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Edition 1.0 2019-04

# INTERNATIONAL STANDARD





## Semiconductor devices – Micro-electromechanical devices – Part 31: Four-point bending test method for interfacial adhesion energy of layered MEMS materials

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# SEMICONDUCTOR DEVICES – MICRO-ELECTROMECHANICAL DEVICES –

## Part 31: Four-point bending test method for interfacial adhesion energy of layered MEMS materials

## FOREWORD



International Standard IEC 62047-31 has been prepared by subcommittee 47F: Micro-electromechanical systems, of IEC technical committee 47: Semiconductor devices.

The text of this International Standard is based on the following documents:

| FDIS | Report on voting |
|---|---|
| 47F/326/FDIS | 47F/331RVD |

Full information on the voting for the approval of this International Standard can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts in the IEC 62047 series, published under the general title *Semiconductor devices – Micro-electromechanical devices*, can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under "http://webstore.iec.ch" in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

**IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.**

# SEMICONDUCTOR DEVICES – MICRO-ELECTROMECHANICAL DEVICES –

# Part 31: Four-point bending test method for interfacial adhesion energy of layered MEMS materials

## 1 Scope

This part of IEC 62047 specifies a four-point bending test method for measuring interfacial adhesion energy of the weakest interface in the layered micro-electromechanical systems (MEMS) based on the concept of fracture mechanics. In a variety of MEMS devices, there are many layered material interfaces, and their adhesion energies are critical to the reliability of the MEMS devices. The four-point bending test utilizes a pure bending moment applied to a test piece of layered MEMS device, and the interfacial adhesion energy is measured from the critical bending moment for the steady state cracking in the weakest interface. This test method applies to MEMS devices with thin film layers deposited on semiconductor substrates. The total thickness of the thin film layers should be 100 times less than the thickness of a supporting substrate (typically a silicon wafer piece).

## 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.

There are no normative references in this document.

## 3 Terms, definitions, symbols and designations

### 3.1 Terms and definitions

For the purposes of this document, the following terms and definitions 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**
**energy release rate**
$G$
strain energy per unit surface area, which is released during the incremental growth of a crack

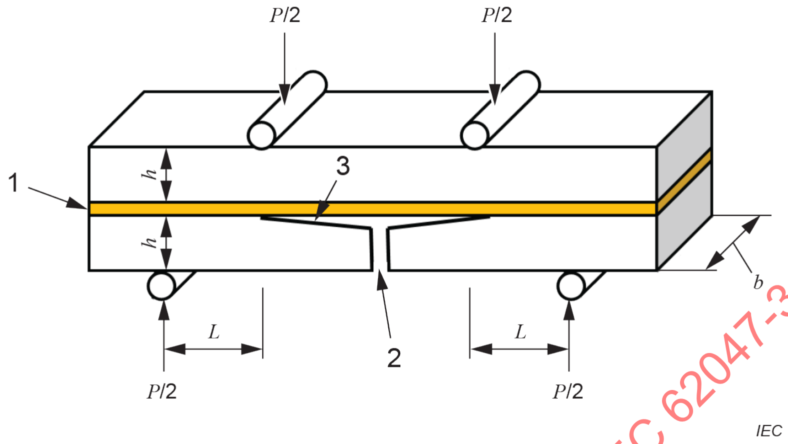
Note 1 to entry: The energy release rate can be regarded as the crack driving force, and its unit is given in J/m$^2$.

**3.1.2**
**interfacial adhesion energy**
$G_C$
critical energy release rate at the moment of crack extension

Note 1 to entry: Its unit is given in: J/m$^2$.

### 3.2 Symbols and designations

The shape of the test piece and the symbols are presented in Figure 1 and Table 1, respectively. The overall shape of the test piece is similar to a sandwiched cantilever beam, and it should have a pre-crack or a notch for crack initiation. After initiation of the crack, the crack follows the weakest interface in the layered materials system.




**Key**

1 layered material

2 notched pre-crack

3 interfacial crack

**Figure 1 – Four-point bending test piece**



**Table 1 – Symbols and designations of a test piece**

| Symbol | Unit | Designation |
|---|---|---|
| $L$ | mm | Spacing between two adjacent pins |
| $b$ | mm | Width |
| $h$ | µm | Thickness of the supporting structure |
| $P$ | N | Force for driving crack |

## 4 Test piece

### 4.1 General

The test piece for the layered MEMS materials shall be prepared using the same fabrication process that applies to actual MEMS devices. Machining of the test piece shall be performed to prevent formation of unintended cracks or flaws and delamination in the test piece.

### 4.2 Shape of a test piece

The overall shape of a test piece is shown in Figure 1. Because the evaluation of the energy release rate relies on several simplifying assumptions, the geometric shape of the test piece should be designed as follows: the thickness of the test piece should be 50 times less than the length and width of the test piece, and the length should be 10 times larger than the width. The total thickness of the layered materials should be 100 times less than the thickness of a supporting substrate. A pre-crack or notch shown in Figure 1 is machined using conventional ways like a diamond saw, laser ablation, or chemical etching. This pre-crack initiates cracking in the supporting substrate under bending, and after that the cracking leads to the introduction of an interface crack between two adjacent layers of the weakest interface in the layered materials.

### 4.3 Measurement of dimensions

To analyze the test results, the test piece dimensions shall be accurately measured because the dimensions are used to determine the mechanical properties of test materials. Spacing between the pins ($L$), width ($b$), and thickness ($h$) should be measured with an error of less than ±5 %. Information on thickness measurement can be found in IEC 62047-2:2006, Annex C and in IEC 62047-3:2006, Clause 6.

### 4.4 Evaluation of energy release rate

The energy release rate ($G$) is evaluated using the following Equation (1):

$$G = \frac{21(1-\nu^2)P^2L^2}{16Eb^2h^3} \qquad (1)$$

where

- $\nu$ is Poisson's ratio;
- $E$ is Young's modulus;
- $b$ is the width of a test piece;
- $h$ is the thickness of the supporting structure;
- $L$ is the spacing between two adjacent pins, and
- $P$ is the applied load.

The critical energy release rate or the interfacial adhesion energy is evaluated from the energy release rate when the interfacial crack lying in the interface starts to grow. This formula is valid for brittle fracture.

## 5 Testing method and test apparatus

### 5.1 Test principle

The test is performed by applying a pure bending moment to a test piece with a layered material interface as shown in Figure 1. The test consists of two steps: the first is the introduction of an interface crack between two layered materials with the weakest interface. By gradually increasing the four-point bending load applied to the test piece, a crack is initiated from a machined pre-crack. The initiated crack extends in a direction of thickness and then becomes an interface crack when it touches the weakest interface. When the interface crack is formed, the bending load is relaxed. The second step is the extension of the interface crack. By reloading the bending load, the energy release rate at the tip of the interface crack increases, and the interface crack starts to extend. The energy release rate of an interface crack is independent of the crack length when the test piece is under pure bending. For this test method, it is unnecessary to measure the length of the interface crack.

### 5.2 Test machine

The test machine is a universal testing machine with a compressive loadcell, a servomotor, a linear guide, and a displacement sensor. Using a four-point bending fixture as shown in Figure 2, a pure bending moment is applied to a test piece with layered materials. The diameter of the loading pins should be larger than the thickness of the test piece and five times less than the length of the test piece. The load and the displacement shall be measured using a loadcell and displacement sensors installed on the universal testing machine during the test. The resolution of the loadcell shall be less than 1/100 of the critical load for interface cracking, and the resolution of the displacement sensor shall be less than 1/100 of the maximum stroke during the test. The loading speed shall be chosen in a range from 0,000 1 mm/s to 0,1 mm/s for steady state interfacial cracking without any inertial effect. The behaviour of crack extension shall be recorded at a speed larger than 10 Hz using a camera with a microscope objective of 20x or more.

### 5.3 Test procedure

The test procedure is as follows:

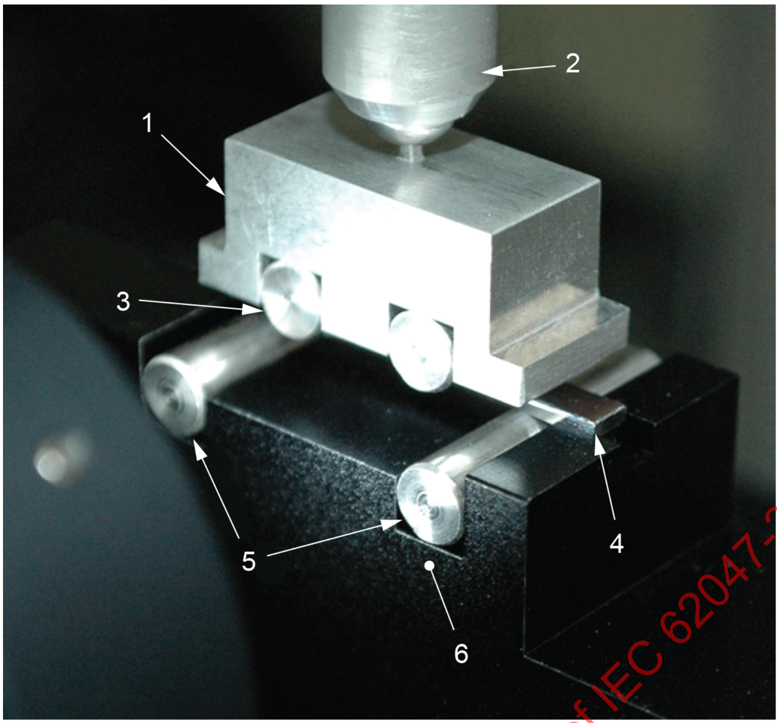
a) Install a test piece on the four-point bending fixture of the test machine. The longitudinal direction of the test piece shall be aligned with the longitudinal direction of the fixture, and the deviation angle shall be less than 1 °.

b) Apply a bending moment to the test piece to initiate a crack from the machined pre-crack or notch. This is for introduction of an interface crack at the weakest interface, and the application of bending moment shall be controlled by a displacement of the test machine. When the crack starts to extend, the actuator of the test machine shall be stopped to prevent the test piece from complete delamination. When the interface is brittle, and the adhesion energy is low, the test piece is susceptible to complete delamination after initiation of the crack at the machined pre-crack. When the initiated crack does not touch any material interface of the test piece, the displacement of the test machine shall be increased to grow the crack further. The test machine shall be stopped and unloaded as soon as the interface crack is formed.

c) Apply a bending moment again to the test piece with the interface crack at a material interface, and record the load and the displacement during the test. The crack motion shall be recorded to interpret the physical meaning of the measured critical energy release rate.

d) Unload the test piece after a testing period. After testing, remove the test piece from the test machine with caution for additional damage on the fractured surfaces of the test piece. If possible, preserve the test piece for investigation using electron and optical microscopes.

e) Check the measured load-displacement curves according to Annex A. To obtain the interfacial adhesion energy, the interfacial cracking should proceed in a steady-state manner and the load-displacement curve should be as in Figure A.1a). This test method is invalid when the interfacial cracking is not in a steady-state.

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**Key**

1 upper fixture

2 loading pin

3 upper loading pin pair

4 specimen

5 lower loading pin pair

6 lower fixture

**Figure 2 – Picture of a four-point bending fixture**



### 5.4 Test environment

Because mechanical properties are temperature sensitive, fluctuations in temperature during the test shall be controlled to be less than ±2 °C. Certain polymeric materials can be sensitive to humidity; thus, the change in relative humidity (RH) in the testing laboratory shall be controlled to be less than ±5 % RH for such materialsu.

## 6 Test report

The test report shall contain the following information:

a) reference to this document;

b) test piece identification number;

c) test piece preparation procedures;

d) test piece dimensions;

e) constituent materials and their fabrication methods;

f) description of the test machine and four-point bending fixture;

g) measured results and properties: applied bending moment versus displacement, critical energy release rate, crack extension behaviour.

# Annex A
(informative)

# Failure modes during the four-point bending test

## A.1 General

Depending on the materials consisting of an interface in a layered MEMS device, the interfacial adhesion energy of the interface can be higher or lower than the fracture toughness of the materials. This four-point bending test method is valid for the interface with the lower interfacial adhesion energy. The measured critical energy release rate has a physical meaning of the interfacial adhesion energy only when the interfacial crack steadily extends along the interface. It is useful to understand some types of load versus displacement curves obtained from the four-point bending test for the interpretation of the measured data.

## A.2 Some failure modes

During the four-point bending test, several types of load versus displacement curves can be obtained for different material combinations and interfaces as shown in Figure A.1. In Figure A.1a), the force peak (1) corresponds to the crack initiation at the notched pre-crack. The force peak (2) indicates the initiation of an interfacial crack, and the data (3) represents steady state crack growth along the interface. In this case, it is possible to evaluate the critical energy release rate from Formula (1). When the interfacial toughness is very low, the initiated crack at the notched pre-crack can lead to unstable growth of interfacial crack as shown in Figure A.1b), and the critical energy release rate at the interface crack is not determined. In Figure A.1c), the crack growth is initiated at the notched pre-crack under the force peak (6), then the arrested crack starts to extend at the force peak (7) through the other supporting structure. This means that the interface is very tough, and the critical energy release rate of the interface is undetermined. When the toughness of the supporting structures is very low or the notched pre-crack is blunt, the initiated crack at the notched pre-crack can extend throughout the two supporting structures as shown Figure A.1d).