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TESTING PROCEDURES FOR CARBON FIBER REINFORCED PLASTIC COMPONENTS

H.J. Gosse, M. Kaitatzidi and S. Roth

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16. Abstract	·					
Tests for studying the basic material are considered and quality control investigations involving preimpr gnated materials (prepreg) are discussed. Attention is given to the prepreg area weight, the fiber area weight of prepregs, the resin content, volatile compontets, the effective thickness, resin flow, the resistance to bending strain, tensile strength, and shear strength. A description of tests conducted during the manufacturing process is also presented, taking into account X-ray methods, approaches of neurton-radiography, ultra-sonic procedures, resonance methods and impedance studies.						
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Abstract (Scientific technical unbiased review)

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In the context of developing carbon fiber reinforced plastic construction techniques for mass production destructive and non-destructive testing procedures are tested or investigated for their possible use. Destructive tests are discussed which are performed upon the receipt-of-goods and during and after processing. In addition, findings are discussed which were obtained by applying the non-destructive testing procedures established. It turned out that the most important defect in carbon fiber reinforced plastic structures can be studied by means of x-ray and ultrasonic tests. By using special test instrument settings it was possible to detect hollow spaces, porosities, delaminations, foriegn body inclusions, broken fibers and density differences in carbon fiber reinforced plastic laminates. For glued combinations of laminates and honeycombs, between two different laminates and between laminates and aluminum covers the following defects were detected in the gluing: porosity, poor cohesive binding, defective adhesive binding and foreign body inclusions. In addition, the resulsts of mechanical impedance tests for the non-destructive testing of materials are also discussed. Prospects for further development of the testing procedures mentioned are commented on.

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TESTING PROCEDURES FOR CARBON FIBER REINFORCED PLASTIC COMPONENTS

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1. Introduction

For a long time fiber technology has been making inroads in the aircraft industry and already a large number of structural components are being made using fiber construction techniques. Additional components are still being built or are in the development stage. The advantages offered by fiber construction techniques point to the fact that all future development in this direction will be increased.

The first component which was mass produced in Europe, after successful testing, using carbon fiber reinforced plastic construction techniques was the airbrake of the Alpha-Jet. The most important individual parts of this airbrake -- the airbrake shell and spars -- are made from individual carbon fiber reinforced laminates which are cemented together (Fig. 1-1).

Other structrual components of the Alpha-Jet made of carbon fiber reinforced plastic, such as the rudder and elevator, are still in the development stages (Fig. 1-2). Plans are also being made for later mass production of these components if they pass their tests.

It is important that reliable quality control for this still relatively new material keeps pace with the practical development of fiber construction techniques.

In this paper we will discuss the quality control tests performed by Dornier for the mass produced airbrake.

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The destructive tests performed on the materials prior to $\frac{8}{8}$ and during fabrication are discussed. In addition, we also give a general overview of the defects which can occur in carbon fiber reinforced plastic laminates of preimpreganted materials (prepreg) and in laminates which have been cemented together.

Next the non-destructive test procedures are briefly outlined and the results obtained from these procedures are reported. Finally, a few characteristic examples are presented to give a better understanding.

This paper does not make any claims of being complete. To be sure better results can be achieved here and there with one or another testing procedure. A certain defect might also be found with a different procedure which was not listed here. This paper is meant to imporve our knowledge of the problems involved and provide stimulus for further progress in this field.

2. Purpose

The purpose of quality control is to set up an effective and <u>/9</u> economical control system for the entire development and manufacture phase which guarantees only flawless products satisfying all tolerable demands are delivered. This includes the suppliers and subcontractors, who in accordance with the contract are fully responsible for meeting the quality requirements within the framework of their supply schedule.

Quality control therefore begins with the selection of material and includes the successive phases of development, construction, production and qualification (testing) up to the successful delivery of the entire performance package to the customer. The problem now consists of proving the usability of a component by means of appropriate destructive and non-destructive tests.

Along these lines within the scope of this lecture we will present all of the test steps which a carbon fiber reinforced plastic component must pass up to its delivery or release for installation.

All of the following statements refer to carbon fiber reinforced plastic laminates which have been produced using the prepreg construction method.

3. Testing of the Basic Material

With fiber reinforced components the testing begins right with the fiber and resin. As for the resin, for carbon fiber reinforced plastics we can essentially refer to the resin norms which were set up in conjunction with fiber glass reinforced plastics. With regard to the carbon fibers and the carbon fiber prepregs, a number of tentative standards and tentative standard specifications have been set up with the cooperation of all aircraft manufacturers and aeronautics institutes.

The tentative standards listed below for carbon fibers are already official and the tentative standard specifications for carbon fiber prepregs will be offical in the near future.

- -- <u>Tentative Standard LN 29 964</u> Carbon fibers; carbon yarn
- -- <u>Tentative Standard LN 29 965</u> Carbon fibers, carbon yarn -- technical terms of delivery

- -- <u>Tentative Standard Specification LN 029 656, as of Aug., 1976</u> Preimpregnated unidirectional carbon fiber laminates (CFK-Pregreg); dimensions
- -- <u>Tentative Standard Specification LN 029 654, as of Aug., 1976</u> Carbon fiber reinforced structural materials; production of test laminates
- -- <u>Tentative Standard Specifica ion LN 029 971, as of Aug., 1976</u> /12 Preimpregnated unidirectional carbon fiber laminates (CFK-Prepreg); technical terms of delivery
- -- <u>Draft Material Performance Bulletin 5.3230, as of Aug., 1976</u> Preimpregnated unidirectional carbon fiber laminates with epoxy resin (prepreg) for use between -55° C and +80° C
- -- Draft Material Performance Bulletin 5.3231, as of Aug., 1976 Preimpregnated unidirectional carbon fiber laminates with epoxy resin (Prepreg) for use between -55° C to +150° C.

Since we are dealing here with fiber composites produced from prepregs, the testing procedures described below begin with tests to determine the characteristics of such prepregs. The performance requirements of these prepregs are naturally such that those of the fiber components are fulfilled.

A prepreg is a flat, resin impregnated fiber laminate. The fibers can be inbedded unidirectionally, woven or distributed at random. The resin is in the so-called B state, which means that at room temperature the resin is in such a viscosity state that the necessary adhesiveness still permits simple working. By the addition of heat the resins once again become highly fluid before they cross-link so that a resin flow develops during hardening. The hardening of prepregs or prepreg laminates is

normally accomplished by means of pressure and temperature.

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Fig. 3-1 shows the destructive testing procedure. These tests begin with a receipt-of goods check of the prepreg material in the state in which it is delivered. The char-/13 acteristics determined here must meet the specifications established in the Material Performance bulletin. The test methods to be used here are described in the technical terms of delivery LN 029 971. Whether the material will be used or rejected is decided by comparing the actual and desired values. Since the quality of laminates is not determined solely by the prepreg characteristics, a receipt-of-goods check must also be made on laminates over and above the receipt-of-goods check of the prepreg material in the delivery state. The requirements in regard to this and/or the associated testing methods to be used are likewise stated in the corresponding Material Performance Bulletin and/or the technical terms of delivery. If the material also meets these requirements then it can be released for the production of component parts.

So-called control laminates are also manufactured along with carbon fiber reinforced plastic components. These control laminates are identical to the laminates checked in the receiptof-goods test.

These control laminates are used to detect changes in prepreg properties as a result of aging and also to check the effect of different autoclave cycles on the laminate characteristics. In addition to these laminate samples, samples are also tested which are taken from the material left over from the component parts. The results of the tests on these samples are compared with the required minimum values for the laminate.

If the established requirements are met both by the laminate

samples and by the samples of material left over from the component parts then the component is released for non-destructive testing.

The testing range and testing methods described below show /14 that the expenditure for quality control is considerable. The reason for this is that there are still large variations in the characteristics of the materials and no mass production experience is available. There is good reason to think, however, that this expense can be reduced. As for the current variations in quality, it is thought that by increased standardization and also in consequence of larger delivery quantities from the producer of such materials stricter demands can be accepted.

3.1. Receipt-of-Goods Check of Prepregs in the Delivery /15 State

In the receipt-of-goods check of prepregs in the delivery state the following characteristics are determined and compared with the desired values:

- Prepreg area weight (fiber + resin)
- Fiber area weight
- Resin content
- Effective thickness
- Volatile components
- Resin flow.

An extremely important quantity is the effecitve prepreg thickness. By this we mean the thickness which is established for a certain fiber volume portion. It is obvious that the tolerance of this thickness determines the thickness tolerances of the laminates and component parts. For this reason as little variation as possible is desired by the user, for with most components thickness tolerances can be compensated for only with great difficulty. Since the effective thickness of prepregs cannot be measured directly, the values for prepreg area weight, fiber area weight and resin content are coordinated in such a way that in meeting the required values neither is the permissable deviation in effective thickness exceeded.

Therefore if one wants to make a statement about the effective thickness on the basis of prepreg characteristics, then it is essential to determine the first thre. characteristics listed above. The volatile components, resin flow and adhesiveness are quantities which give indications of the aging state of the $\frac{16}{16}$ resin. Over and above this these quantities are specified because they essentially determine the ability to convert prepregs into laminates.

Since for example volatile components are relatively difficult to remove, there is a risk of pore formation with a high proportion of these components.

That the resin flow must remain within certain limits is due to the fact that the hardening systems, i.e. hot-press and vacumn bag process in the autoclave, are not completely sealed. If the resin is too fluid there is a risk of its being washed out. If the resins are very viscous then this impairs the binding of the individual layers. As for the adnesiveness, this should be such that at room temperature two adjacent layers stick right together when lightly pressed together. In addition, one must make sure that the protective sheeting of the prepreg can be easily removed without destroying the prepreg.

In order to determine the prepreg characteristics in the delivery state the following testing methods are used.

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Prepreg Area Weight

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The prepreg area weight is to be determined as per DIN 53 /17854. The surface area of the sample should normally be 100 x 100 rm ± 0.5 mm. For tapes whose width is less than 100 mm but greater than or equal to 75 mm the samples are to be taken over the entire width. If the width is less than 75 mm then the length of the sample should be increased so that the above surface area is obtained.

Fiber Area Weight of the Prepreg

The fiber area weight is determined by dividing the previously determined fiber weight $G_F = G_3 - G_2$ by the area of the prepreg sample used.

Resin Content

The resin content of the prepreg is to be determined by washing the resin out of samples in methylethylketone (MEK):

Measurements:	G2	=	wieght of the test vessel
	Gl	=	G ₂ + prepreg sample (approx. 0.5-0.8g)
	G ₃	=	weight of the test vessel and fiber residue
	F	=	surface area of the prepreg sample

Procedure: a) weight determinations (G₂, G₁)

- b) treat sample with MEK
- c) shake vessel, let stand about 3 minutes repeat 3 times. Pour off MEK, being careful not to pour off any fibers.
- d) Repeat Step c) at least 3 times until the fibers no longer stick together.
- e) Drying: 120° C, two hours.

f) Standard atmosphere conditioning. $\frac{18}{2}$ g) weighing (G₃).

The resin content is calculated using the following formula:

$$\left(\frac{G_1 - G_3}{G_1 - G_2}\right) \cdot 100$$

<u>Weight (Prepreg sample-fiber residue)</u> Prepreg Sample Weight

In order to determine the fiber area weight the surface areas of the samples for determining the resin content must also be determined, and to be sure with an accuracy of $\pm 1\%$. The areas of the samples should not be smaller than 10^4 mm².

Volatile Components

The volatile components are to be tested using the following method 100 x 100 mm samples:

Weight determinations: G_1 = Weight of the prepreg sample G_2 = Weight of the hardened sample

Procedure:

- a) Weight determination (G_1)
- b) Hardening of the sample in a preheated oven: temperature: hardening temperature according to manufacturer's instructions. Time: 1 hour. The sample is then hung from a clip in the open air.

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c) Cooling in a desiccator.

d) Weighing (G₂)

The proportion of volatile components [%] is determined <u>/19</u> using the following formula:

$$\left(\frac{G_1 - G_2}{G_1}\right) \cdot 100$$

(Prepreg sample - hardened smaple) weight Prepreg sample weight

Effective Thickness

For a given fiber volume content the effective thickness is calculated using the prepreg area weight, the resin content in percentage weight and the density of the fibers. Since the usual proportion of fiber volume desired in laminates is 60%, the effective thickness is calculated for this percentage.

Notation: M = Prepreg area weight $[g/m^2]$ ψ_H = resin content [% weight] $\hat{\mathbf{g}}_F$ = fiber density [g/cm³] a = laminate length [cm] b = laminate width [cm] t = effective thickness [cm] of a layer.

The effective thickness is computed as follows:

Fiber area weight
$$= \frac{M \cdot (100 - \Psi_{H})}{100} [g/m^{2}] \qquad (1)$$

Fiber volume
$$= \frac{M \cdot (100 - \Psi_{H})}{100} \cdot \frac{1}{9_{F}} [cm^{3}/cm^{2}] \qquad (2)$$

Volume of a hardened layer of a laminate

Fiber volume of a hardened layer of a laminate

Fiber volume per unit area of a hardened layer of a laminate

$$= \frac{a \cdot b \cdot t \cdot 0, 6}{a \cdot b} \left[cm^{3} / cm^{2} \right]$$
(5)

= a · b · t · 0,6 [cm³]

[cm³]

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(3)

(4)

It is:
$$(2) = (5) \cdot 10^4$$

From this it follows that the effective thickness is: $t = \frac{M}{0,6} \cdot \frac{(100 - \Psi_H)}{100} \cdot \frac{1}{g_F} \cdot \frac{1}{10^4}$ [cm]

= a · b · t

$$t = 1,66 \frac{\text{Fiber area weight}}{\text{Fiber density}} \cdot 10^{-4}$$

Resin Flow_

The resin flow is tested on samples using the following method:

Sample description: Dimensions: 100 x 100 mm 3 layers laminated 0° - 90° - 0°.

Procedure: a) Determination of sample weight G_1 and sample surface area S_1 .

- b) Hardening of the sample in a plane parallel press. Hardening cycle as per manufacturer's instructions.
- c) Allow sample to cool and remove absorbent tissue.
- d) Cut a square 70 x 70 mm out of the middle of the sample.

e) Weigh and measure surface area of the square piece which has been cut out (G₂ and S₂).

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The resin flow [7] is computed using the following formula:

 $\left(\frac{\frac{G_1 - \frac{S_1}{S_2} \cdot G_2}{G_1}}{\frac{G_1}{G_1}}\right) \cdot 100$

3.2. Receipt-of-Goods Check of Prepregs in the Hardened State

Since, as already mentioned, the quality of laminates is not determined solely by the characteristics of the prepreg, it is necessary to perform a receipt-of-goods check on laminates. The purpose of this receipt-of-goods check is to provide indications on the state of the fibers, the resin, the bond between fibers and resin and also the degree of pretreatment of the fiber surfaces. Over and above this the behavior of the laminates at the maximum use temperature should also be checked.

In particular, the following mechanical characteristics are determined and compared with desired values:

- Bending strength and bending modulus of unidirectional laminates at room temperature and at maximum use temperature.
- Tensile strength and tensile modulus of unidirectional laminates at room temperature.
- Interlaminar shear strength of multidirectional laminates at room temperature.

The unidirectional bending samples are studied primarily in order to check the behavior at maximum use temperature. Over and above this, of course, the strength and moduli give indications of the corresponding fiber characteristics. The bending sample is an inexpensive sample in terms of its manufacture and testing and therefore is especially suited for receipt-of-goods checks.

In order to get a good bond between the fibers and the resin $\frac{24}{24}$ it is necessary to pretreat the surface of the fibers. The degree of surface pretreatment influences not only the bond between the fibers and the resin but also the tensile strength and the tensile modulus.

Since a direct method for determining the degree of fiber surface pretreatment is not known, one tries to get an indication of this in terms of characteristic mechanical values. These characteristic values are obtained from the tensile test because the amount at which the break is to be expected, and thus the absolute defect number, is considerably greater with tensile samples than with bending samples.

The interlaminar shear strength of multidirectional laminates is determined in the receipt-of-goods check for the following reasons:

- to determine the resin rupture strain. For crossed composites this is more pronounced than in unidirectional composites.
- To determine the shear strength of crossed composites (crossed composites have the smallest shear strength).
- To get an indication of the degree of fiber surface pretreatment.

In order to determine the mechanical characteristics of laminates in the final state the following testing methods are used. The samples used for determining mechanical characteristics are shown in Fig. 3-2.

Bending Strength and Modulus of Elasticity from a Bending /25 Test of Samples with Fibers Oriented Parallel to the Bending Strain

Bending samples were taken from the hardened unidirectional laminate. The samples are to be tested in a 3-point bending device as shown Fig. 3-2.

The ratio of the distance between supports to the thickness of the laminate L/d must be 40:1. However, the distance between the supports for <u>1-laminate</u> samples can be constant. The distance between the supports is to be determined from the average thickness of the samples to be tested. The rate of load application must be 4.0 mm/min.

The bending strength $\sigma_{bBO^{\circ}}$ and the bending modulus $E_{b\alpha O^{\circ}}$ are to be computed using the following formulas:

$$\mathbf{\mathfrak{S}}_{bBO}^{\circ} = \frac{3 \cdot F \cdot L}{2 \cdot b \cdot d^2}$$

$$E_{b \circ c0}^{\circ} = \frac{F \cdot L}{48 \cdot J \cdot f'}$$

wherein:	F	=	Rupture strain	[N]
	ſ١	=	Deflection at F	[mm]
	J	=	Moment of plane area	[mm ⁴]
	b	=	Sample width	[mm]
	d	=	Sample thickness	[mm]
	\mathbf{L}	=	Distance between supports	[mm]

The term f' is the deflection which is given by a linear force/displacement curve. If the curve is not linear then a tangent must be drawn to the curve which passes through the origin. The deflection f' is to be taken from this straight line at the corresponding load F.

Tensile Strength and Modulus of Elasticity Determined by /26 the Tensile Test

Flat tensile samples are taken from the hardened laminate. The samples are to be stretched parallel to the direction of tension. During the testing a force/dispalcement diagram is recorded. The rate of load application must be 1.0 mm/min.

The tensile strength and the tensile modulus are to be determined using the following formulas:

$$\mathbf{E}_{BO}^{o} = \frac{F}{b \cdot d}$$

$$E_{\infty 0} = \frac{F \cdot L}{b \cdot d \cdot \Delta L'}$$

wherein:	F	=	Rupture strain	[N]
	b	=	Probe width	[mm]
	d	=	Probe thickness	[mm]
	ΔL'	=	Change in length of the	[mm]
			length L due to the	
			load F	
	L	н	Test length	[mm]

AL' is the change in length given by a linear force/displacement curve. If the curve is not linear then a tangent is to be drawn to the curve which passes through the origin. The change in length L' is to be taken from this straignt line at the corresponding load F.

Interlaminar Shear Strength of Multidirectional Short /27 Bending Samples

Short bending samples are taken from the hardened laminate. The short bending samples are to be tested in a bending device as shown in Fig. 3-2 in such a way that the fibers in the 0° direction lie parallel to the bending strain.

The ratio between the distance between the supports and the thickness of the sample L/d must be 5:1, whereby, as with the bending samples, the distance between the supports for a single laminate can be constant.

During the testing a force/dispalcement curve is to be recorded. The reading is to be selected in such a way that the beginning of an interlaminar failure can also be clearly determined before the maximum rupture strain is reached.

The rate of load application must be 0.5 mm/min.

The interlaminar shear strength is computed using the following formula:

$$\boldsymbol{\tau} = 0,75 \frac{F}{b \cdot d}$$

wherein:	τ =	Interlaminar shear strength	[N/mm ²]
	$\mathbf{F} = \mathbf{i}$	Strain at the first failure	[N]
		(diagram)	
	b = ;	Sample width	[mm]
	d = 1	Sample thickness	[mm]

Since the test results are compared with desired values which are based on a fiber volume percentage of 60%, the fiber content of the laminates for bending samples and tensile samples must be determined. The test values are then corrected by a factor of 60/fiber content.

4. Tests During Manufacture

The checks and tests to be performed during the manufacture of a component part are specified in component-specific manufacture and control instructions. Normally such instructions contain references to:

- other instructions to be used
- the procedure to be followed such as
 - monitoring the room climate
 - monitoring the test instruments
 - processing instructions
 - hardening conditions
 - cutting instructions
 - surface treatment for adhesiveness
- check of individual manufacturing steps such as
 - cutting individual prepreg layers
 - layering the cut prepreg pieces
 - closing the form for hardening
 - hardening
- checks during final assembly such as during
 - riveting of individual elements
 - gluing
 - painting.

It is also true of fiber components that their quality is

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not only determined by the properties of the prepregs used. For this reason it is essential to perform destructive tests on the component part and/or on the so-called control laminates. The reason behind these tests is to make sure that the component $\underline{/30}$ is properly hardened and to verify that the prepreg used, which can only be stored for a limited amount of time, was still all right.

These points can be verified by preparing a control laminate along with the component part which is identical to a laminate taken from the receipt-of-goods. Over and above this the quality of the component parts must be determined by testing samples from the material left over from the component parts. Normally these samples are short bending samples used to determine the shear strength and samples used for microscopic examinations.

5. Non-destructive Testing of Component Parts

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- By component parts we mean here two kinds:
- a) finished single laminates
- b) finished component parts consisting of different individual laminates bound together in a superior unit.

With the individual laminates the testing is concerned with discovering internal and external defects.

With the glued and finished component parts the testing is restricted to merely the quality of the bond, since of course the individual layers have already been tested prior to being released for further processing, i.e. gluing.

Whether or not we are concerned with the laminate itself or the gluing bond, the object of the non-destructive testing is to detect all the defects (effects) which can have a negative effect on the strength or the component part. Thus the results of non-destructive testing serve as a basis for deciding whether the part can be used immediately, or only after being repaired or if it must be discarded.

5. 5.1. Requirements of Non-destructive Testing Procedures /32

If the non-destructive test can result in a decision on whether the part is to be used or discarded then the testing procedure must meet the following important requirement:

- reliable recognition of defects according to type and magnitude.

This goes hand in hand with these requirements:

- reproducibility of the results and
- ability to record the results for purposes of documentation.

For economic reasons the following demands must also be met:

- fast, efficient testing,
- fast output of data,
- sure recognition of defects, also by instructed personnel,
- possibility of inspections in the installed state.

It is almost certain that there is no procedure which simultaneously fulfills all these requirements. In this respect the requirement for reliability listed first is the primary measure for the usefullness of a testing procedure, even if the test lasts a long time or if the apparatus requires highly qualified service personnel, etc.

Moreover it happens that not all defects are detected by a /33

single testing procedure. As a result different procedures must be used for testing different types of defects, and naturally this right away calls in the requirement that the procedures be economical.

Nevertheless, one can assume that with increasing development activity in this field there will be further developments, extensions and refinements of the existing testing procedures which can then better meet the demands posed.

5.2. Possible types of defects

As already briefly mentioned, the following types of defects can appear in finished component parts:

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In Laminates

a) External Defects

In addition to irregularities of form and dimension inaccuracies the following defects can be present:

- surface cracks
- damage-due to improper handling
- poor surface condition
- rough edges due to poor mechanical processing.

Such defects can generally be detected visually or by penetration testing and in most cases -- depending on the extent -- result in the part in question being sorted out.

b) Internal Defects

- Wrong number of layers

Since the mechanical properties of a laminate depend heavily on the layers of which it is made, missing layers can lead to sericus consequences. To be sure it is /36 easy to determine the right number of layers after hardening (indirectly by measuring the thickness or by bending the laminate in the case of multidireclaminates), but it makes tional more sense to perform a check during the layering process so that the absence of 1 or more layers can be discovered in time.

check during the layering process.

Wrong layer The proper layer orientation
orientation decisively determines the direction-dependent mechanical
properties of the material. Here,
too, it makes sense to perform a

- Hardening Defects The degree of hardness affects the mechanical and aging properties of the material. For limited local variations, however, this defect is not very important because the resin goes through a second hardening with time.

- Hollow Cavities By this is meant all the hollow

spaces caused by the manufacturing process, for example air bubbles produced by careless processing. The presence of hollow spaces affects the load capacity of the laminate and can cause the laminate to be sorted out if the number of hollow cavities is large.

- Porosity As above.

- Delaminations These are also hollow cavities which are caused by the incomplete binding of two layers. To be sure, they appear only locally, but they represent a stress concentration factor which can cause serious damages. Over and above this, like the hollow cavities they weaken the crosssection, which gives rise to shear softness.
- Density Local fiber or resin concentrations variations likewise weaken the mechanical properties of the material and cause premature rupture if they extend over large areas.

- Bonding By this is meant the bond between the resin and contact surface of the fiber. Insufficient bonding also leads to a decrease in the mechanical porperties of the material.

- Inclusions By inclusions are meant foreign substances which can unintentionally get between the individual layers. They interrupt the continuous bond and can represent another stress concentration factor which can cause serious damage.

- Cut fibers Cut fibers not inconsiderably <u>/38</u> weaken the strength of the material transversely to the fiber direction.

In Gluing Bonds

Depending on which type of laminate is to be glued to another there are a number of defects which are characteristic for each special case. Here we will limit ourselves to only those defects which apply to all gluing combinations.

- Lack of glue A rare defect if the gluing is done with glue films. The lack of glue weakens the bond between the glued partners and causes shear softness which can cause further damage.
- Poor or no In most cases this is due to adhesive binding insufficient pretreatment of the layers to be glued together and

in time results in defective glue bindings.

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Poor cohesive This is due to local concentrations
binding of glue whereby the strength of
the glue binding drops sharply.
Results as above.

Other glue defects are:

- porosity in the glue
- poor splice gluing
- water inclusions in honeycomb gluings
- age-hardening defects
- foreign body inclusions in the glue
- unremoved protective layers

- incorrect positioning of the layers to be glued and many more.

5.3. Suitable Non-destructive Testing Procedures

There is a large number of testing procedures which are $/41^{1}$ suitable for revealing one or another type of defect.

Fig. 5-1 reviews which types of defects can be discovered with which testing procedures.

A cross in parentheses indicates that for this type of defect the procedure is either assumed to be suitable or has not yet been conclusively clarified.

It can be seen from this table that a large number of defects can be discovered using the following methods:

- X-ray

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- Neutronography
- Ultrasound
- Holography
- Resonance.

At Dornier we have decided to limit ourselves to these testing procedures, especially since the reproducability and ease with which the results of these methods can be recorded are very good.

For the ready installed airbrakes the mechanical impedance is also tested. The results of this procedure are discussed further on.

5.3.1. X-ray Method

X-rays are electromagnetic oscillations in the wave length range from 2 x 10^{-6} to 2 x 10^{-10} mm. Depending on their penetrating ability x-rays are generally divided into two groups between which it is not possible to draw a sharp line. These groups are:

the longwave x-rays, which are also referred to as soft radiation and possess low penetrability, and the shortwave x-rays, which are referred to as hard radiation and posess high penetrability (fig. 5-2).

The smaller the atomic weight of the object to be studied, the easier it is penetrated by x-rays.

Longwave x-rays are required for studying carbon fiber reinforced plastic components which have a low atomic weight. In order to produce longwave x-rays it is necessary to use a tube with small inherent filtration. Tubes with beryllium windows produce radiation over the entire wave spectrum in contr st to

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conventional industrial tubes which produce only hard radiation and thus are suitable only for irradiating materials of high atomic weight.

Long wave x-rays are produced by connecting a small voltage <u>/44</u> to beryllium tubes (Fig. 5-2). This follows from the following equation:



whereby I_1 , I_2 stand for the radiation intensities behind the non-defective and defective material, μ_1 stands for the attenuation coefficient of the material, μ_2 the attenuation coefficient of the defect and x stands for the length of the defect in the direction of the radiation.

If $\frac{-2}{I_1} = \frac{1}{1}$, then the radiation is not at all modified by the defect, in which case the defect does not show up.

If $I_1 \stackrel{12}{\longrightarrow} I_1$, then the defect becomes easier to detect the greater this number differs from 1.

I₂ Thus the ratio I₁ depends on μ_1 , μ_2 and x, and the greater 1 and x and the smaller μ_2 , then the greater this ratio.

Since the dimensions of an existing defect are constant, then when a smaller defect is found a larger one $(\mu_1 - \mu_2)$ must be sought if no reduction in the $\frac{I_2}{T_1}$ ratio is to occur.

Also for carbon fiber reinforced plastic, the smaller the tube voltage, the greater the attenuation coefficient. Therefore smaller defects must be tested with smaller voltage and/or softer radiation. Moreover, we can only get a sharp picture when the radiation source is a point source or the film is directly in back of the object. In practice it is not possible to make a point source radiation source, nor is it always possible to put the film directly behind the object. For this reason every x-ray photograph has a certain lack of definition, but the smaller the radiation source, Fig. 5-3, (focal point) the smaller this effect and the closer the film can be placed to the object.

Finally, the ability to detect failures can also be decisively influenced by the selection of the proper film, i.e. in terms of sensitivity, quality, grain size.

An adverse factor for the detection of defects in carbon fiber reinforced plastic laminates is the fact that the length of the defect in the direction of radiation, which is determined by the layering of the individual prepreg layers, is very short. However, with properly adjusted instruments it is possible to detect the smallest defects.

In Fig. 5-4 small hollow cavities as fine as a strand of hair can clearly be seen in a multidirectional laminate 4.6 mm thick.

Other defects which were detected at Dornier are the following:

- cut fibers

 density	differences	(Fig.	5-9)
 hollow c	avities	(Fig.	5-4)

- thickness differences (Fig. 5-9)
- inclusions (metallic (Fig. 5-8)
 - and non-metallic)

(Fig. 5-8)

In addition, it is planned to check the number and direction <u>/46</u> of layers using the x-ray method by imbedding fine wire in the

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laminate.

In testing glue bindings it is more difficult to use the x-ray method. If one of the layers being glued is metallic it is even impossible to test the quality of the glue binding. In this case other methods must be used.

In the case of carbon fiber reinforced plastic layers glued together or in the case of carbon fiber reinforced plastic glued to honeycomb it was possible to detect the following defects:

- porosity of the glue
- places without glue in the case of a carbon fiber reinforced plastic cover laminate glued to a Nomex honeycomb
- defective splice glvings
- water inclusions in honeycombs
- honeycomb deformations
- incorrect positioning of the two pieces being glued together.

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5.3.2. Neutronography Method

The first neutron radiography experiments and photographs were made in 1935 in Germany by Kallmann and Kuhn. Extensive application of neutron radiography (Neutronography) first began in 1960 in various laboratories.

Since the nonmetallic components, for example in glued combinations of carbon fiber reinforced plastic and aluminum, are stronger neutron absorbers than metallic combination components, this property stands in sharp contrast to pentrating radiation such as gamma rays, beta rays and x-rays.

These types of radiation require a large amount of energy in order to penetrate the outer metal layer, whereas the low density and small atomic weight of the glue and fiber material make it impossible to simultaneously get a picture of the carbon fiber reinforced plastic. For this reason gamma rays, beta rays and x-rays are not to be used for testing combinations of this type glued together. A check of the mass absorption coefficient as a function of atomic weight (Fig. 5-5) shows that the attenuation of x-rays increases with increasing atomic weight. By contrast the attenuation of thermic neutrons is completely independent of atomic weight.

From Fig. 5-5 it can be seen that the absorption of neutrons by glues, resins and borosilicate (see points H and B in Fig. 5-5) is significantly greater in comparison with aluminum.

Thus the nonmetallic portions are important for the photographic density on the film.

In contrast to the strongly divergent x-rays, in neutron radiography arrangements are used exclusively which produce almost parallel neutron rays. This produces a parallax-free picture.

In making the photographs a different reporduction is used than with x-rays which shortens the illumination time by a factor of 50 to 100. Test of the laminate/aluminum.

Test of the Laminate/Aluminum Bond

For suitable thermic neutron study the sample as shown in Fig. 5-6 was made available to the Karlsruhe Nuclear Research Center. The results of the irradiation can be seen in Fig. 5-6. (Glued in aluminum strip in the lower edge of the picture.) /48
In contrast to the x-ray photograph, the less absorbent aluminum component can clearly be seen. Separating films and teflon strips glued onto the aluminum cannot be seen however. It is just possible to make out the 45° fiber orientation in the carbon fiber reinforced plastic laminate.

Test of the Laminate/Honeycomb Combination

Here the missing glue between the honeycomb and carbon fiber reinforced plastic covering laminate is shown in strong contrast (Fig. 5-6). Likewise visible are the triangles of separating film inserted between the glue and honeycomb. The glue dispalced by the separating film built up around the edges of the triangle. Also clearly visible are:

- the small bubble structure in the glue layer between the cover laminate and the honeycomb core,

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- the very absorbent splice core and
- the parallax-free image of the honeycomb contour.

5.3.3. Ultrasound Method

The non-destructive test of metallic materials using ultrasound has won wide success because of the excellent results. The strength of this method in comparison with the x-ray method consists in the fact that it can be used in practice for materials of almost any thickness, whereby the depth of the defect can also be determined.

Ultrasound belongs to the group of mechanical tests and includes a range of 20 kHz to 100 MHz. For testing puproses the range of 0.25 to 24 MHz is used.

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In practice ultra sound is used mostly according to the magnetostrictive, peizoelectric and electrostrictive methods.

For testing purposes the following methods can be used in practice:

- impulse-echo method
- ultrasonic wave irradiation method
- resonance method.

The Impulse-Echo Method

In general two versions of the sound reflection method are used today in the metal industry for detecting defects. Both methods have the following property in common: the electronically created electric vibrations of the piezoelectric crystal are converted into ultrasonic vibrations. The beam of vibrations /52 is sent out from the transmitter end of the instrument into the interior of the object being tested and, being reflected from the opposite boundary layer, it strikes the second peizoelectric crystal located in the receiver end of the instrument.

In another version of the ultrasonci method only one piezoelectric crystal is used instead of two and this single crystal is responsible both for transmitting and receiving the reflected waves.

The advantage of the second method is that it can be performed with more convenient portable instruments and, in particular, only one point on the section of material be tested has to be accessible.

The Ultrasonic Wave Irradiation Method

This method is based on the measurement of the decrease in sound intensity. If a defect in the material, such as an inclusion, a cavity, etc., gets in the path of the ultrasonic waves when they are passing through the portion of material being tested, then the entering energy is reduced far in excess of the otherwise normal deviation as a result of the amount of energy absorbed and/or reflected by the defect. Thus only a sharply reduced amount of energy reaches the head portion of the piezoelectric crystal being used as the receiver.

The method is very fast and convenient and it allows the shape and point of the defect to be recorded. It has the disadvantage, however, that it can only be used on samples with parallel surfaces. The depth of the defect cannot be determined with this method.

Both of these methods are used for testing carbon fiber reinforced plastic laminates.

Because of the imhomogeneity of carbon fiber reinforced plastic $\underline{/5}$ laminates it is difficult to test them using ultrasound because the inhomogeneity causes sound attenuation, reflections and deviations of the sound beam. As a result the monitor readings are difficult to interpret. Reliable interpretation of the signals requires accurate knowledge of the consistency of the material, the laminate structure, e.g. thickness flaws, and extensive experience of the tester in dealing with carbon fiber reinforced plastic components. On the other hand, it is advantageous for ultrasonic testing that the most important defects, such as delaminations, air bubbles and inclusions, which should be detected by ultrasonic irradiation, for the most part lie perpendicular to the direction of the sound waves. This is

due to the layered structure of the laminates.

In addition, by appropriately setting the ultrasound instrument one can determine before hand the size of the defects to be indicated. This makes the testing simpler, more efficient and easier to understand (Fig. 5-15).

In the testing of mass produced parts it is advisable to use semi and fully automatic testing systems which mover the piece being tested along a step as a time and give an exact reading of the defect points. By this method the piece being tested is immersed completely in water (immersion technique), whereby the testing head and the test sample is produced by means of the surrounding water. This technique can be used either with the pulse-echo method or with the ultrasonic irradiation method.

Using such systems has the following advantages in comparison with manual testing:

- fast, efficient testing
- high reproducibility
- good recordability
- noncontact and thus wear free testing
- optimal coupling.

The result shown in Fig. 5-15 is particularly impressive. This concerns a C-scan recording of a defective laminate 4.6 mm thick using the pulse-echo method. This was recorded by a small laboratory apparatus produced by Automation Industries in Rotterdam.

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Key: A) Width

- B) Height
- C) Thickness of the material
- D) Threshold value

With this apparatus it is possible to adjust a threshold value which can be varied both in height and width. By varying the height we can determine the size of a defect, and by varying the width we can determine the depth range of a defect (a).

If the threshold value is set on the rear wall echo then the recorder registers "defect" as soon as the echo off the rear wall -- attenuated by absorption or reflection due to a defect in front of the wall -- remains beneath the threshold value (b).

The lower the threshold value setting, the larger the defects which are registered by the recorder. This relationship can clearly be seen in Fig. 5-15.

With a threshold setting of 32.5% of the tubescreen -- set $\frac{55}{55}$ for the rear wall echo -- the C-scan recording correspond fairly precisely to the x-ray picture.

If the threshold value is set lower (25%) then smaller defects

are no longer recorded.

On the other hand, if the threshold value is set at 50% then the recorder registers a large number of smaller defects as well which are shown by the large white areas on the recorded scan. Thus, depending on the size of the defects which one is looking for, it is possible to determine this size by appropriately setting the threshold value.

At Dornier the following defects have been detected using ultrasound:

***	delaminations	(Fig.	5-7)	
-	inclusions	(Fig.	5-8)	
-	hollow spaces	(Figs	. 5-4,	5-15).

In testing gluing bonds the following defects have been detected:

- no adhesive binding (Fig. 5-11)
- foreign bodies in the glue layer
 poor cohesive binding (Fig. 5-10)

5.3.4. Resonance Method

This method is based on the following principle: the object to be tested is caused to co-vibrate by a transmitter whose oscillatory circut is adjusted so as to have the same vibration frequency as one of the inherent vibration frequencies of the object.

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The defect is evaluated on the basis of changes registered in the resonance frequency and amplitude.

This test is generally refered to as the Fokker-Bond test.

The testing head of the instrument consists of an oscillator crystal which is excited to resonace frequency (eigen frequency f_1) by means of an alternating current.



Key: A) Glue B) Honeycomb

By connecting a substance (here the upper cover sheet) to the oscillator this creates a new vibration system with a different eigen frequency and amplitude (a). The eigen frequency of the system drops to frequency f_2 . This frequency is the reference frequency and indicates zero gluing. The frequency amplitude is likewise a reference value (100%).

If the oscillator crystal is connected to the glued cover $\frac{58}{58}$ (b) another downward resonance shift occurs (frequency f_3). The resonance shift depends on the thickness of the adhesive, which works as a spring, and the mass of the cover s_2 . The amplitude drops insignificantly to a value < 100% (fall value on the A scale).

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If an oscillator crystal is connected to a sandwhich (c) hardly any frequency change occurs in comparison with the situation in which it is connected to the single cover of thickness s_1 . This is due to the fact that the mass of the honeycomb is small. The vibrations are strongly absorbed. With good gluing the amplitude drops off sharply (fall value on the B scale).

Testing of gluing bonds between two carbon fiber reinforced plastic laminates with the Fokker-Bond tester has not yet been sufficiently tried out.

The anisotropic structure of carbon fiber reinforced plastic laminates is an obstacle to the continuity of the vibrations over the entire surface. Moreover, even slight changes in thickness of a laminate with one glue layer considerably affect the oscillatory behavior on the testing head.

To be sure, we at Dornier were able to sporadically detect the following defects:

- places lacking glue

- no adhesive binding

- poor cohesive binding.

However, a systematic and comprehensive test series is required in order to statistically insure the results and plot performance curves.

The first results available in this series of investigations are test values of tapered laminates consisting of two carbon fiber reinforced plastic layers glued together and carbon fiber reinforced plastic glued to a honeycomb layer.

Since in the case of carbon fiber reinforced plastic layers /59

glued together we are basically dealing with the determination of resonance frequency shifts, the so-called A scale of the Fokker-Bond tester is the main indicator of defects. Fig. 5-12 shows the relationship between the indicating signal of the test instrument and the glue thickness. A continuously increasing thickness of the glue layer between 0.1 and 0.7 mm was produced by inserting small wedges. It was possible to detect a clear change in resonance which is expressed in the scale divisions on the A scale.

Fig. 5-13 shows the test results plotted for a second laminate combination. The resonance shift as a function of the glue thickness is more pronounced. Presumably this is connected with the fact that the testing head is better coordinated with the thickness combination of the laminates.

Because of the anisotropy of prepreg laminates the attempt was not made to empirically set up generally valid quality curves. Rather, for the individual components to be tested the approved instrument readings were determined by means of master pieces and published as a part of the testing instructions pertaining to component parts.

A similar program must be carried out for laminate/honeycomb combinations in order to determine the absorption which is characterized in the Fokker-Bond test by the values on the B scale.

The procedure is shown schematically in Fig. 5-14. Here too the glue layer is tapered by inserting a wedge. The dependence of the frequency amplitude, expressed in scale divisions on the B scale, on the thickness of the glue layer clearly stands out.

5.3.5. Holography

Holography has been reported on in detail in the lecture given by K. Grünewald entitled "Holographic Determination of Thermic and Mechanical Deformations in Components and Structures Used for Aerospace Applications." It is therefore unnecessary to discuss this subject within this lecture.

5.3.6. Impedance Method

If a body is excited to mechanical vibrations its response can be determined with measuring instruments (for example by measuring its mechanical impedance Z). It is a vectorial quantity defined as the quotient of the exciting force P divided by the vibration velocity V.

$$Z = \frac{P}{V} \left[\frac{N}{m/sec}\right]$$

If Z is plotted over the frequency a typical impedance curve will result dependent on the shape and condition of the body. If the shape and condition of the body, for example due to manufacturing defects or damages incured during service, then the impedance curve will also change.

The evaluation of this effect forms the basis for the "impedance" test method. Two conclusions follow from what has been said above:

- Data on defects is possible only in the light of a nondefective reference structure whose behavior must be known beforehand.

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- Only those defects are detected which affect the behavior of the vibrations, i.e. which cause a measurable loss of rigidity.

Initial preliminary studies gave satisfactory results with good reproducibility. With structural elements, such as a flat bar and skin-stringer joint, and a complete Alpha-Jet airbrake it was shown that the following defects can be detected by impedance measurements:

- breaks in the resin
- glue defects in the laminate
- breaks in the fibers.

For the most part the defects were detected by the shift in resonance frequencies. The changes were on a order of magnitude of 2-10% for an instrument-dependent resolution of < 1%. They suggest that by measuring mechanical impedance or a similar quantity a high quality testing procedure can be developed.

6: Prospects

The problem of non-destructive testing of carbon fiber reinforced plastic components is solvable.

Although the field of application is relatively new it is already possible to detect a large number of defects with existing methods and instruments. Adaptation and further development of testing procedures and test instruments for carbon fiber reinforced plastic material will certianly expand the possibilities in the near future and further increase the reliability of the test results.

Up to now, for example, neutronography equipment could only

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be used as laboratory apparatus in the laboratories of nuclear power centers. According to the latest information neutronography equipment is already being developed in the form of mass produced instruments for use in industry.

As a further example, Dornier is presently testing the imporved application of the x-ray method for evaluating the quality of the glue in carbon fiber reinforced plastic components. This is being done by increasing the absorption of the glue which in turn imporves the detectability of defective gluings.

In addition it is expected that in the near future it will be possible to specify the defect depth in laminates by means of the 3-dimensional x-ray method.

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Fig. 3-1. Flow diagram for destructive testing procedure used in the production of carbon fiber reinforced components. [Key on following page].

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- Key: A) Prepreg
 - B) Fiber
 - C) Resin
 - D) Receipt-of-goods check Verification of required prepreg properties by test certificates of the supplier
 - E) Receipt-of-goods check User checks prepreg properties in the delivery state
 - F) Comparison of actual and desired values
 - G) Requirements fulfilled
 - H) No
 - I) Rejection
 - J) Yes
 - K) Laminate sample
 - L) Receipt-of-goods check Verification of required mechanical properties by test certificate of the supplier

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- M) Receipt-of-goods check User checks properties in age-hardened state
- N) Release for processing
- 0) Testing of samples from leftover component material
- P) Testing of samples manufactured along with the component
- Q) Rejection
- R) Panel deciding on the use of the material
- S) Release for nondestructive testing



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Key:

A) Unidirectional long bending sample B) Fiber orientation

- C) Bending device for long bending samples
- D) Flat tensile tests
 E) Multidirectional short bending sample
 F) Device for short bending samples
 G) Radius of supports

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Fig. 5-1. Non-destructive testing procedures for carbon fiber reinforced plastic components. [Key on following page]

- Key: A) Laminates
 - B) Glue layers
 - C) Type of defect
 - D) Poor surface
 - E) Rough edges
 - F) Surface cracks
 - G) Incorrect fiber orientation
 - H) Age-hardening defect
 - I) Hollow spaces
 - J) Porosity
 - K) Thickness differences
 - L) Cut fibers
 - M) Delaminations
 - N) Inclusions
 - 0) Crack
 - P) Places without glue
 - Q) Poor adhesive binding
 - R) No adhesive binding
 - S) Poor cohesive binding
 - T) Splice gluing
 - U) Position of the components
 - V) Damaged components
 - W) Water Inclusions
 - X) Foreign body in the glue layer
 - Y) Testing procedures
 - Z) Visual test

- AA) Penetration test
- BB) Ultrasonic pulse-echo method
- CC) Ultrasonic sound wave irradiation method
- DD) Neutronography
- EE) X-ray
- FF) Holography
- GG) Impedance
- HH) Thermography
- II) Fokker-Bond
- JJ) Resonance test
- KK) Assumed or not yet fully clarified
- LL) Carbon fiber reinforced plastic glued to carbon fiber reinforced plastic

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- MM) Carbon fiber reinforced plastic glued to aluminum
- NN) Honeycomb sandwhiched Between two carbon fiber reinforced plastic layers
- 00) Only with aluminum-honey comb







Key: A) Non-destructive testing procedures

for carbon fiber reinforced plastic components.

- B) X-ray photograph of the flange regions
 - of an airbrake shell





Fig. 5-6. Neutronography photograph of a standard testing sample [Key on following page].

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- Key: A) Non-destructive testing procedures for carbon fiber reinforced plastic components.
 - B) Neutronography photograph
 - C) Standard test sample
 - D) Nomex honeycomb
 - E) Aluminum honeycomb
 - F) Recess in glue sheet
 - G) Honeycomb depression, top
 - H) Separating film, bottom
 - I) 0.3 mm Milled recess, underside
 - J) Good gluing

 - K) Teflon layer, undersideL) Carbon fiber reinforced plastic
 - M) Splice gluing
 - N) ... oneycomb



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Fig. 5-7. Micrographs of a delamination in fine hollow spaces in the flange region of an airbrake shell. [Key on following page] Key:

- A) Magnification 0 times
- B) Magnification 50 timesC) Delamination in the flange section Non-destructive test: Ultrasound 25-times
- D) Hollow spaces in the flange section of the shell

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- Non-destructive test: x-rays
- E) Non-destructive test procedure for carbon fiber reinforced plastic components



Fig. 5-8. X-ray photograph of a laminate with an inserted bit of paper.

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Fig. 5-9. X-ray photograph of a laminate with different thicknesses and densities.

Key: A) Variations in density B) Laminate thickness

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Fig. 5-10. C-scan recording of a poor cohesive binding [Key on following page].

Key: A) Glue

- A) Glue
 B) Aluminum-honeycombs
 C) Carbon fiber reinforced plastic
 D) Ultrasound C-scan (sandwhich)
 E) Ultrasound C-scan (laminate)
 F) Increase in glue layer thickness
 G) Non-destructive test procedures
 for carbon fiber reinforced plastic components

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Fig. 5-11. Ultrasonic C-scan recording of a standard test sample with specific gluing defects [Key on following page].

- Key: A) Non-destructive testing procedures for carbon fiber reinforced components
 - B) Ultrasound C-scan (sandwhich)
 - C) Standard test sample D) Nomex honeycomb

 - E) Aluminum honeycomb

 - F) Recess in glue sheet G) Honeycomb depression, top
 - H) Separating film, bottom
 - I) 0.3 mm Milled recess, underside

 - J) Good gluing K) Teflon layer, underside
 - L) Carbon fiber reinforced plastic
 - M) Splice gluing
 - N) Honeycomb

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Fig. 5-12. Relationship between the scale reading of the A scale of the Fokker-Bond tester and the glue thickness [Key on following page].
Key: A) Test points at intervals of 16 x 20 mm

- B) Glue thickness
- C) Testing head D) Laminate thicknesses

E) Left

- F) Position of the wave head G) Right



Key: A) Test points at in- C) Left tervals of 8 x 20 mm D) Position of the wave head B) Glue thickness E) Right

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- D) Testing head
- E) B scale [Scale divisions]

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Fig. 5-15. C-scan of a defective laminate 4.6 mm thick for different threshold value settings (25%, 32.5%, 50%) in comparison with an x-ray photograph.

Key: A) X-ray

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4