

TECHNICAL SPECIFICATION

**Nanomanufacturing - Key control characteristics -
Part 6-26: Graphene-related products - Fracture strain and stress, Young's
modulus, residual strain and residual stress: bulge test**



THIS PUBLICATION IS COPYRIGHT PROTECTED
Copyright © 2025 IEC, Geneva, Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either IEC or IEC's member National Committee in the country of the requester. If you have any questions about IEC copyright or have an enquiry about obtaining additional rights to this publication, please contact the address below or your local IEC member National Committee for further information.

IEC Secretariat
3, rue de Varembe
CH-1211 Geneva 20
Switzerland

Tel.: +41 22 919 02 11
info@iec.ch
www.iec.ch

About the IEC

The International Electrotechnical Commission (IEC) is the leading global organization that prepares and publishes International Standards for all electrical, electronic and related technologies.

About IEC publications

The technical content of IEC publications is kept under constant review by the IEC. Please make sure that you have the latest edition, a corrigendum or an amendment might have been published.

IEC publications search -
webstore.iec.ch/advsearchform

The advanced search enables to find IEC publications by a variety of criteria (reference number, text, technical committee, ...). It also gives information on projects, replaced and withdrawn publications.

IEC Just Published - webstore.iec.ch/justpublished
Stay up to date on all new IEC publications. Just Published details all new publications released. Available online and once a month by email.

IEC Customer Service Centre - webstore.iec.ch/csc
If you wish to give us your feedback on this publication or need further assistance, please contact the Customer Service Centre: sales@iec.ch.

IEC Products & Services Portal - products.iec.ch

Discover our powerful search engine and read freely all the publications previews, graphical symbols and the glossary. With a subscription you will always have access to up to date content tailored to your needs.

Electropedia - www.electropedia.org

The world's leading online dictionary on electrotechnology, containing more than 22 500 terminological entries in English and French, with equivalent terms in 25 additional languages. Also known as the International Electrotechnical Vocabulary (IEV) online.

Warning! Make sure that you obtained this publication from an authorized distributor.

CONTENTS

FOREWORD	3
INTRODUCTION	5
1 Scope	7
2 Normative references	7
3 Terms and definitions	7
3.1 General terms	7
3.2 Mechanical properties related terms	8
3.3 Key control characteristics measured according to this standard	8
3.4 Terms related to the measurement method	9
4 General	9
4.1 Measurement principle	9
4.1.1 Measurement principle of a single kind of material layer	9
4.1.2 Measurement principle of composite layer by two kinds of material	10
4.2 Sample preparation method	11
4.2.1 General	11
4.2.2 Graphene	11
4.2.3 Nanometre-thick film deposited on Si wafer	13
4.2.4 Nanometre-thick film deposited on Cu or Ni foil	14
4.3 Measurement system	16
4.4 Description of measurement equipment and apparatus	18
4.5 Supporting materials	18
5 Measurement procedure	18
5.1 Calibration of measurement equipment	18
5.2 Detailed protocol of the measurement procedure	18
6 Results to be reported	18
6.1 General	18
6.2 Product or sample identification	18
6.3 Test conditions	19
6.4 Test results	19
Annex A (informative) Format of the test report	20
Annex B (informative) Effect of sample geometry on stress–strain relation for three typical geometries	22
Annex C (informative) Worked examples	23
C.1 Graphene bulge test	23
C.2 Si ₃ N ₄ and SiN _x /SiO ₂ film bulge test	23
Bibliography	26
Figure 1 – Applications of mechanical properties to electrical devices	5
Figure 2 – Various measurement methods to evaluate mechanical properties of 2D and thin films	6
Figure 3 – Schematic view of the central section of sample during bulge testing	10
Figure 4 – Determination of the plane-strain modulus, the residual stress (σ_0) and residual strain (ϵ_0) from the experimental stress–strain curve	10
Figure 5 – Schematic view of the central section of a composite sample during bulge testing: a long rectangular membrane	10

Figure 6 – Typical steps for the preparation of a freestanding graphene (or 2D material) sample with polycarbonate (PC) supporting layer	12
Figure 7 – Typical PC/graphene samples: (a), (b) PC/graphene on SiO ₂ wafer from separate experimental runs; (c), (d) PC/graphene freestanding samples from separate experimental runs.....	12
Figure 8 – Typical steps for the preparation of a freestanding metal sample using Si wafer.....	14
Figure 9 – Typical steps for the preparation of a freestanding nanometre-thick film of graphite on metal foil substrate	15
Figure 10 – Typical freestanding samples after sample preparation process	16
Figure 11 – Measurement system consisting of bulge test chamber, pressure sensor, auto-focus light source for measuring deflection of film, high-speed camera and mass flow controller	17
Figure B.1 – Overview of three different geometries	22
Figure C.1 – Stress–strain curve from bulge test for graphene and PC/graphene	23
Figure C.2 – Bulge tests comprising both loading and unloading	24
Figure C.3 – Stress–strain curve from bulge test for SiN _x of 50 nm.....	24
Figure C.4 – Stress–strain curve from bulge test for graphite film of 50 nm.....	25
Figure C.5 – Stress–strain curve from bulge test for graphite film of 50 nm synthesized on Ni/Si wafer	25
Table A.1 – Product identification (in accordance with the relevant blank detail specification)	20
Table A.2 – General material description	20
Table A.3 – Measurement condition.....	21
Table A.4 – Test result.....	21

INTERNATIONAL ELECTROTECHNICAL COMMISSION

**Nanomanufacturing - Key control characteristics -
Part 6-26: Graphene-related products - Fracture strain and stress,
Young's modulus, residual strain and residual stress: bulge test**

FOREWORD

- 1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
- 2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.
- 3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
- 4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.
- 5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
- 6) All users should ensure that they have the latest edition of this publication.
- 7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
- 8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
- 9) IEC draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). IEC takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, IEC had not received notice of (a) patent(s), which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at <https://patents.iec.ch>. IEC shall not be held responsible for identifying any or all such patent rights.

IEC TS 62607-6-26 has been prepared by IEC technical committee 113: Nanotechnology for electrotechnical products and systems. It is a Technical Specification.

The text of this Technical Specification is based on the following documents:

Draft	Report on voting
113/924/DTS	113/939/RVDTS

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this Technical Specification is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

A list of all parts of the IEC TS 62607 series, published under the general title *Nanomanufacturing - Key control characteristics*, 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 webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn, or
- revised.

INTRODUCTION

When the characteristic dimensions of materials are reduced to the nanoscale regime, their mechanical properties exhibit significant changes compared to their bulk counterparts. These changes include enhancements in elasticity, residual strain, and fracture resistance, which are critical for reliable and high-performance nanoscale devices. Low-dimensional materials, such as graphene and nanometre-thick films, have gained widespread attention because of their exceptional thermal, optical, electrical, and mechanical properties. These unique characteristics make them indispensable in the development of advanced nanoscale technologies.

The mechanical properties of two-dimensional (2D) materials, such as Young's modulus (or elastic modulus), residual strain, residual stress, and fracture stress, are essential for their integration into diverse applications. As shown in Figure 1, these properties are utilized in several applications. These include

- strain sensors for precise mechanical deformation detection,
- energy harvesting devices using piezoelectric effects to convert mechanical to electrical energy,
- vibrational acoustic applications supporting sound generation or absorption, and
- pellicle membranes for EUV lithography that maintain structural stability under high thermal and mechanical stresses during device operation [1]¹, [2], [3], [4], [5].

These applications highlight the versatility of mechanical properties in enabling innovative engineering solutions at the nanoscale.

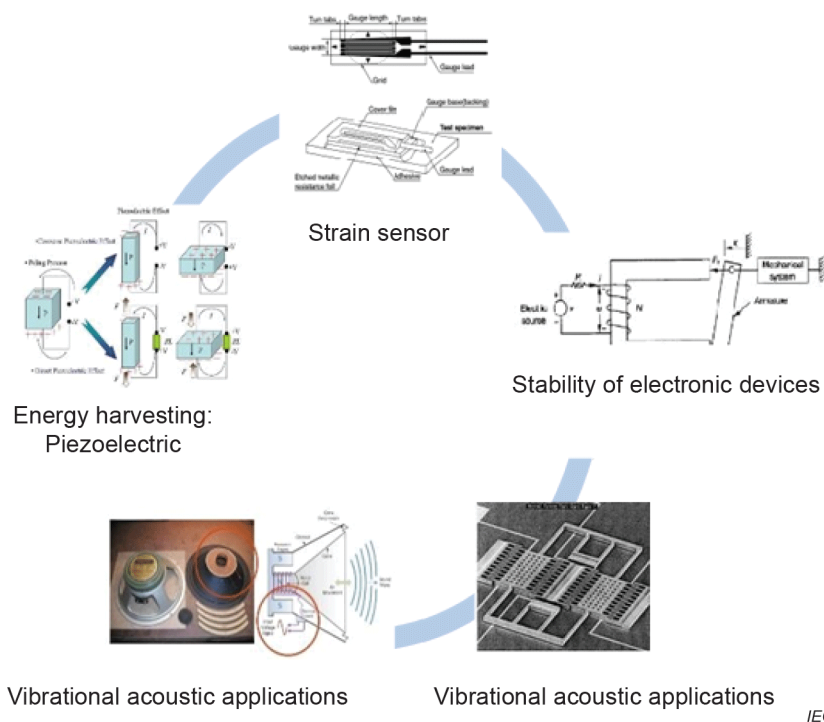


Figure 1 – Applications of mechanical properties to electrical devices

¹ Numbers in square brackets refer to the Bibliography.

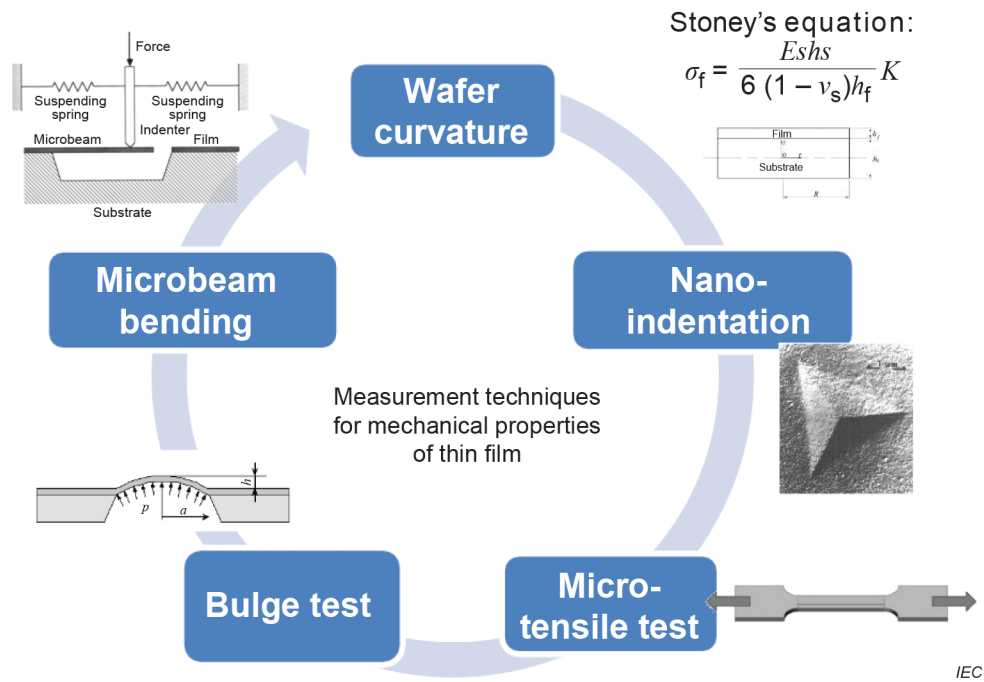


Figure 2 – Various measurement methods to evaluate mechanical properties of 2D and thin films

Accurate characterization of the mechanical properties of 2D materials and thin films is essential for their effective use in applications. However, conventional methods face significant challenges. For example, (i) substrate interference often affects techniques like nano-indentation or wafer curvature, making it difficult to isolate the intrinsic material properties, and (ii) sample preparation complexity limits the applicability of methods such as micro-tensile tests and microbeam bending. To overcome these limitations, it is important to adopt methods designed specifically for freestanding films, which can provide more accurate and reproducible results.

Figure 2 presents the various techniques used for evaluating the mechanical properties of 2D materials and thin films. These techniques include

- nano-indentation, suitable for localized measurements but limited by its focus area,
- wafer curvature, effective for measuring residual stress but influenced by the substrate,
- microbeam bending and micro-tensile tests, useful in specific cases but requiring labour-intensive preparation, and
- bulge test, a reliable method for freestanding films, which measures Young's modulus (or elastic modulus), residual strain, residual stress, and fracture stress under well-controlled conditions.

Among these, the bulge test stands out for its practicality and scalability, enabling the characterization of large areas of freestanding films without substrate interference.

1 Scope

This part of IEC TS 62607 establishes a standardized method to determine the mechanical key control characteristics (KCCs)

- Young's modulus (or elastic modulus),
- residual strain,
- residual stress, and
- fracture stress

of 2D materials and nanoscale films using the

- bulge test.

The bulge test is a reliable method where a pressure differential is applied to a freestanding film, and the resulting deformation is measured to derive the mechanical properties.

- This method is applicable to a wide range of freestanding 2D materials, such as graphene, and nanometre-thick films with thicknesses typically ranging from 1 nm to several hundred nanometres.
- This document ensures the characterization of mechanical properties essential for assessing the structural integrity and performance of materials in applications such as composite additives, flexible electronics, and energy harvesting devices.

2 Normative references

There are no normative references in this document.

Bibliography

- [1] P. J. van Zwol, M. Nasalevich, W.P.Voorthuijzen et al., "*Pellicle films supporting the ramp to HVM with EUV*", Proc. SPIE 10451, Photomask Technology 2017
 - [2] Ivan Pollentier, Jae UK Lee, Marina Timmermans et al., "*Novel membrane solutions for the EUV pellicle: better or not?*", Proc. SPIE 10143, Extreme Ultraviolet (EUV) Lithography VIII, 101430L, SPIE Advanced Lithography, 2017
 - [3] Dario L. Goldfarb, "*Fabrication of full size pellicle based on silicon nitride*", Proc. SPIE 9635, Photomask Technology 2015, 96350A
 - [4] Youke Ono, Kazuo Kohmura et al., "*Development of a novel closed EUV pellicle for EUVL manufacturing*", Proc. SPIE 9985, Photomask Technology 2016, 99850B
 - [5] Carmen Zoldesi, Kursat Bal et al., "*Progress on EUV Pellicle development*", Proc. SPIE 9048, Extreme Ultraviolet (EUV) Lithography V, 90481N, SPIE Advanced Lithography, 2014
 - [6] Benoit Merle, "*Mechanical Properties of Thin Films Studied by Bulge Testing*", Erlangen, FAU University Press (2013)
 - [7] Jin ho Ahn, <http://msenano.hanyang.ac.kr>
 - [8] Ji-Beom Yoo, <http://web.skku.edu/~nmd>
-