



**International  
Standard**

**ISO 8065**

**Composites and reinforcements  
fibres — Mechanoluminescent  
visualization method of crack  
propagation for joint evaluation**

*Composites et fibres de renforts — Méthode de visualisation  
mécanoluminescente de la propagation des fissures pour  
l'évaluation des assemblages*

**First edition  
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## Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

## Introduction

This document specifies a method for mechanoluminescent visualization of position of crack tip and crack propagation for joint evaluation of the bonded plates of carbon fibre reinforced plastics (CFRPs) to metal assemblies, with a standard specimen and under specified conditions of preparation, conditioning and testing. This method is intended for testing only those bonded plates used in bonding carbon fibre reinforced plastics (CFRPs) to metal assemblies.

The potential benefits to the users of mechanoluminescent visualization method of crack propagation for joint evaluation of the bonded plates of carbon fibre reinforced plastics (CFRPs) to metal assemblies based on this document are:

- a) providing precise position and amount of crack propagation, required for quantitatively calculating evaluation of the fracture toughness energy through double cantilever beam (DCB) test for opening mode.
- b) providing precise position and amount of crack propagation for end notched flexure (ENF) test and end-loaded split (ELS) test, which evaluate the fracture toughness (mode II) of crack propagation without opening against shear stress.
- c) providing visual information of crack propagation behaviour that occurs around adhesive bond layer inside the joint during the adhesive evaluation test, such as tensile shear test, cross tension, etc;
- d) for reliable evaluation, providing visual information of asymmetric behaviour of deformation in both adherends, originated from asymmetric flexural rigidity of both adherends, which occurs especially when joining dissimilar materials;
- e) for reliable evaluation to confirm health of adherend, providing of visual information of minor damage and fracture in one or both adherends, which causes scattering in the evaluation value of adhesive strength and performance, originated from asymmetric flexural rigidity of both adherends, especially in joining dissimilar materials;
- f) expanding CFRP applications to the fields of the combinations with metallic components;
- g) the detection or the prevention of physical properties loss - such as ion migration and time-related degradation in sealant film, injected calking layer and glass fibre reinforced plastics (GFRPs) layer;
- h) demonstrating the conformity to specified conditions for type certification requirements in the engineering such as aircraft developments;
- i) evaluating the procedures for maintenance, repair and overhaul (MRO) in the engineering operations such of CFRP aircrafts.

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# Composites and reinforcements fibres — Mechanoluminescent visualization method of crack propagation for joint evaluation

## 1 Scope

This document applies to the measurement of crack tip position and crack propagation on the bonding surface of carbon fibre reinforced plastic (CFRPs) and metal assemblies bonded panels.

This document does not apply to the visualization measurement of strain distribution or defects during load application to specimens.

This document does not intend to:

- a) omit relevant field tests for CFRP related engineering;
- b) generally specify the dimensions of test specimen to represent CFRPs related bonded or fastened structures;
- c) superimpose test results for specific applications of the parameters that exceed the range of this document.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 10365, *Adhesives — Designation of main failure patterns*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 10365 and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

### 3.1 mechanoluminescence

**ML**  
luminescence generated by mechanical stimulation

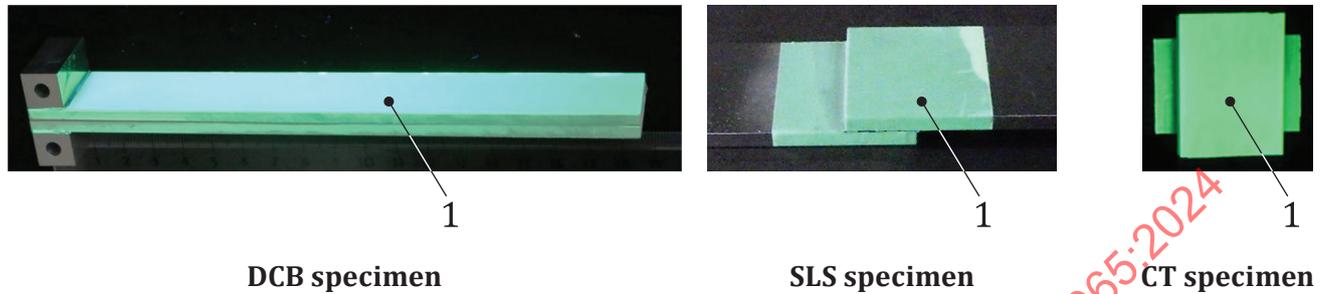
## 4 Principle

Mechanoluminescence (ML) intensity is proportional to the strain energy of measurement object. Therefore, at crack tip, strain is concentrated under loading, and intense mechanoluminescence can be generated reflecting the strain concentration at the crack tip to visualize the position of the crack tip.

## 5 Specimen preparation

### 5.1 Specimen

Various type of bonded plates can be used, such as double-cantilever beam (DCB) specimen, end-notch flexure (ENF) specimen, single lap shear (SLS), end-load split (ELS), double lap shear (DLS), cross tension (CT), etc. (see [Figure 1](#)).



#### Key

1 ML paint

**Figure 1 — Examples of test specimens for mechanoluminescence visualization of crack tip and crack propagation such as DCB, SLS and CT test**

### 5.2 Surface pre-treatment

Surface pre-treatment can be used to make fine adhesion between mechanoluminescence (ML) paint and surface of specimen, but it is not mandatory. As the method of surface pre-treatment, solvent degreasing, sanding, atmospheric pressure plasma treatment, etc. can be used. However, a method that does not affect the adhesive bonding performance and the strength of the adherend should be selected.

### 5.3 Preparation for mechanoluminescence paint

Mechanoluminescence (ML) paint is prepared by mixing ML material and polymer resin. It is possible to use any ML materials and polymer materials. Candidate ML material is shown in [Annex A](#).

### 5.4 Method of applying mechanoluminescence paint

Mechanoluminescence (ML) paint can be apply on the surface of specimen by spraying, dipping, or brushing, etc. It is preferable that the film thickness is uniform, however, even if it is not uniform, it does not affect the crack tip monitoring mechanoluminescence.

NOTE Conditions for applying mechanoluminescence paint are shown in [Annex B](#) ([B.1](#) for DCB test, [B.2](#) for SLS test, [B.3](#) for CT test). Effect on quality of mechanoluminescent paint coating is described for monitoring performance of crack tip in [Annex C](#).

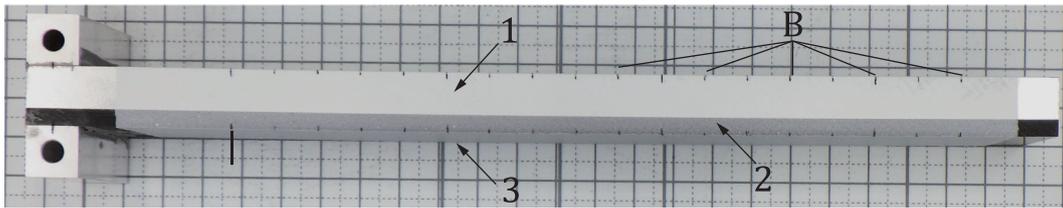
### 5.5 Post-treatment

As a post-treatment, in order to cure the mechanoluminescent (ML) paint, thermosetting, photocuring, room temperature curing, etc. can be performed depending on the polymer material of the ML paint. However, condition of the post-treatment should be selected within a range of conditions that does not affect the test piece and bonding performance.

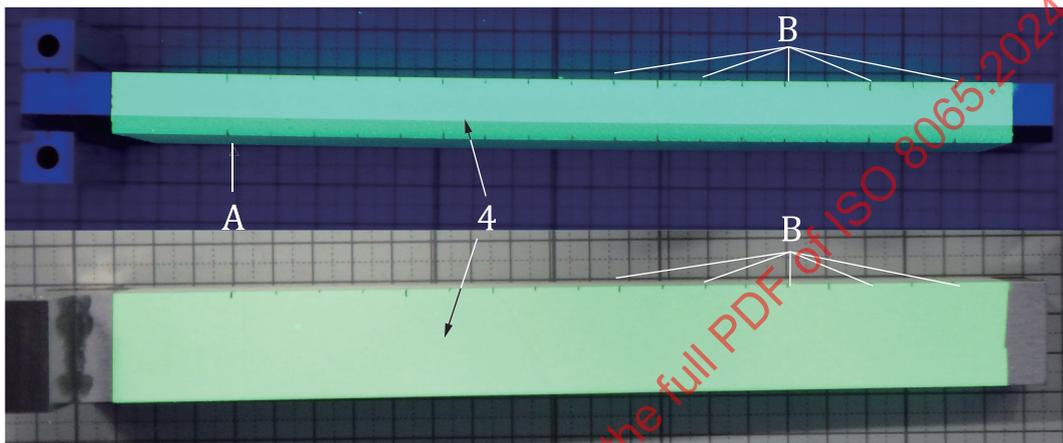
### 5.6 Scale labelling on specimen

In case it is necessary to determine the length of crack propagation from initial, for example for calculating fracture energy and toughness in DCB test, ENF and c-ELS test, scale labelling from initial crack should be

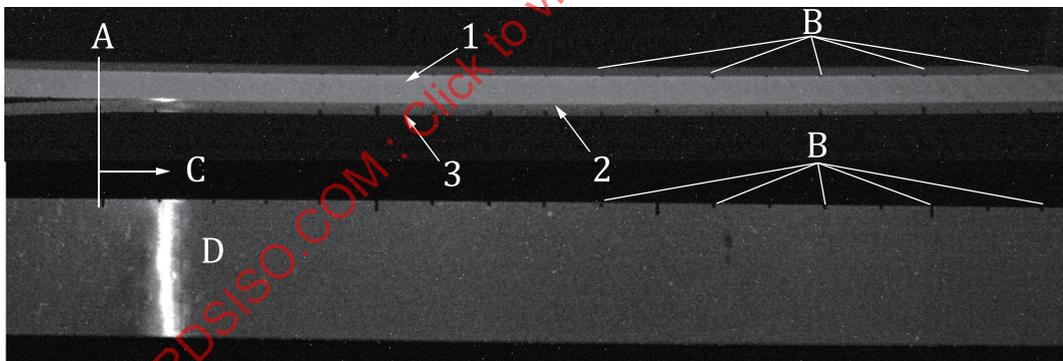
marked with black pen at least every 10 mm (see [Figure 2](#)). The scale labelling should be beneficial not only at only one side but also other surface. For example, the scale inside surface can be used to determine crack length and the ones on top and bottom surface should be beneficial to identify the position of mechanoluminescent (ML) line reflecting fracture front in bond line.



a) Photo in sideview



b) Side and top views under UV light



c) ML image

**Key**

- |   |                |   |                                |
|---|----------------|---|--------------------------------|
| 1 | metal plate    | A | position of initial crack      |
| 2 | adhesive layer | B | scale labelling                |
| 3 | CFRTP plate    | C | direction of crack propagation |
| 4 | ML paint       | D | ML line                        |

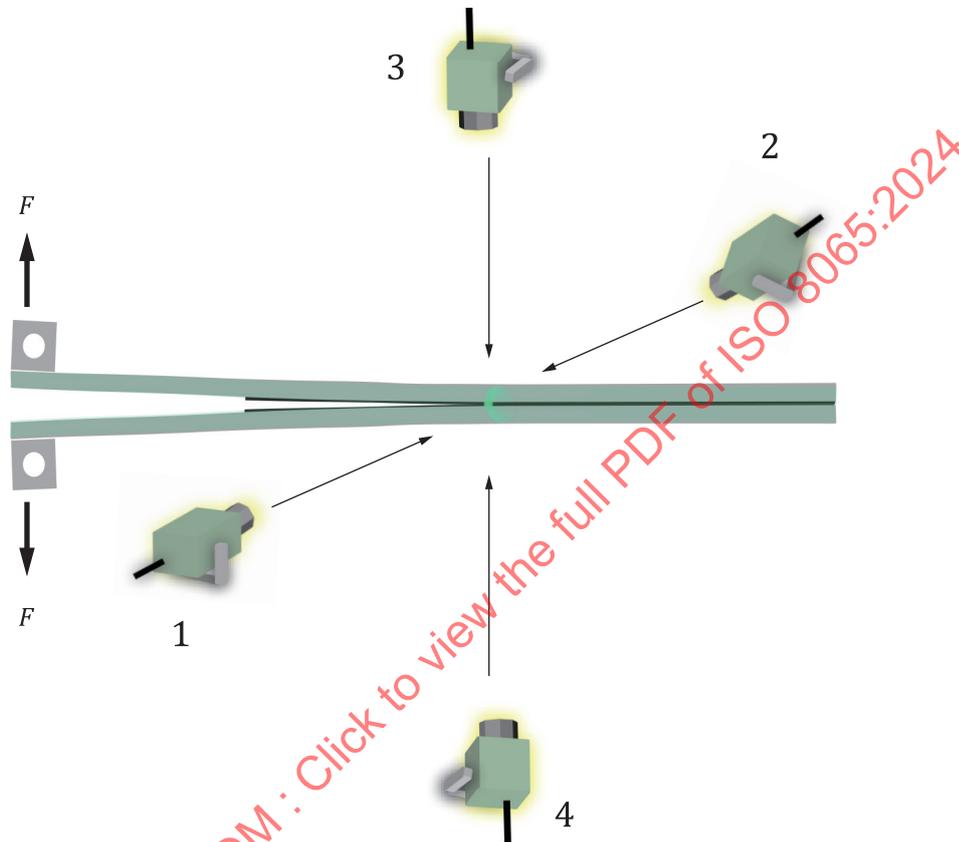
**Figure 2 — Example of scale labelling for DCB testing**

## 6 Test equipment and testing procedure

### 6.1 Measurement equipment for mechanoluminescence

A four-way camera setup of the test piece is recommended (see [Figure 3](#)), but at least the tip of the crack to be monitored should be set up with the camera facing the position where it can be recorded.

NOTE 4-way camera system is not mandatory. Number of cameras depends on which face of specimen you want to focus and record.



#### Key

- 1 camera 1
- 2 camera 2
- 3 camera 3
- 4 camera 4
- F load

**Figure 3** — Example of equipment for mechanoluminescence visualization of crack tip and crack propagation

### 6.2 Test conditions

Mechanical conditions of testing bonded samples should follow the respective joining test standards:

- ISO 22838
- ISO 22841

### 6.3 Recording conditions of mechanoluminescence

Recording rate, frame-per-second (fps), should be determined in consideration of the crack growth rate in each adhesion evaluation test. In addition, frame-per-second (fps), exposure time, gain, etc. are determined in consideration of the characteristics of the applied mechanoluminescent material. Specifically, the conditions can be set, in which mechanoluminescence should be recorded during crack growth from the initial crack to the end of the testing.

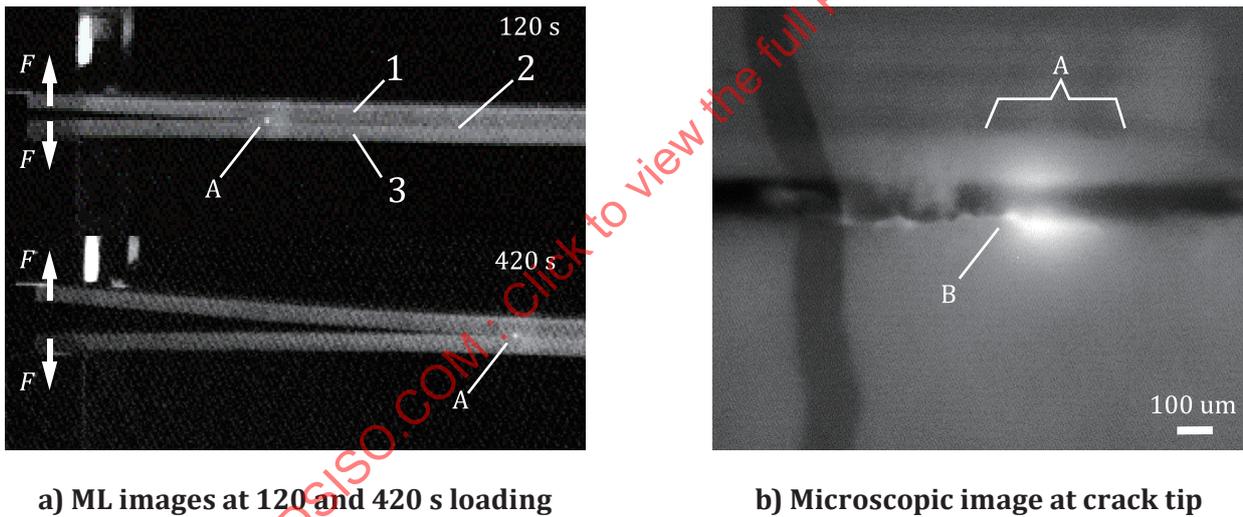
NOTE Examples of recording conditions responding to each testing are shown in [Annex B](#) (B.1 for DCB test, B.2 for SLS test, B.3 for CT test).

## 7 Data analysis

### 7.1 Position of crack tip

Position of crack tip during testing can be determined as a point with highest mechanoluminescent (ML) luminance in adhesive layer, by reading the position of the rearmost edge (see [Figure 4](#)). The ML contour image converted from raw image should be easier to identifying highest ML point reflecting position of crack tip (see [Figure 5](#) and [Annex D](#)). From this information of crack tip position, the crack length can be calculated and used to evaluate the adhesive fracture toughness energy in DCB test, ENF test, c-ELS test, etc.

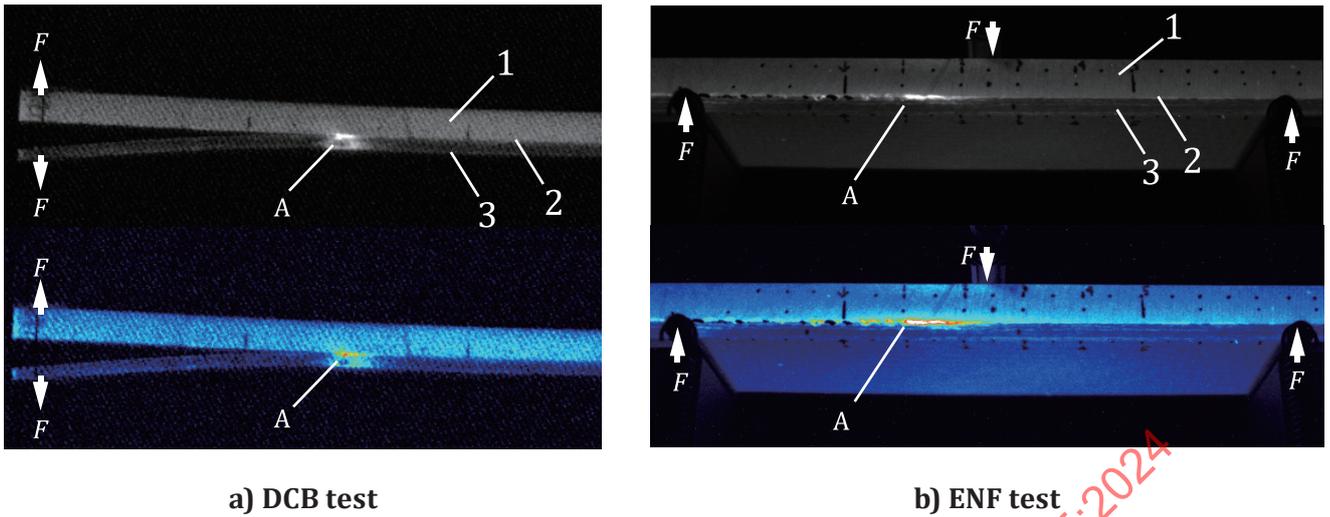
In addition, by visualizing the crack tip by mechanoluminescence, it is possible to identify the fracture origin point and propagation of the adhesive bond during joint evaluation (see [Figure 6](#)).



#### Key

- 1 metal plate
- 2 adhesive layer
- 3 metal plate
- A ML points
- B position of crack tip
- F load

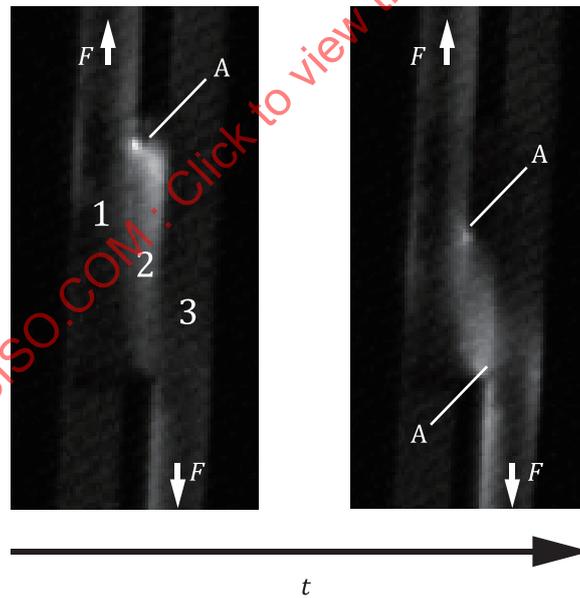
**Figure 4 — Example of crack tip monitoring using mechanoluminescence (DCB test)**



**Key**

- 1 metal plate
- 2 adhesive layer
- 3 CFRTP plate
- A ML point reflecting position of crack tip
- F load

**Figure 5 — Mechanoluminescent (ML) image in raw and contour map in luminance**



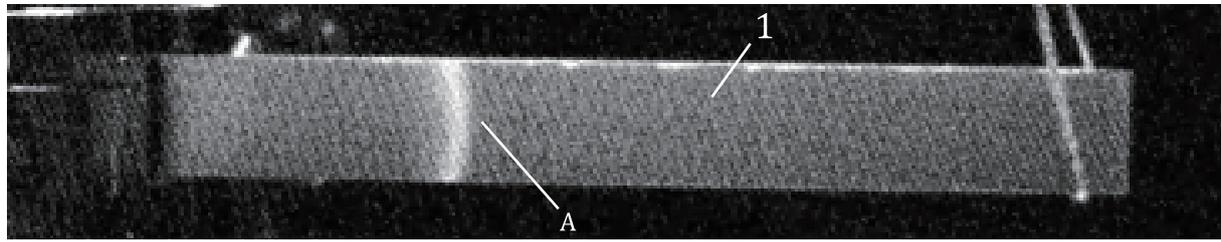
**Key**

- 1 metal plate
- 2 adhesive layer
- 3 metal plate
- A ML points reflecting crack tip
- F load
- t time during SLS test

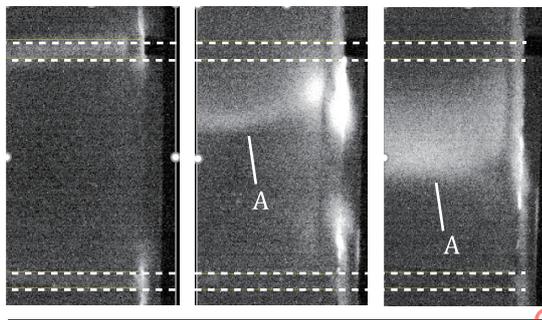
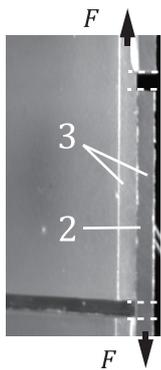
**Figure 6 — Example of identifying of fracture origin using mechanoluminescence (SLS test)**

## 7.2 Delamination analysis

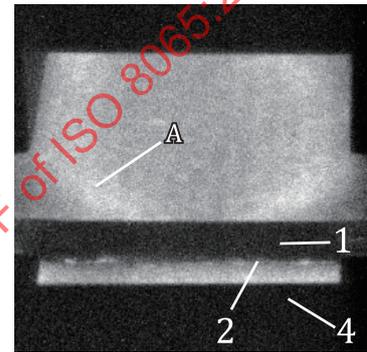
During delamination, adhesive failure front in bond line can be identified by using mechanoluminescence (ML) line on the outside surface of the adherend (see [Figure 7](#)). The validity of ML line is additionally explained to reflect adhesive failure front in bond line in [Annex E](#).



a) DCB test



b) SLS test



c) CT test

### Key

- 1 metal plate
- 2 adhesive layer
- 3 CF RTP plate
- 4 plastic plate
- A ML line reflect adhesive failure front in bond line
- F load

Figure 7 — Example of visualising of fracture process using mechanoluminescence

## 8 Test report

The test report shall contain at least the following information:

- a) a reference to this document, i.e. ISO 8065:2024;
- b) the bonding specimen employed;
- c) the pre-treatment and post treatment employed;
- d) the mechanoluminescent (ML) materials, polymer material employed;
- e) the camera used;
- f) the recording condition of mechanoluminescence;
- g) the conditions of mechanical testing;

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- h) mechanoluminescence (ML) image;
- i) special remarks, if any.

NOTE Examples of usability and round robin test are described in [Annexes F](#) and [G](#).

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## Annex A (informative)

### Mechanoluminescence (ML) material

#### A.1 Examples of commercially available mechanoluminescent (ML) material

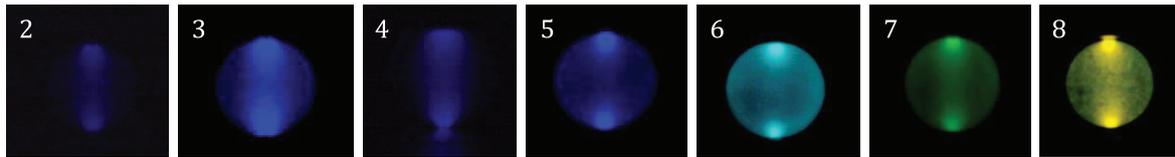
Examples of commercially available mechanoluminescent (ML) material are shown in [Table A.1](#).

#### A.2 Mechanoluminescent (ML) material, shown in academic papers

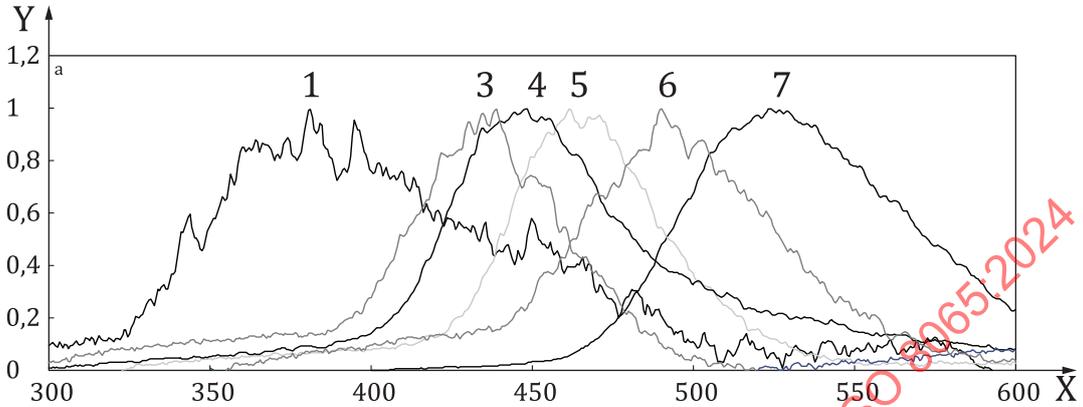
Examples of mechanoluminescent (ML) material, shown in academic papers are listed below and shown in [Figure A.1](#):

- $\text{SrAl}_2\text{O}_4:\text{Eu}$  (green, 510 nm),
- $\text{SrAl}_2\text{O}_4:\text{EuMg}$ ,  $\text{SrAl}_2\text{O}_4:\text{EuCa}$ ,
- $\text{SrAl}_2\text{O}_4:\text{EuHo}$ ,  $\text{SrAl}_2\text{O}_4:\text{EuNd}$  (green, red and near infra-red, 510 nm, 690, 850, 1 100 nm),
- $\text{SrAl}_2\text{O}_4:\text{EuCe}$ ,  $\text{SrAl}_2\text{O}_4:\text{HoCe}$ ,  $\text{SrAl}_2\text{O}_4:\text{EuNdCr}$ ,
- $\text{CaGdAl}_3\text{O}_7:\text{Eu}$  (blue, 437 nm),
- $\text{CaYAl}_3\text{O}_7:\text{Eu}$  (blue, 437 nm),
- $\text{CaLaAl}_3\text{O}_7:\text{Eu}$  (blue, 440 nm),
- $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}$  (blue, 460 nm),
- $\text{SrCaMgSi}_2\text{O}_7:\text{Eu}$  (Greenish Blue, 489 nm),
- $\text{Ca}_2\text{MgSi}_2\text{O}_7:\text{EuDy}$  (Green, 524 nm),
- $\text{Sr}_2\text{SiO}_4:\text{Eu}$ , Dy (Yellow, 540 nm),
- $\text{Sr}_2\text{Si}_2\text{O}_7:\text{EuDy}$ .

$\text{ZnS}:\text{Cu}$ ,  $\text{ZnS}:\text{Cu,Mn}$ ,  $\text{ZnS}:\text{Cu}$ ,  $\text{ZnS}:\text{Cu}$ ,  $\text{ZnS}:\text{Al,Cu}$ ,  $\text{ZnS}:\text{Mn}$ , in [Figure A.1](#) and organic ML materials in [Figure A.2](#).



a) Pictures during ML emission of compression load to cylindrical pellet

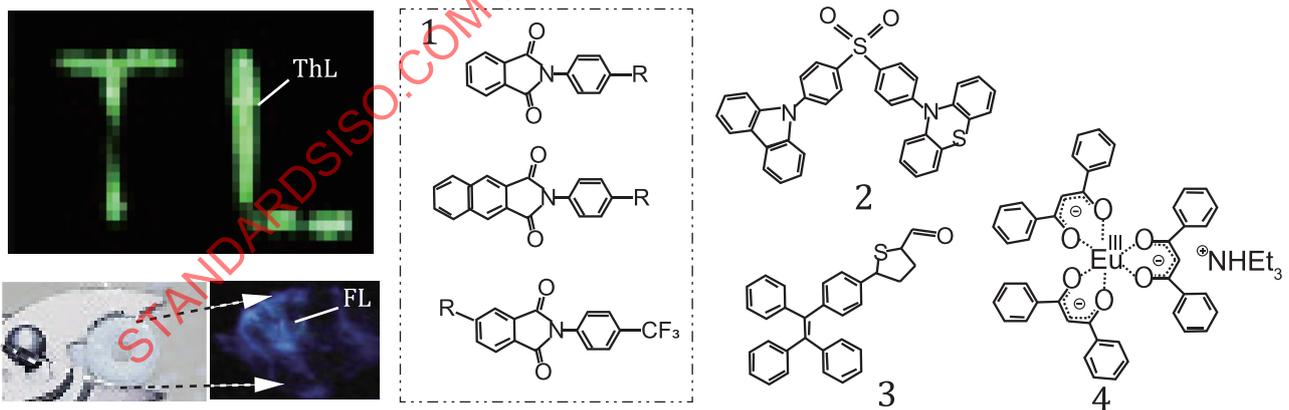


a) ML spectra

**Key**

X	Wavelength (nm)	5	$\text{Sr}_2\text{MgSi}_2\text{O}_7\text{:Eu}$ (blue, 460 nm)
Y	normalized intensity (arb.u.)	6	$\text{SrCaMgSi}_2\text{O}_7\text{:Eu, Dy}$ (Greenish Blue, 489 nm)
1	$\text{SrAl}_2\text{O}_4\text{:HoCe}$ (purple, 375 nm)	7	$\text{Ca}_7\text{MgSi}_2\text{O}_7\text{:Eu, Dy}$ (Green, 524 nm)
2	$\text{CaGdAl}_3\text{O}_7\text{:Eu}$ (blue, 437 nm)	8	$\text{Sr}_2\text{SiO}_4\text{:Eu, Dy}$ (Yellow, 540 nm)
3	$\text{CaYAl}_3\text{O}_7\text{:Eu}$ (blue, 438 nm)	a	ML spectra
4	$\text{CaLaAl}_3\text{O}_7\text{:Eu}$ (blue, 440 nm)		

Figure A.1 — Examples of inorganic ML materials reported in papers<sup>[4]</sup>



**Key**

1	phenoiazin derivatives	ThL	Thribo luminescence
2	SPFC or derivatives	FL	Fracture-luminescent
3	$\text{P}_4\text{TF}$ or derivatives		
4	$\text{EuD}_4\text{TEA}$ or derivatives		

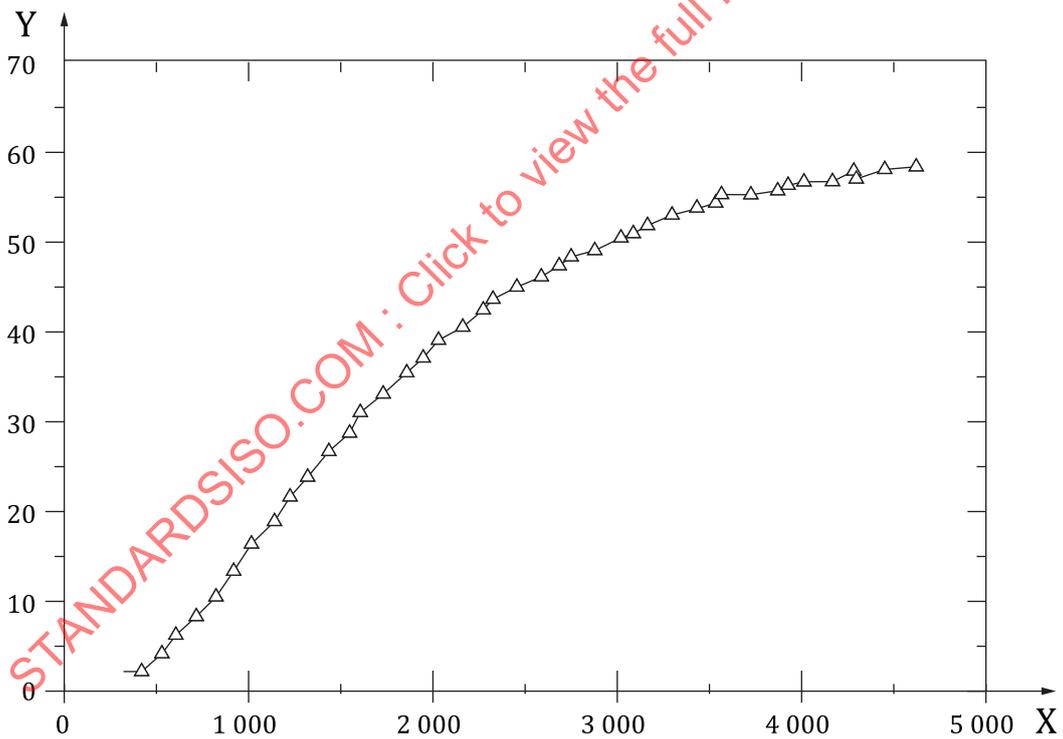
Figure A.2 — Examples of organic ML materials reported in papers<sup>[5]</sup>

### A.3 Commercially available mechanoluminescent (ML) paint

Any polymer material can be used for ML paint, but performance depends on the young's modulus of the polymer. On the market, Sakai Chemical<sup>1)</sup> (Japan) sells epoxy-based and urethane-based mechanoluminescent (ML) paint as products (see [Figure A.3](#)). Water-based paint can also be used when preparing ML paint. Although the ML performance has been lower than the organic solvent -based ML paint, it can be used in consideration of the safety regulations of each country (see [Figure A.4](#)).



Figure A.3 — Commercially available ML paint and spray



**Key**

- X luminance [mcd/mc<sup>2</sup>]
- Y Strain [ust]

Figure A.4 — Performance of water-based ML paint (not commercially available)

1) This information is given for the convenience of users of this document and does not constitute an endorsement by ISO.

Table A.1 — Examples of commercially available mechanoluminescent (ML) material

Company	User				
	USA	China	Korea	Eu	Japan
<b>ZnS series</b>					
Osram Sylvania Inc. (Product#: GG45) ZnS:Cu	✓		✓		
Osram Sylvania Inc. (Product#: GGS12) ZnS:Cu,Mn			✓		
LONCO Company Limited ZnS:Cu			✓		
Global Tungsten & Powders Co. ZnS:Cu, ZnS:Al,Cu		✓	✓		
Phosphor Technology Ltd (Lot 19252) ZnS:Mn	✓			✓	
<b>SAO series</b>	<b>USA</b>	<b>China</b>	<b>Korea</b>	<b>Eu</b>	<b>Japan</b>
Nemoto & Co., Ltd., Nemoto Portugal, United Mineral and Chemical Co. (LumiNova G300M, High ML performance version: MLG-1000)	✓		✓	✓	✓
Shenzhen Yaodexing Technology Ltd. (KYD-7) SAOED		✓			
Taiko Refractories Co., Ltd., (TAIKO-ML-1)					✓
Sakai Chemical Industry Co., Ltd., (ML032, etc.)					✓
NOTE This information is given for the convenience of users of this document and does not constitute an endorsement by ISO.					

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## Annex B (informative)

### Example of conditions for ML measurements

#### B.1 Example of conditions in DCB test

In order to assist in use of mechanoluminescence (ML) visualization of crack position and propagation for joint evaluation, concrete example of conditions such as sample preparation, measurement and results are described in the form of [Clause 8](#) and shown in [Figure B.1](#). In detail, see, Reference [\[6\]](#).

- a) a reference to the International Standard used, i.e. ISO 22838, ISO 15024;
- b) the bonding specimen employed:  
DCB specimen.  
Adherend: sandblasted aluminium (A5052).  
Adhesive: structural epoxy adhesive (Denatite 2204<sup>2)</sup>, curing temperature of adhesive: 100 °C; thickness: ~100 µm)
- c) the pre-treatment and post treatment employed;  
Pre-treatment: Solvent degreasing using iso-propyl alcohol (IPA).  
Post-treatment: curing temperature of ML paint: 100 °C, 30 min
- d) the mechanoluminescent (ML) materials, polymer material employed;  
Commercial ML paint (ML-32ET<sup>3)</sup>) shown in A-3, consisting of SrAl<sub>2</sub>O<sub>4</sub>:Eu<sup>2+</sup> ML material and epoxy resin, dispersed in organic solvent.  
Applying method: air spraying.
- e) the camera used;  
Charge coupled device (CCD) camera (TXG04 or VLU-12<sup>4)</sup>  
Lens: LM8HC<sup>5</sup> (f1.4, Kowa, Japan)
- f) the recording condition of mechanoluminescence;  
Dark condition.  
Before loading, blue light at the wavelength of 470 nm was irradiated to the ML paint covered sample for 1 min and waiting in dark condition for 1 min, for quantitative and reproducible ML result using SrAl<sub>2</sub>O<sub>4</sub>:Eu<sup>2+</sup> type ML material (λ<sub>em</sub> = 520 nm).

2) Denatite 2204 is the trade name of a product supplied by Nagase ChemteX Co. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO or of the product named. Equivalent products may be used if they can be shown to lead to the same results.

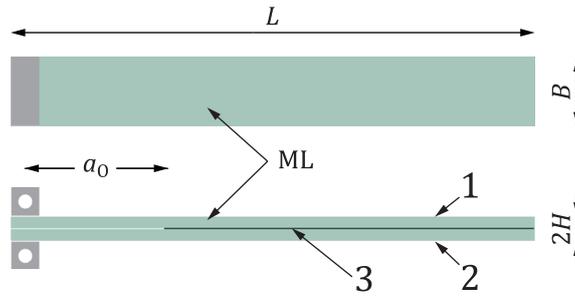
3) ML-32ET is the trade name of a product supplied by Sakai Chemical, Japan. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO or of the product named. Equivalent products may be used if they can be shown to lead to the same results.

4) TXG04 or VLU are the trade names of products supplied by Baumer, Germany. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO or of the product named. Equivalent products may be used if they can be shown to lead to the same results.

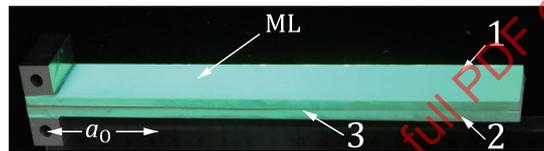
Recording rate: 1 frame-per second (fps). Exposure time: 1 s. Gain: maximum.

- g) the conditions of mechanical testing;  
Tensile load: 1 min/mm.
- h) mechanoluminescence (ML) image; shown in [Figure B.1](#)
- i) special remarks, if any.

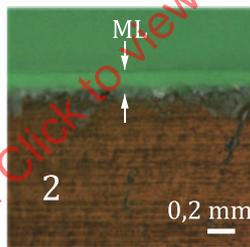
Dimensions in millimetres



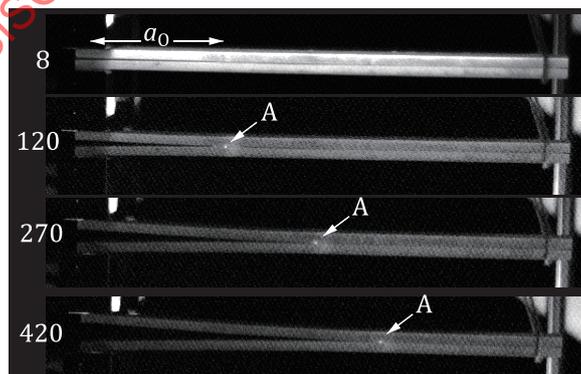
a)



b)



c)



d)

**Key**

1	metal plate (A5052)	A	ML point, reflecting position of crack tip
2	metal plate (A5052)	ML	ML paint
3	adhesive layer	L	length of specimen
		B	width of specimen
		H	thickness of adherend
		$a_0$	Length of initial crack.

**Figure B.1 — Example synchronising ML line on adherend and failure from in bond line**

**B.2 Example of conditions — SLS test**

In order to assist in use of mechanoluminescence (ML) visualization of crack position and propagation for joint evaluation, concrete example of conditions such as sample preparation, measurement and results are described in the form of [Clause 8](#) and shown in [Figure B.2](#). In detail, see Reference [8].

- a) a reference to this International Standard, i.e. ISO 4587, ISO22841;
- b) the bonding specimen employed;
  - LS specimen.
  - Adherend: sandblasted aluminium (A5052).
  - Adhesive: structural epoxy adhesive (Denatite 2204, Nagase ChemteX Co., curing temperature of adhesive: 100 °C; thickness: ~100 µm)
- c) the pre-treatment and post treatment employed;
  - Pre-treatment: Solvent degreasing using iso-propyl alcohol (IPA), sand blast.
  - Post-treatment: curing temperature of ML paint: 100 °C, 30 min
- d) the mechanoluminescent (ML) materials, polymer material employed;
  - Commercial ML paint (ML-32E1, Sakai Chemical, Japan) shown in A-3, consisting of  $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}$  ML material and epoxy resin, dispersed in organic solvent.
  - Applying method: air spraying.
- e) the camera used;
  - Charge coupled device (CCD) camera (TXG04 or VLU-12, Baumer, Germany)
  - Lens: LM8HC (f1.4, Kowa, Japan)
- f) the recording condition of mechanoluminescence;
  - Dark condition.
  - Before loading, blue light at the wavelength of 470 nm was irradiated to the ML paint covered sample for 1 minute and waiting in dark condition for 1 min, for quantitative and reproducible ML result using  $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}$  type ML material ( $\lambda_{em} = 520 \text{ nm}$ ).
  - Recording rate: 25 frame-per second (fps). Exposure time: 40 ms. Gain: maximum.
  - (Recording condition in 10-30 fps can be used in the next)

- g) the conditions of mechanical testing;  
Tensile load: 5 min/mm.
- h) mechanoluminescence (ML) image; shown in [Figure B.2](#)
- i) special remarks, if any.

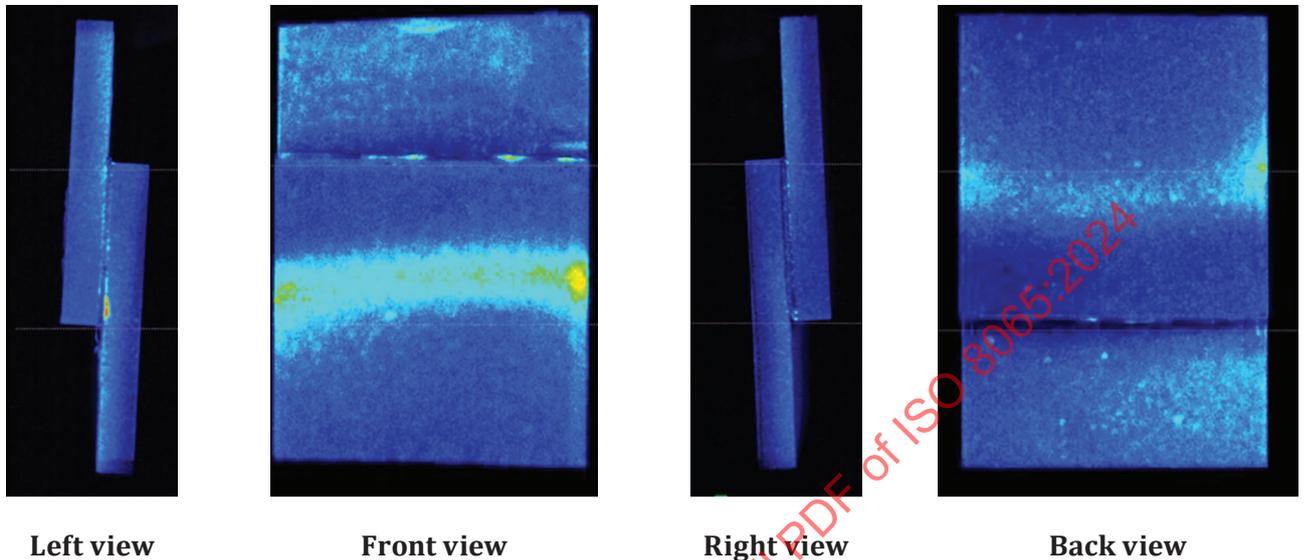


Figure B.2 — Example of ML monitoring for tensile single lap-shear (SLS) test.

### B.3 Example of conditions: Cross tension (CT) test

In order to assist in use of mechanoluminescence (ML) visualization of crack position and propagation for joint evaluation, concrete example of conditions such as sample preparation, measurement and results are described in the form of “8. Test report” as below and shown in [Figure B.3](#). In detail, see Reference [8].

- a) a reference to the International Standard used, i.e. ISO 4587, ISO22841;
- b) the bonding specimen employed;

Ct specimen.

Adherend: sandblasted aluminium (A5052) and carbon fiber-reinforced thermoplastic (CFRTP, 50 mm × 25 mm × 4 mm, twill woven, ICHIMURA SANGYO CO., LTD) substrate.

Adhesive: structural epoxy adhesive (Denatite 2204, Nagase ChemteX Co., curing temperature of adhesive: 100 °C; thickness: ~100 µm)

- c) the pre-treatment and post treatment employed;

Pre-treatment:

1. Solvent degreasing using iso-propyl alcohol (IPA),
2. Flame treatment for CFRTP,
3. Aqueous etching acid to prepare a nanoporous surface for strong bonding (so-called nano-molding technology (NMT) for Al substrate.

Post-treatment: curing temperature pf ML paint: 100 °C, 30 min

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- d) the mechanoluminescent (ML) materials, polymer material employed;

Commercial ML paint (ML-32ET, Sakai Chemical, Japan) shown in A-3, consisting of  $\text{SrAl}_2\text{O}_4\cdot\text{Eu}^{2+}$  ML material and epoxy resin, dispersed in organic solvent.

Applying method: air spraying, thickness = 50  $\mu\text{m}$

- e) the camera used;

Charge coupled device (CCD) camera (TXG04 or VLU-12, Baumer, Germany)

Lens: LM8HC (f1.4, Kowa, Japan)

- f) the recording condition of mechanoluminescence;

Dark condition.

Before loading, blue light at the wavelength of 470 nm was irradiated to the ML paint covered sample for 1 minute and waiting in dark condition for 1 minute, for quantitative and reproducible ML result using  $\text{SrAl}_2\text{O}_4\cdot\text{Eu}^{2+}$  type ML material ( $\lambda_{\text{em}} = 520 \text{ nm}$ ).

Recording rate: 1 frame-per second (fps). Exposure time: 1 s. Gain: maximum

(Recording condition in 1-2 fps was appropriate with the conditions above.)

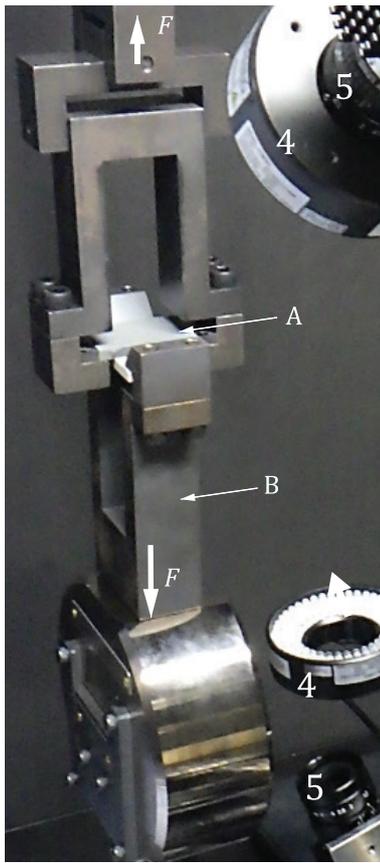
- g) the conditions of mechanical testing;

Tensile load: 1 min/mm.

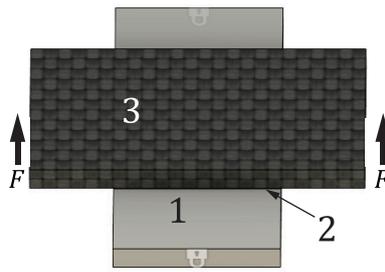
- h) mechanoluminescence (ML) image; shown in [Figure B.3](#)

- i) special remarks, if any.

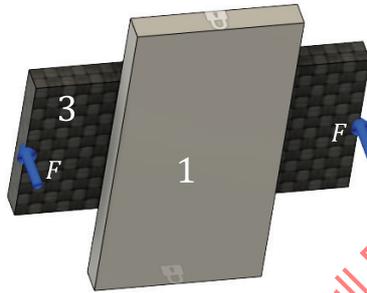
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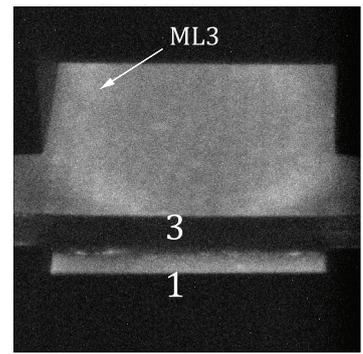
a)



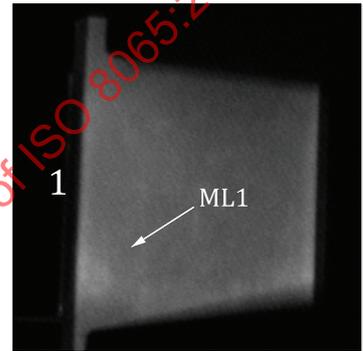
b)



c)



d)



e)

**Key**

- |   |                |     |   |
|---|----------------|-----|---|
| 1 | metal plate    | A   | ML paint sprayed cross tension specimen           |
| 2 | adhesive layer | B   | Zig for cross tension test, described in ISO24360 |
| 3 | CFRTP plate    | F   | load  |
| 4 | LED blue light | ML1 | ML line on metal plate (1)                        |
| 5 | CCD camera     | ML3 | ML line on CFRTP plate (3)                        |

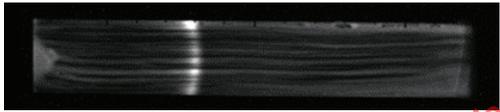
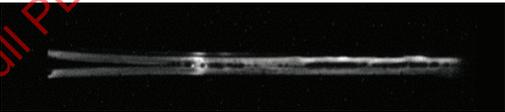
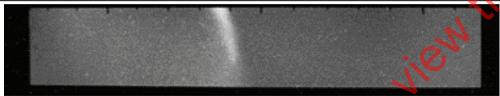
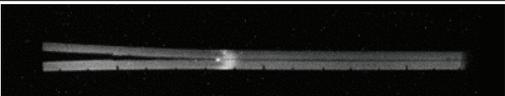
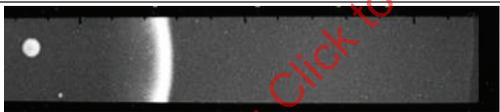
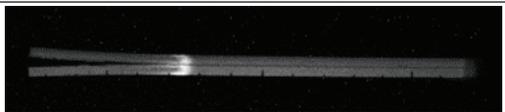
**Figure B.3 — Example of ML monitoring for cross tension (CT) test**

## Annex C (informative)

### Demonstration: Effect on quality of mechanoluminescent paint coating for monitoring performance of crack tip

In order to clarify the requirement of mechanoluminescence (ML) paint coating, DCB tests were carried out with various coating qualities and painting methods. By using brush for paint, high roughness can be recognized (case 1). On the other hand, by using air spray, comparably fine coating film was prepared on the substrate surface (case 2 and 3), even in the case of roughly paint (case 2).

In general, low quality ML coating spoils the quality of result for visualising dynamic strain distribution through ML pattern. However, when focusing on monitoring of position of crack tip and the propagation using mechanoluminescence, ML lines and points can be clearly distinguished at the top and side view respectively in every case no matter what quality of ML paint coating were employed (see [Figure C.1](#)). This shows that fine quality is recommended for ML paint coating, but it is not mandatory for monitoring of crack propagation.

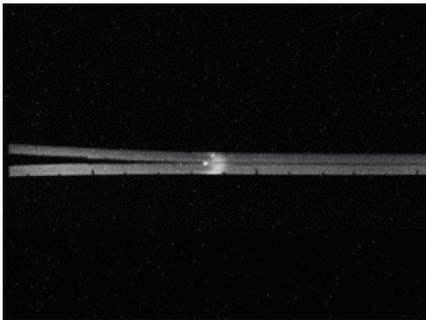
	Top view	Side view
<b>1. Paint by brush</b>		
<b>2. Rough paint by air spray</b>		
<b>3. Careful paint by air spray</b>		

**Figure C.1 — Effect on quality of mechanoluminescent paint coating for monitoring performance of crack tip**

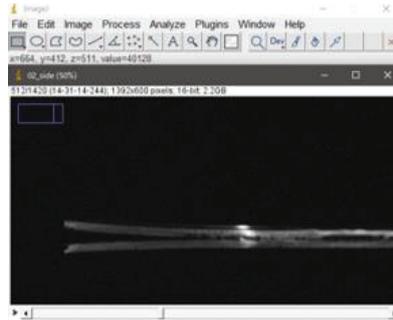
## Annex D (informative)

### Examples of determination methods of the highest ML point

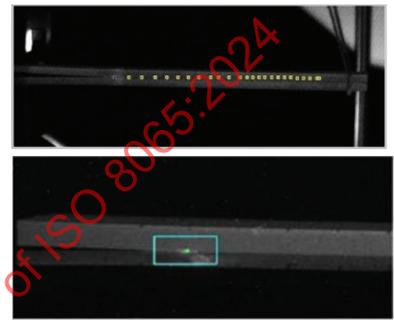
The position with highest ML is decided through visual inspection from ML movie [Figure D.1 a\)](#), and image processing software [for example image processing software in [Figure D.1 b\)](#), and auto monitoring system in [Figure D.1 c\)](#) can be used].



a) Visual inspection from ML movie



b) Image processing software



c) Auto monitoring system

Figure D.1 — Synchronising ML line on adherend and failure from in bond line

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## Annex E (informative)

### Demonstration — Synchronising ML line on adherend and failure front in bond line

The validity of ML line on the surface of adherend is explained to reflect adhesive failure front in bond line (see [Figure E.1](#)). In this case, flame pre-treated transparent polycarbonate plate (thickness: 3 mm) was used as the adherends and they bonded together using epoxy adhesive (Denatite 2204) to be DCB specimen. ML paint was applied using air spray at only top and side surfaces, and bottom surface was remained clear for observation of failure front in bond-line. [Figure C.1](#) shows a comparison of the ML behaviour from the top view (CCD 1) and the crack front line from the bottom view (CCD 2) during the DCB test. In brief, a straight ML line gradually appeared in a perpendicular orientation to the adhesive frontline at the position of the initial crack (143 s), and started moving ahead from the central part of the line (157 s), and reached a curved arc shape during a steady state of crack propagation (within 161 s to 605 s). Interestingly, from a comparison of the ML and bright images at each recording time, it was clearly confirmed that the crack failure frontline in the bright image from bottom view was almost the same as those of the ML line from the top view in position and shape. This result can be explained that the ML line is originated from the strain concentration owing to a bending deformation of the adherend accompanied by the crack opening at the failure front, and the ML line reflects it well. In detail, see Reference [7].

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