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Test method for the evaluation of the adhesive properties of fibre reinforced polymer composite joints

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This CEN Workshop Agreement (CWA 17896:2022)has been developed in accordance with the CEN-CENELEC Guide 29 “CEN/CENELEC Workshop Agreements – A rapid prototyping to standardization” and with the relevant provisions of CEN/CENELEC Internal Regulations - Part 2. It was approved by a Workshop of representatives of interested parties on 2022-04-06, the constitution of which was supported by CEN following the public call for participation made on 2021-07-26. However, this CEN Workshop Agreement does not necessarily include all relevant stakeholders.

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Introduction

Advanced fibre reinforced polymer composites, due to their lightweight, are used in aeronautics, aerospace, automotive, and naval activities (e.g., aircraft fuselage, wind turbines, gears, chassis, etc.). Skin-stiffened or "stringer run-outs" structures are used mostly in aerospace and are very sensitive to local damages. Usually, the stringer tends to debond from the skin, and then the delamination may further propagate in the skin. The mechanical characterization of these specimens is both time-consuming and material intensive.

This document describes a modified test method used in a European project to characterize delamination at the tip of the flange and to understand 'stringer run-out' experienced in the manufacture of composite large panel, typically greater than 0,5 m in any in-plane direction. The method employed a simplified joint configuration via a lap-strap geometry. The results of the work showed that the simplified lap-strap specimens showed the same damage mechanisms as the stringer run-out.

Firstly, the lap debonds from the strap and then the delamination may further propagate interplay in the strap. It should be mentioned that failure in the lap -strap geometry is manifested in a mixed-mode. At the early stages of the test, the adhesive layer between lap and strap fails in mode II, followed by mode I failure at higher stress levels. This test method could also be used to evaluate the healing or repair efficiency at self-healing or repairable composites or their knockdown effect (see 5.6).

Non-destructive Evaluation (NDE) techniques, for example Acoustic Emission, can be optionally applied to the Lap Strap specimen with the mechanical testing. NDE techniques include Electrical Resistance Change Method (ERCM) and Acoustic Emission (AE). These techniques could provide information about the failure of the geometry and, additionally, information about the damage that was induced before failure. They are strongly suggested in cases of poor mechanical properties of the adhesive.

1 Scope

This document provides a test method for the determination of the adhesive properties in joints of continuous fibre reinforced polymer matrix composite structures using the Lap Strap specimen.

The evaluation includes the optional concurrent use of the non-destructive technique of the Electrical Resistance Change Method (ERCM) and/or Acoustic Emission (AE) for the monitoring of the debonding of the lap from the strap optionally. The ERCM NDE technique has a limited application only on carbon fibre composites due to the inherent electrical conductivity of the carbon fibres.

This test applies to composites manufactured with continuous carbon fibres (woven or unidirectional) and thermoset or thermoplastic matrices, with quasi-isotropic lamination. This methodology can be used on repairable or self-healing composites in order to estimate the repair or healing efficiency respectively.

Safety aspects about manufacturing and mechanical testing of the composites are excluded.

2 Normative references

There are no normative references in this document.

3 Terms, definitions, symbols and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the following terms, definitions, symbols and abbreviated terms apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1.1
non-destructive evaluation
NDE
process or procedure for determining the quality or characteristics of a material, part or assembly without permanently altering the subject or its properties

[SOURCE: ISO 21648:2008, 2.1.29]

3.1.2
on-line monitoring
any inspection activity carried out concurrent with the mechanical testing

3.1.3
knockdown effect
the change of the initial mechanical properties of a composite material after the incorporation of a self-healing or a self-repairing system

3.1.4
balanced laminate
continuous fibre-reinforced laminate that each +θ° (angle) lamina is balanced by a -θ° (angle) lamina in regard to a reference axis

3.1.5
symmetric laminate
continuous fibre-reinforced laminate that each ply above the mid-plane is identically the same in terms of position and orientation with one below the mid-plane

3.2 Symbols and abbreviated terms

AE	Acoustic Emission
b	measured specimen width
E	apparent stiffness
ERCM	Electrical Resistance Change Method
F^{max}	value of the load at the drop-off point
g	measured grip-to-grip distance
h	measured lap thickness
l	extension
NDE	Non-Destructive Evaluation
R_0	Initial value of the resistance
ΔR	The final value of the resistance minus the initial value of the resistance
ε	lap strap strain
θ°	angle
$\sigma_{max}^{Initial}$	lap strap strength from a initial composite
$\sigma_{max}^{Modified}$	lap strap strength from a modified composite
σ^{max}	lap strap strength at the drop off point
$\sigma_{max}^{Reference}$	lap strap strength from a reference composite
$\sigma_{max}^{Repaired}$	lap strap strength from a repaired composite

4 Significance and Use

The most common failure mode of stiffened composite panels is the debonding between the skin and the stringer. The proposed test method provides the mechanical strength of the lap strap specimen that simulates the mechanical and failure behaviour of a stiffened panel and reduces consumption of the materials and manufacturing time. The geometry described is a simplified joint configuration that reduces consumption of the materials and manufacturing time, while simultaneously indicating similar damage mechanisms as the stringer run-out configuration. The results from this test method can provide information about the damage mechanisms of such composite structures made from the specific materials. Furthermore, for repairable or self-healing structures, it may establish quantitatively the knockdown effect between the conventional and repairable composites. Therefore, it should offer quantitively knowledge about the values before and after repair in order to assess the repair efficiency of these structures.

5 Lap Strap Geometry

5.1 Sampling

At least five specimens should be tested per test condition. The use of fewer specimens should be avoided. For statistically significant data, the procedures outlined in ASTM E122 should be followed. The number of specimens shall be reported.

5.2 Laminate Configuration

Multidirectional quasi-isotropic laminates shall be tested which should be both balanced and symmetric with respect to the span direction of the specimen. The lamination sequence should be $[45/0/-45/90]_{NS}$ if a unidirectional tape used or $[(+45/-45)/(0/90)]_{NS}$ if a woven fabric is used, where N is a whole number. The thickness of both the lap and the strap should be between 2 mm to 5 mm. The thickness and the lamination sequence of the lap, the strap and the endtab have to be the same. The variation in thickness for the individual parts of the lap strap specimen shall not exceed 0,1 mm. The panels may be manufactured by hand or wet lay-up method or tow-placement and moulded by (hot) press, vacuum bag, autoclave, or resin transfer moulding. The panels should be free of voids or should be calculated according to ASTM D2734. The volume content should be reported in accordance with ASTM D3171.

5.3 Specimen Dimensions

The typical configuration for the lap strap specimens is shown in Figure 1. The dimensions of the individual parts of the specimen shall be the following:

Lap length & width	= 100 mm & 20 mm
Strap length & width	= 200 mm & 20 mm
Endtab length & width	= 50 mm & 20 mm

Measure the width and the thickness of each part to the nearest 0,05 mm at the midpoint and from either end. The average values of the measurements should be recorded and reported.

5.4 Specimen preparation

5.4.1 General

The specimen preparation is important for this geometry. The parts of the specimens should be cut from the same laminate or should be moulded to the desired final dimensions. Edges should be flat and parallel without any fluctuations at the surface. A method that produces sharp cut edges should be used as cutting method.

5.4.2 Surface preparation

The bonding surface of each part may be rough by lightly grinding or scrubbed with sandpaper and then they should be cleaned with a volatile solvent such as acetone or ethanol in order to remove the residuals. The surface preparation process should be performed according to ASTM D2093 or EN 13887:2003 or ISO 17212:2012 and shall not affect the composite properties.

5.4.3 Joint procedure of the measured bonding area

The adhesive (film, resin, etc.), as defined in ASTM D907 and ASTM D4800, should be applied in accordance with the manufacturer's guidelines between the lap and the strap (measured bonding area). In case of a two-part adhesive, the mixture shall be prepared according to the supplier’s directions. The curing of the adhesive shall be performed according to supplier recommendations and conditioning should be done according to ASTM D618. The thickness of the adhesive bonding may be varied, with a minimum thickness of 0,2 mm to 0,4 mm. The control of the thickness can be achieved using the minimum number of metal beads that are needed. A maximum pressure of about 3 bar is acceptable. Final specimens should be allowed to cool down to room temperature for at least 3 h if an elevated temperature is used for the curing. If the curing is performed at room temperature, allow full-cure time plus 20 % prior to the testing. If no additional adhesive may be used and the welding process is followed, the initial thickness of the lap and the strap should be remained. After the bonding process of the lap to the strap, the end tab can be secondary bonded.

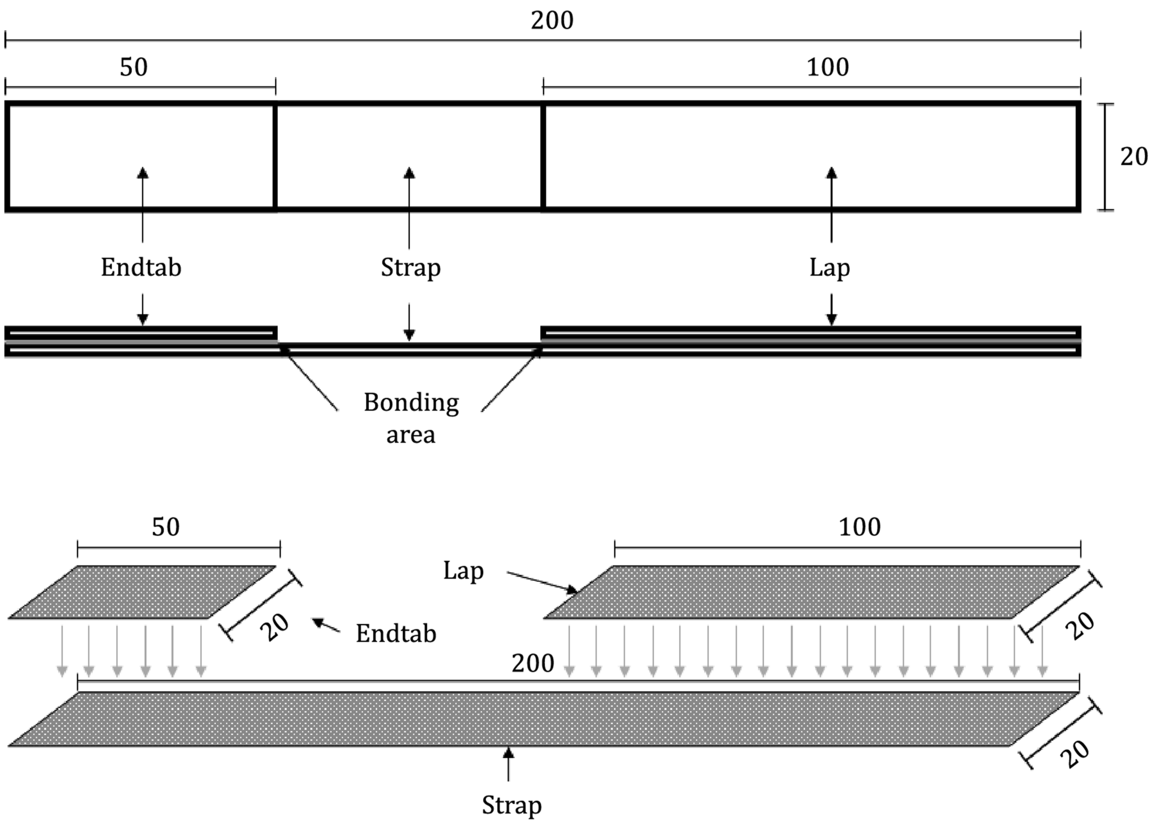

Figure 1 — Geometry of the Lap Strap specimen

5.5 Conditioning

Conditioning should be done according to ASTM D5229, Procedure C, or EN 2743:2001, unless different environmental conditions is part of the experiment. The storage should take place at a standard atmosphere of 23 ± 3 °C and 50 ± 10 % humidity. If a dry condition is measured, the conditioning should be done according to ASTM D5229, Procedure D, or the EN 2823. Any other conditioning (dry or wet) prior to testing shall be reported.

5.6 Repairable or self-healing composites

For repairable or self-healing composites, the specimens shall be manufactured as described at the previous subclauses. The repair/self-healing technology (capsule-based, vascular, intrinsic polymers, vitrimer technology, etc.) shall be applied at least at the bonding zone between the lap and the strap (Figure 2). The specimens shall be initially tested as described in 6. After the initial testing, the appropriate method of triggering the repair or self-healing method shall be applied (according to the self-healing or repair technology employed, e.g. pressure, temperature healing agent injection, or combination of those). When the repair or self-healing process is complete the specimens are tested again, according as described 6. The knockdown effect shall be calculated from equation 4 (Section 8.4) and the repair efficiency shall be calculated from equation 5 (8.5), for each specimen separately. If the applied technology allows it more than one, self-healing/repair cycles can be applied according to the above.

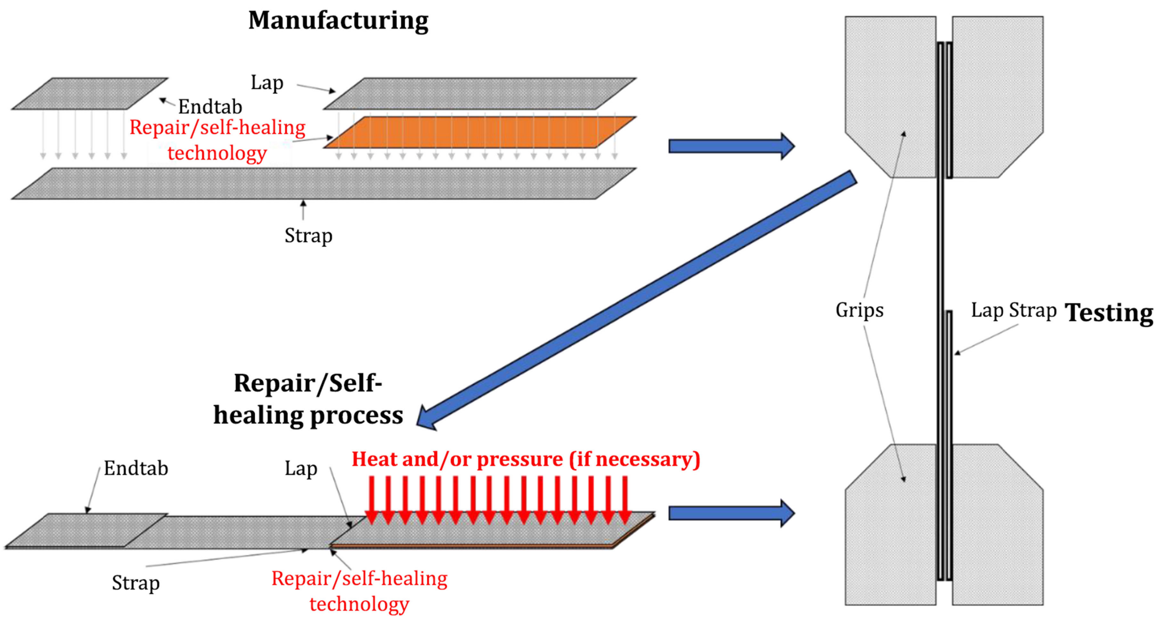

Figure 2 — Process for the testing method on self-healing or repairable composites

6 Mechanical testing

6.1 Testing machine

A calibrated testing machine shall be used at constant rates of crosshead motion in the range from 0,5 mm/min to 2 mm/min. It shall be equipped with a strain measuring device. The strain may be estimated from the crosshead displacement provided the deformation of the machine with the grips attached is less than 1 % of the extension of the test specimen. If not, then the extension should be measured from a properly calibrated external extensometer attached to the specimen. The load measuring system error should not exceed ±1 % of the maximum load that is expected to be measured. An X-Y plotter or a similar device may be used in order to record during the test the load versus extension at the point of load application.

6.2 Grips

Each head of the testing machine shall be equipped with grips bigger than 50 mm at length to hold the specimen. The force applied to the specimen should be in coincident with the longitudinal axis of the specimen. The grips should apply enough pressure in order to avoid the slippage effect between the grip and the specimen. It is recommended to use grips that are rotationally self-aligning to minimize bending stresses in the coupon that reduce the composite properties.

6.3 Placement of the specimen

Insert the specimen into the testing machine as shown in Figure 3. The specimen should be placed from both edges at the upper and bottom grip. The endtab shall be inside the upper grip at its all length (50 mm). The other edge, that is the lap, and the strap shall be inside the bottom grip at 50 mm ± 0,5 mm leaving an initial grip-to-grip distance of 100 mm. The grips should be tightly closed without reducing the specimen thickness and composite properties.

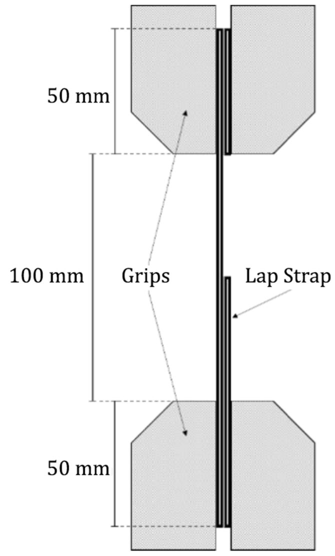

Figure 3 — Placement of the specimen into the grips

6.4 Testing process

The speed of testing shall be at a rate of crosshead movement of 1 mm/min under tensile mode. The test shall be stopped at a load drop-off of at least 5 %. The debonding of the lap from the strap should be visible (Figure 4). Alternatively, the test shall be stopped when the lap debonds from the strap optically.

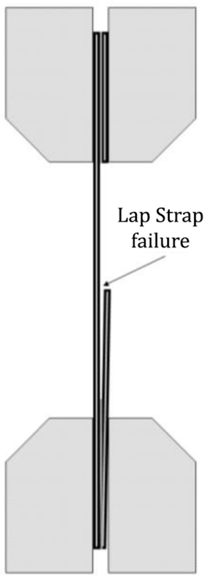

Figure 4 — Debonding of the lap from the strap.

7 Non-destructive evaluation (NDE)

NDE techniques can optionally be applied to identify the failure of the Lap Strap, especially in case of poor adhesiveness between the lap and the strap, where the critical failure stress is not distinct at the stress-strain plots. The suggested techniques are ERCM and AE.

Electrical Resistance Change Method (ERCM) can be optionally utilized on the Lap Strap geometry to monitor the deformation and specimen failure. ERCM can be applied only on Lap Strap specimens manufactured by carbon fibres. The two-probe method is suitable for the specific geometry. The preparation of the specimens requires two electrodes (e.g., sort copper wires) and a conductive adhesive (e.g. silver conductive paste). The surface should be grinded with sandpaper until the carbon fibres are exposed in the locations shown in Figure 4. The dimensions of this area should be sufficient to be adequate for a successful adhesion of the electrode. The exposure of the carbon fibres can be confirmed using a multimeter. The measured resistance at two immediate areas should be equal to the resistance of the carbon fibres. The results of the ERCM should be illustrated in double-X $\Delta R/R_0$ and stress versus time plots. Failure should be designated by a sharp increase of the resistance, while the deformation by a continuous decrease of the resistance.

The Acoustic Emission (AE) can provide crucial information about the failure of the Lap Strap. An accumulation of acoustic events should designate the failure. One piezoelectric AE sensor should be placed in the designated area of Figure 4. A coupling agent (e.g., Ultrasonic gel) should be used between the sensor and the specimen. The sensor should not be detached at the areas where the electrodes of the ERCM were positioned. It is recommended the sensor be placed at the designated-on Figure 5.

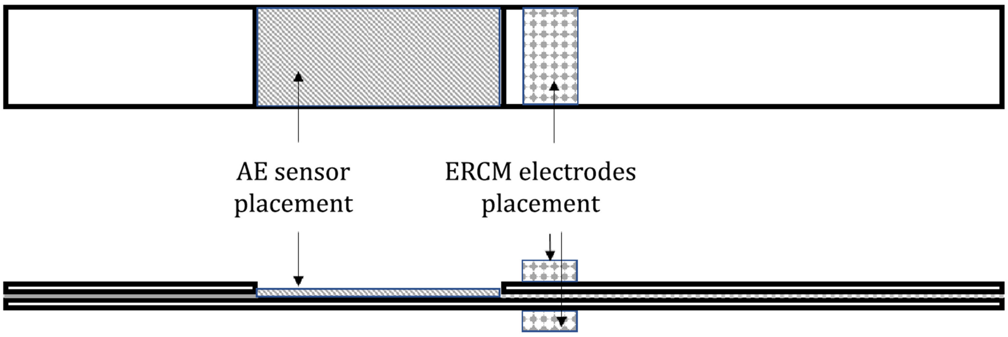

Figure 5 — NDE schematic setup on Lap Strap geometry

8 Calculation

8.1 Maximum strength (MPa)

Calculate the lap strap maximum strength, in megapascals (MPa) using the following equation:

$$\sigma^{max} = \frac{F^{max}}{h \times b} \tag{1}$$

where

σ^{max}	lap strap maximum strength, MPa;
F^{max}	value of the load at the drop-off point, N;
h	measured lap thickness, mm;
b	measured specimen width, mm.

NOTE Lap shear calculation is performed the same way in ASTM D3163, EN 1465:2009, EN 2243-6:2005, ISO 4587:2003 and ISO 22841:2021.

8.2 Strain

Calculate the lap strap strain using the following equation:

$$\varepsilon = \frac{l}{g} \times 100 \qquad (2)$$

where

- ε lap strap strain, %;
- l extension (or displacement), mm;
- g measured grip-to-grip distance, mm.

8.3 Stiffness

The apparent stiffness is calculated from the slope of the line (Δ_y / Δ_x) of the measured stress-strain plot using the following equation (see Figure 6):

$$E = \frac{\Delta y}{\Delta x} \qquad (3)$$

where

E the apparent stiffness, GPa.

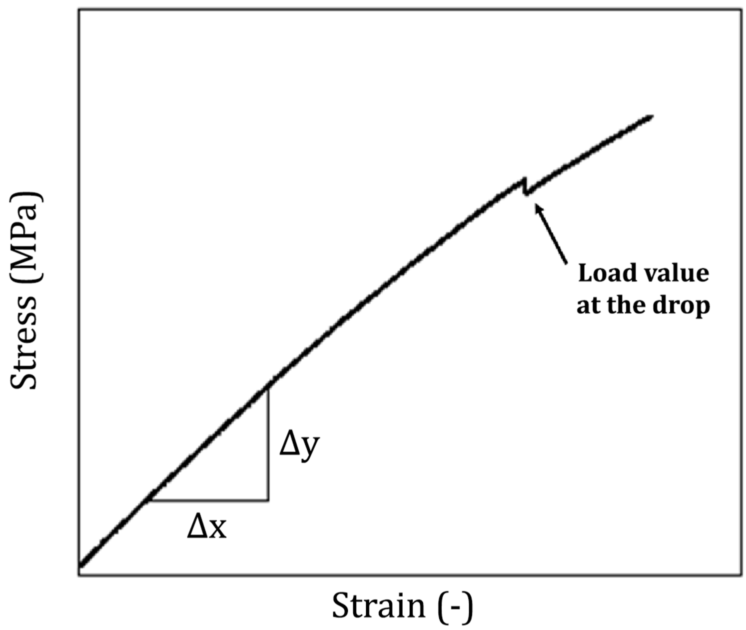

Figure 6 — Stress- Strain plot from Lap Strap test

8.4 Knockdown effect

If a modified composite material is compared with a reference, the knockdown effect in terms of strength is calculated using the following equation:

$$k\,\% = 1 - \frac{\sigma_{max}^{Modified}}{\sigma_{max}^{Reference}} \times 100\,\% \tag{4}$$

where

$\sigma_{max}^{Modified}$	lap strap strength of the modified specimen, MPa;
$\sigma_{max}^{Reference}$	lap strap strength of the reference specimen, MPa.

8.5 Repair efficiency

In case of a repairable/self-healing material, the repair efficiency in terms of strength is assessed using the following equation:

$$n\,\% = \frac{\sigma_{max}^{Repaired}}{\sigma_{max}^{Initial}} \times 100\,\% \tag{5}$$

where

$\sigma_{max}^{Repaired}$	lap strap strength of the modified specimen, MPa;
$\sigma_{max}^{Initial}$	lap strap strength of the reference specimen, MPa.

9 Test report

The text report shall contain at least the following information:

a) The test method.

b) The location and the date of the test.

c) The name of the test operator.

d) Any variations from this test method, problems during testing or equipment problems.

e) Description of the material tested: specification of materials, design, manufacturing process, curing cycle, consolidation method and the equipment used.

f) Ply orientation and stacking sequence of the laminate.

g) Results of the non-destructive evaluation test.

h) Measured dimensions for each specimen.

i) Conditioning parameters.

j) Humidity and temperature of the testing environment.

k) Number of specimens tested.

l) Speed of mechanical testing.

m) Data of force versus extension and the related curves.

n) Data of stress versus strain and the related curves.

o) The mean and the standard deviation of the calculated quantities.

p) The knockdown effect and the repair efficiency mean values (if any).

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