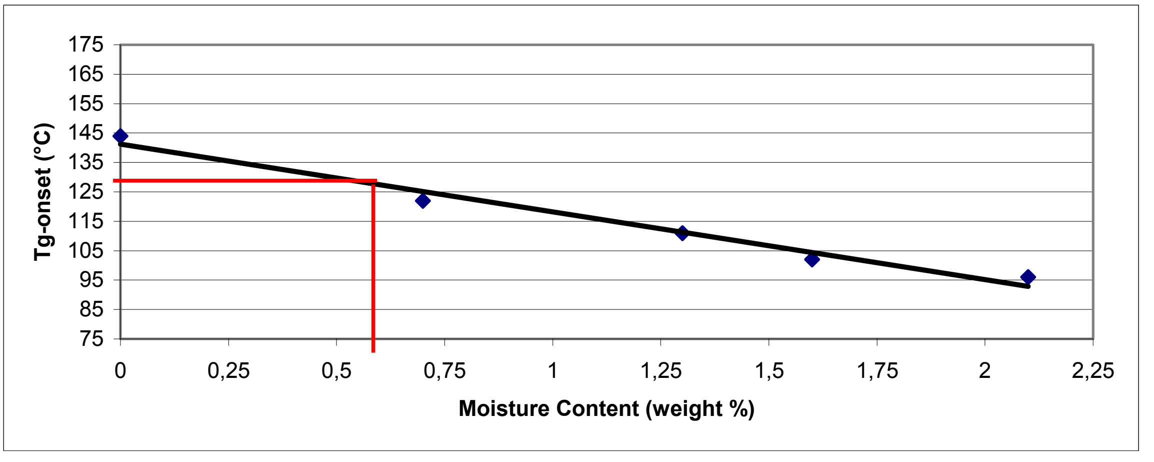

Fig 3. Correlation between moisture pick-up and Tg-onset

The dried test specimen have shown an increase in the $T_{g\text{-}onset}$ value, see *table 9.*

By comparing this MSN 420 $T_{g\text{-}onset}$ result of **149 °C**, it is noticeable that this figure is comparable to the $T_{g\text{-}onset}$ value of **144°C** of the previously performed DMA tests obtained at qualification testing, see *table 9*.

Table 9 The T_g test results after drying for 913

Specimen	**4**
Tg-onset [°C]	***149***
Tg-loss [°C]	*181*

It has been verified that the observed values didn't show any offset.

No significant difference of Tg-performance between MSN 420 - and previously DMA-tested specimens was observed

Remark

The first two DMA specimen showed a secondary relaxation peak in the temperature region between 90 and 110 °C. After slightly removing the gray paint this secondary relaxation peak has not been observed anymore.

3.2.2 Results for F550/EHG250

The DMA results for F550/EHG250 of “as received” and “dried” specimen are shown in *table 10 and 11*.

Table 10 DMA results for “as received” specimen for F550/EHG250

Specimen		**5** rudder skin	**6** rudder skin	**7** rudder skin	**average**
Condition		as received	as received	as received	
Tg-onset	[°C]	81.6	83.1	84.0	**82.9**
Tg-loss	[°C]	132.4	137.2	136.8	135.5

Table 11 DMA results after drying for F550/EHG250

Specimen		**8** rudder skin	**9** rudder skin	**10** rudder skin	**average**
Condition		dry 1)	dry 1)	dry 1)	
Tg-onset	[°C]	97.9	105.0	104.5	**102.5**
Tg-loss	[°C]	140.1	143.7	137.7	140.5

1) Dried at 90°C

Previous investigations showed a similar Tg-onset for F550/EHG250 [5]. After analyzing the diagrams of [5] according to AITM 1-0003 [1] there were the following values for dry and wet (70 °C/70 % RH) specimens:

- dry ca. 102 °C
- wet ca. 75 °C

Compared to the values shown in *table 10 and 11* there is no significant difference observed.

3.3 MDSC

3.3.1 Results or 913

The test results from the residual heat analysis and the Tg measurements for the tested specimens are shown in *table 7.*

Typical *MDSC* curves are shown in the appendix (2 and 2a) as examples.

For kinetic calculations relevant values, which were reported in test report K219/94 [4], were used for the matrix system 913C.

When comparing the measured residual heat release values it is verified that this low value of 4.5 J/g indicates that this minimal excess of energy is correlative with a **sufficient degree of cure of > 97%.**

The observed Tg value (154 °C) has the same level as the DMA-$T_{g\text{-loss}}$ (156 °C)

Table 7 The residual heat values and T_g test results

Specimen Condition	Xav. 1)	**5** as received		**6** as received		**7** as received		**8** as received	
		res. Heat (J/g)	Tg (°C)	res. Heat (J/g)	Tg (°C)	res. Heat (J/g)	Tg (°C)	res. Heat (J/g)	Tg (°C)
	4.5 J/g 154 °C	2.8	155	7.0		4.3	155	3.8	152

Note 1) Xav.: mean value

The observed physical/chemical properties show no difference prior to qualification performance.

3.3.2 MDSC results for F550/EHG250

None of the investigated specimens of F550/EHG250 have shown a residual heat for "as received" or dried specimens. **The degree of cure is 100%**. The diagrams are shown in the appendix (3).

4 DISCUSSION / COMMENT

$T_{g\text{-}onset}$ measurements by DMA and DSC-Analysis by MDSC have been performed. The tested specimens were cut out of the composite VTP (vertical tail plane) of the Airbus A300-600, MSN 420 (flight AAL587). Referenced by testing during qualification (testing period of time 1982-1988) the DMA - and MDSC - tested specimens showed the same level for both wet and dry test specimens.

- **No significant difference in material performance could be noticed.**
- **The curing of the matrix was sufficient.**

5 REFERENCES

[1] AITM 1-0003 (issue 2), Determination of the glass transition temperatures (1995)

[2] AITM 3-0008 (issue 1), Determination of the extent of cure by Differential Scanning Calorimetric (1995)

[3] Qualification report W6/87, authorized in 1988

[4] Test report K219/94

[5] TN-BT25-21/82, „*Mechanische Untersuchungen zur Harz-Kompatibilität für FVW-Hybride im Airbus-Seitenleitwerk (Phase 2), Teil 1: Torsionsschwingungsmessungen*“ (1982)

Record of Revisions

Issue	Date	Page	§	Reason of Revision
1	24.09.2002	-all-	-all-	

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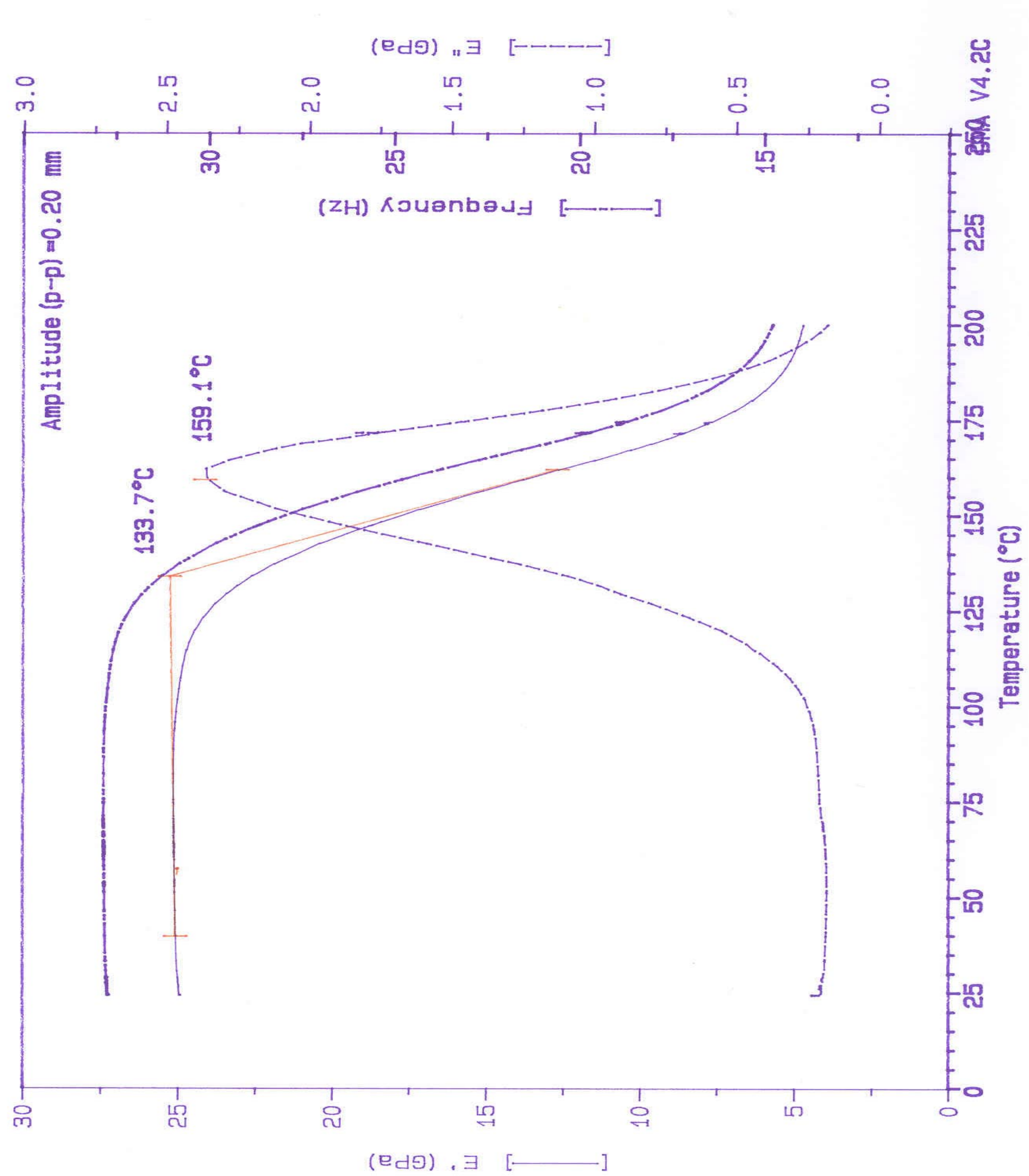
	Date Prepared Checked					

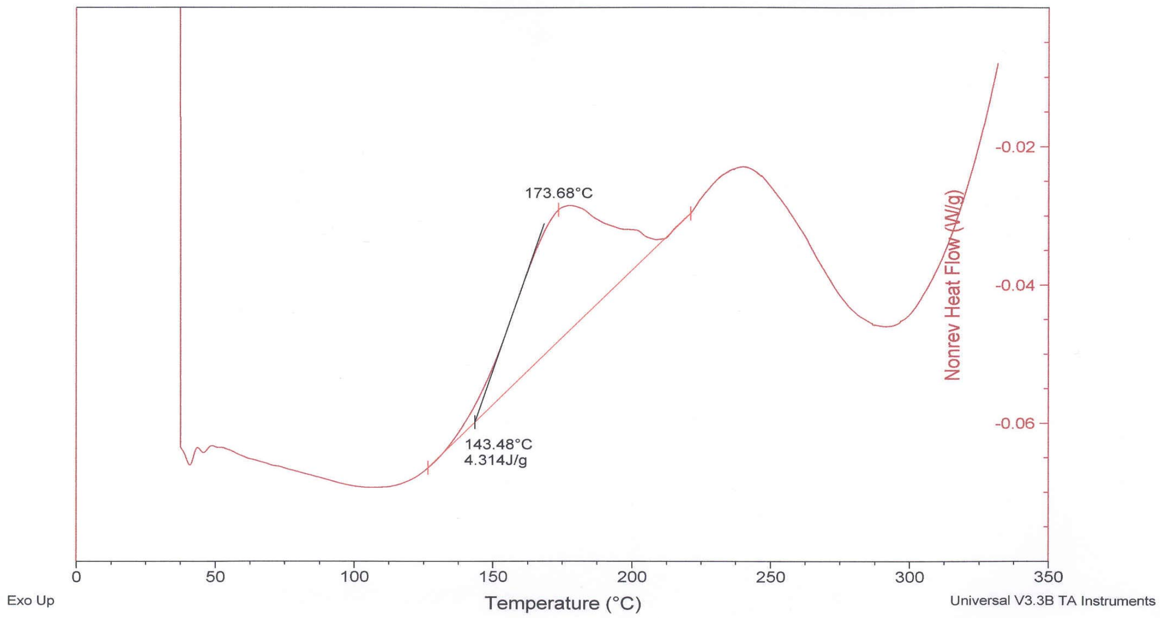
Appendix 1



DMA-testing of C-913

Tg-performance (sample 3) in "as received " condition

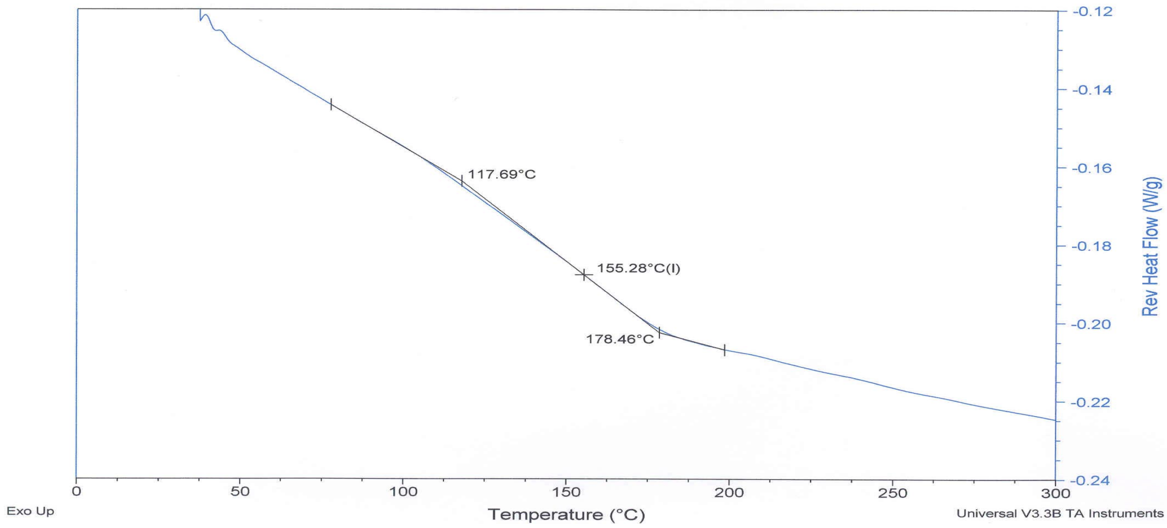
Appendix 2



<u>**MDSC-testing of C-913**</u>

determination the residual level of curing

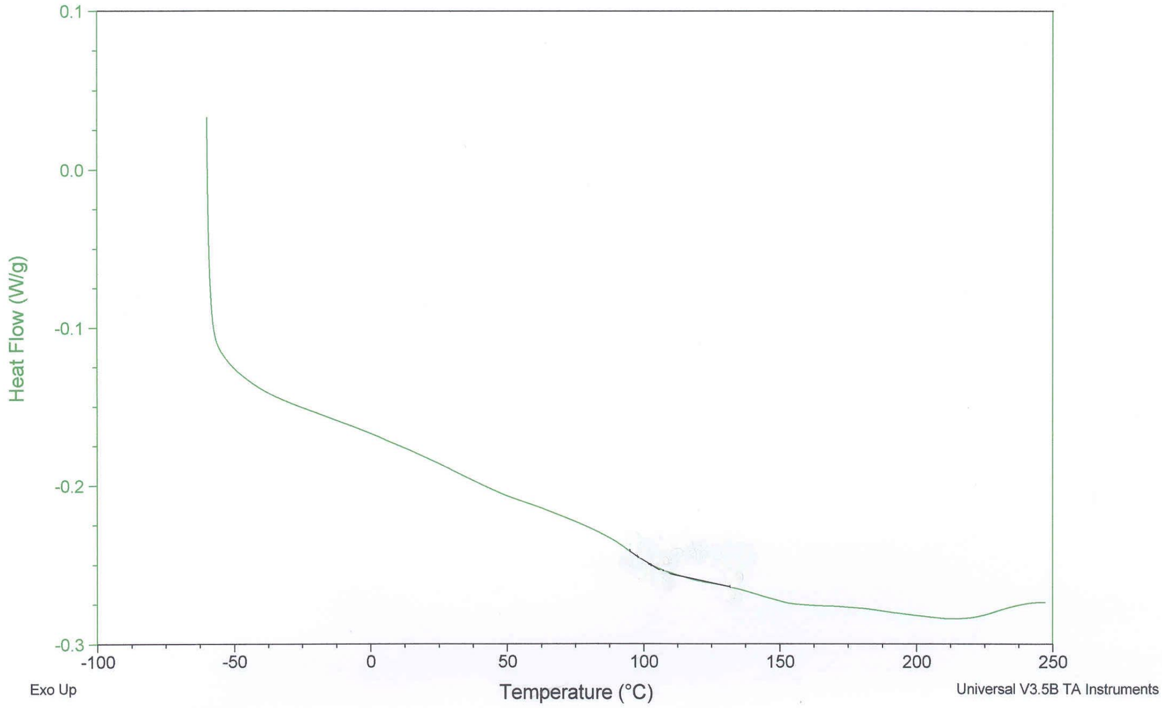
Appendix 2a



<u>**MDSC-testing of C-913**</u>

determination of the glass transition event

Appendix 3



MDSC-testing of F550/EHG250

no significant level of residual heat release could be analysed

	Issue 1					
	Date Prepared Checked					

Appendix B
Layup Comparison Tables

The following tables document the layup observed on the polished cross-sections of specimens cut from the vertical stabilizer. Sample locations are as listed in table 1 of the main body of text. The tables also show the expected layup based on the engineering drawings.

In each table, the sequence number is the layer in the sample numbered from the outside surface for the skin panel specimens, from the forward surface for the spar web specimens, and the upper surface for the rib specimens. The drawing reference number is the layer number in the applicable engineering drawing. Layer orientations are labeled 45 for ±45-degree fabric, 90 for 0/90-degree fabric, and 0 for zero-degree tape. The text in the table is in bold where discrepancies were observed between the sample and the drawings.

Sample RS1

Sequence Number	Drawing Reference Number	Layer Orientation	
		Sample	Drawing
Skin Layers			
1	5	45	45
2	6	45	45
Rear Spar Flange			
3	39	0	0
4	40	0	0
Skin Layers			
5	8	45	45
6	9	0	0
7	10	0	0
8	11	0	0
9	12	45	45
10	13	45	45
11	14	45	45
12	14 (splice)	45	45
Reinforcing Fitting Layers			
13	5	90	90
14	8	45	45
15	9	45	45
16	10	45	45
17	11	45	45

Sample RS2

Sequence Number	Drawing Reference Number	Layer Orientation	
		Sample	Drawing
Skin Layers			
1	5	45	45
2	6	45	45
3	8	45	45
4	12	45	45
5	14	45	45

Sample RS3

Sequence	Drawing Reference	Layer Orientation	
		Sample	Drawing
Skin Layers			
1	5	45	45
2	6	45	45
3	8	45	45
4	14	45	45
Reinforcing Fitting Layers			
5	35	90	90
6	36	45	45

Sample RS4

Sequence Number	Drawing Reference Number	Layer Orientation	
		Sample	Drawing
Skin Layers			
1	5	45	45
2	6	45	45
3	8	45	45
4	14	45	45
Reinforcing Fitting Layers			
5	35	90	90
6	36	45	45

Sample RA3

Sequence Number	Drawing Reference Number	Layer Orientation	
		Sample	Drawing
Outer Precured Half Layers			
1	9	45	45
2	42	45	45
3	57	90	90
4	7	45	45
5	38	0	0
6	68	90	90
7	56	45	45
8	45	90	90
9	58	45	45
10	52	45	45
11	63	0	0
12	53	90	90
13	67	45	45
14	43	0	0
15	66	90	90
16	1	45	45
17	64	0	0
18	54	45	45
19	32	0	0
20	4	90	90
21	5	45	45
22	47	0	0
23	50	45	45
24	48	45	45
25	8	0	0
26	51	90	90
27	24	45	45
28	6	0	0
29	34	90	90
30	37	45	45
31	55	0	0
32	41	90	90
33	33	45	45
34	69	45	45
35	2	0	0
36	22	45	45
37	31	45	45
38	39	0	0
39	49	90	90
40	46	45	45
41	35	45	45
42	23	0	0
43	30	90	90
44	3	45	45
45	44	45	45
46	55a	0	0
47	40	45	45
48	36	90	90
49	65	45	45
Skin Layers			
50	5	45	45
51	6	45	45
		delamination*	
Rear Spar Flange Layers			
52	52	0	0
53	53	0	0
54	54	0	0
55	55	0	0
Skin Layers			
56	8	45	45
57	9	0	0
58	10	0	0
59	11	0	0
60	12	45	45
61	13	45	45
62	14	45	45
Reinforcing Fitting Layers			
63	5	90	90
64	6	90	90
65	7	90	90
66	8	45	45
67	9	45	45
68	10	45	45
69	11	45	45
Inner Precured Half Layers			
70	122	45	45
71	53	90	90
72	22	45	45
73	16	45	45
74	92	45	45
75	79	0	0
76	67	90	90
77	4	45	45
78	13	0	0
79	20	45	45
80	125	0	0
81	80	45	45
82	107	0	0
83	95	90	90
84	52	45	45
		delamination†	
85	17	0	0
86	119	90	90
87	94	45	45
88	65	0	0
89	15	90	90
90	104	45	45
91	23	45	45
92	85	0	0

93	120	45	45
94	126	45	45
95	93	0	0
96	3	90	90
97	18	45	45
98	110	45	45
99	77	0	0
100	100	45	45
101	12	45	45
102	24	0	0
103	66	45	45
104	14	45	45
105	21	0	0
106	91	90	90
107	114	45	45
108	124	45	45
109	51	0	0
110	26	90	90
111	88	45	45
112	106	45	45
113	135	0	0
114	81	90	90
115	1	45	45
116	118	45	45
117	37	0	0
118	82	45	45
119	116	45	45
120	73	0	0
121	8	45	45
122	10	45	45
123	115	90	90
124	84	45	45
125	75	90	90
126	86	45	45
127	96	45	45
128	89	0	0
129	109	90	90
130	90	45	45
131	6	45	45
132	129	0	0
133	83	90	90
134	50	45	45
135	5	0	0
136	11	90	90
137	25	45	45
138	38	45	45
139	2	0	0
140	7	90	90
141	98	45	45
142	64	45	45
143	19	90	90
144	70	45	45
145	9	0	0
146	39	90	90
147	108	45	45
148	121	0	0
149	103	90	90
150	36	45	45
151	101	0	0
152	123	90	90
153	76	45	45
154	99	90	90
155	102	45	45
156	136	45	45
157	97	0	0
158	87	90	90
159	72	45	45
160	78	45	45
161	111	90	90
162	117	0	0
163	74	45	45
164	105	0	0
165	71	90	90
166	137	45	45
Compensation Layers			
167	1	0	0
168	2	45	45
169	3	90	90
170	4	45	45
171	5	90	90
172	6	45	45

*Delamination opening was approximately equal to a full layer thickness.

†Delamination opening was approximately equal to half a layer thickness.

Sample RF4

Sequence Number	Drawing Reference Number	Layer Orientation	
		Sample	Drawing
Outer Precured Half Layers			
1	6	45	45
2	**5**	**45**	**90**
3	14	45	45
4	41	90	90
5	24	45	45
6	79	0	0
7	44	45	45
8	30	45	45
9	15	0	0
10	4	45	45
11	38	45	45
12	11	0	0
13	29	90	90
14	**76**	**90**	**45**
15	12	45	45
16	27	0	0
17	73	90	90
18	16	45	45
19	47	0	0
20	21	90	90
21	28	45	45
22	46	45	45
23	25	90	90
24	45	90	90
25	74	45	45
26	18	45	45
27	75	0	0
28	77	90	90
29	8	45	45
30	23	0	0
31	40	45	45
32	26	45	45
33	37	90	90
34	48	45	45
35	78	45	45
36	39	0	0
37	13	90	90
38	22	45	45
39	43	0	0
40	20	45	45
41	50	45	45
42	7	0	0
43	9	90	90
44	10	45	45
45	31	0	0
46	49	90	90
47	80	45	45
48	42	45	45
49	3	0	0
50	17	90	90
51	32	45	45
52	2	45	45
53	19	0	0
54	1	90	90
Skin Layers			
55	5	45	45
56	6	45	45
Front Spar Flange Layer			
57	26	0	0
Skin Layers			
58	8	45	45
59	9	0	0
60	12	45	45
61	13	45	45
62	14	45	45
Inner Precured Half Layers			
63	82	90	90
64	35	45	45
65	56	0	0
66	46	90	90
67	9	45	45
68	42	90	90
69	27	45	45
70	52	0	0
71	33	45	45
72	16	0	0
73	22	90	90
74	45	45	45
75	3	45	45
76	41	45	45
77	28	0	0
78	51	45	45
79	37	45	45
80	6	90	90
81	15	45	45
82	17	45	45
83	20	0	0
84	30	90	90
85	55	45	45
86	21	45	45
87	14	90	90
88	7	45	45
89	4	0	0
90	38	90	90
91	19	45	45
92	24	0	0
93	5	45	45
94	12	0	0
95	58	90	90
96	13	45	45
97	40	0	0

98	18	90	90
99 100 101 102 103	**11 25 34**	**45 45 45 90 90**	**45 45 90**
104	31	45	45
105	53	45	45
106	8	0	0
107 108	**2 1 10 43**	**90 45**	**90 45 90 45**
109	29	45	45
110	44	0	0
111	39	45	45
112	32	0	0
113	54	90	90
114	23	45	45
115	57	45	45
116	36	0	0
117	26	90	90
118	83	45	45
Compensation Layers			
119	1	0	0
120	2	45	45
121	3	90	90
122	4	45	45
123	5	0	0
124	6	45	45

Sample LS1

Sequence Number	Drawing Reference Number	Layer Orientation	
		Sample	Drawing
Skin Layers			
1	5	45	45
2	6	45	45
3	8	45	45
4	9	0	0
5	12	45	45
6	13	45	45
7	14	45	45
Stringer Outer Flange Layers			
8	1	0	0
9	2	0	0
10	3	0	0
11	4	0	0

Sample LS2

Sequence	Drawing Reference	Layer Orientation	
		Sample	Drawing
Skin Layers			
1	5	45	45
2	6	45	45
Forward Spar Flange Layers			
3	13	0	0
4	14	0	0
5	15	0	0
6	16	0	0
7	17	0	0
8	18	0	0
9	19	0	0
Skin Layers			
10	7	45	45
11	8	45	45
12	12	45	45
13	13	45	45
14	14	45	45
Stringer Outer Flange Layer			
15		0	0
Module Flange Layers			
16		45	45
17		45	45
18		45	45

Sample LS3

Sequence Number	Drawing Reference Number	Layer Orientation	
		Sample	Drawing
Skin Layers			
1	5	45	45
2	6	45	45
Forward Spar Flange Layers			
3	13	0	0
4	14	0	0
5	15	0	0
6	16	0	0
Skin Layers			
7	7	45	45
8	8	45	45
9	14	45	45
Stringer Outer Flange Layers			
10		0	0
11		0	0
Module Flange Layers			
12		45	45
13		45	45
14		45	45

Sample LF3f

Sequence Number	Drawing Reference Number	Layer Orientation	
		Sample	Drawing
Outer Precured Half Layers			
1	2	45	45
2	1	0	90
Additional Layer (Concession)			
3		45	45
Skin Layers			
4	5	45	45
5	6	45	45
6	8	45	45
7	9	0	0
8	12	45	45
9	13	45	45
10	14	45	45
Inner Precured Half Layers			
11	82	90	90
12	9	45	45
13	16	0	0
14	22	0	90
15	3	45	45
16	6	90	90
17	15	45	45
18	17	45	45
19	20	0	0
20	21	45	45
21	14	90	90
22	7	45	45
23	4	0	0
24	19	45	45
25	5	45	45
26	12	0	0
27	13	45	45
28	18	90	90
29	11	45	45
30	8	90	0
31	2	90	90
32	1	45	45
33	10	90	90
34	83	45	45
Compensation Layers			
35	1	0	0
36	2	45	45
Rib 1 Flange Layers			
37		45	45
38		45	45
39		90	0
40		90	90
41		90	0
42		45	45
43		45	45
Stringer Outer Flange Layers			
44		0	0
45		0	0
46		0	0
47		0	0

Sample AS1

Sequence Number	Drawing Reference Number	Layer Orientation	
		Sample	Drawing
1		45	45
2		0	0
3		45	45
4		45	45
5		0	0
6		45	45
7		0	0
8		45	45
9		0	0
10		45	45
11		0	0
12		45	45
13		45	45
14		0	0
15		45	45
16		0	0
17		45	45
18		45	45
19		45	45
20		0	0
21		45	45
22		45	45
23		0	0
24		45	45
25		45	45
26		45	45

Sample CS1

Sequence Number	Drawing Reference Number	Layer Orientation	
		Sample	Drawing
1		45	45
2		0	0
3		45	45
4		0	0
5		45	45
6		0	0
7		45	45
8		45	45
9		0	0
10		45	45
11		45	45
12		0	0
13		45	45
14		45	45
15		45	45
16		45	45
17		0	0
18		45	45
19		45	45
20		45	45
21		45	45
22		0	0
23		45	45

Sample FS1

Sequence Number	Drawing Reference Number	Layer Orientation	
		Sample	Drawing
1		45	45
2		45	45
3		45	45
4		45	45
5		45	45
6		45	45
7		45	45
8		45	45

Sample R1-1

Sequence Number	Drawing Reference Number	Layer Orientation	
		Sample	Drawing
1		45	45
2		45	45
3		45	45
4		45	45
5		45	45
6		45	45

Sample R3-1

Sequence	Drawing Reference	Layer Orientation	
		Sample	Drawing
Cap Section			
1		45	45
2		45	45
3		0	0
4		0	0
5		0	0
6		45	45
7		45	45
Flange Section			
1		45	45
2		45	45
3		0	0
4		0	0
5		0	0
6		0	0
7		45	45
8		45	45