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TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Reliability assessment – Part 3-1: Graphene-based material – Stability: Temperature and humidity test

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Nanomanufacturing – Reliability assessment – Part 3-1: Graphene-based material – Stability: Temperature and humidity test

INTERNATIONAL ELECTROTECHNICAL COMMISSION

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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NANOMANUFACTURING –
RELIABILITY ASSESSMENT –

Part 3-1: Graphene-based material –
Stability: Temperature and humidity test

FOREWORD

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IEC TS 62876-3-1 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
113/592/DTS	113/616/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/standardsdev/publications.

A list of all parts in the IEC TS 62876 series, published under the general title *Nanomanufacturing – Reliability assessment*, 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- withdrawn,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

Graphene, a single-layer of carbon atoms arranged in a honeycomb lattice, has a high potential for future nanotechnology applications due to the excellent conductivity, transparency and flexibility of the material. Many research organizations and industrial companies are developing fabrication technologies for graphene films on substrates, as dry powders or graphene in liquid dispersions for a wide variety of applications. Therefore, the need for a system of standardized tests for the key control characteristics to benchmark graphene material in its different physical and chemical modifications remains critical. The absence of such a system slows down the development process and hinders companies to be commercially successful. To improve this situation, IEC TC 113 is developing a comprehensive system of material specifications (IEC 62565-3-X series) which list application relevant key control characteristics (KCCs) and the related measurement standards (IEC 62607-6-X series) for graphene materials in general. That includes graphene, few-layer graphene and multilayer graphene, graphene oxide and other graphene-related materials.

In addition to the KCCs to qualify graphene material after fabrication, it is also important to obtain information on its long-term stability. This document defines the conditions for a set of stress tests to qualify graphene material for an acceptable level of reliability and durability in its performance category and operating service environment.

These tests are performed using samples with graphene material layers on the same substrate as is used in the final product. If the graphene material layer in the final product is embedded between other materials, the test samples are prepared in the same way. The basic idea is to prepare test samples which are representative for the application, so that the results of the reliability screening allow the prediction of the reliability of the layer in the final product. The results of these tests will help to establish acceptance criteria for the graphene material including its manufacturing process. This document does not address the quantitative reliability measures and life predictions of the product, which would involve further testing based on the knowledge of failure mechanisms.

The objectives of this document are to:

– specify the requirements for a general reliability stress screening (RSS) standard for nano-enabled electrotechnical products using graphene and other graphene-based materials;
– give direction to the supplier and to the end user on the production and purchase of nano-enabled electrotechnical products to meet and verify reliability qualification standards for certain specified service environments;
– provide a list of reliability qualification stress tests and conditions;
– establish guidance for selection of appropriate measurements and pass/fail criteria.
– give relevant references; and
– establish the minimum reporting requirements.

This document is meant to be a general document that can be applied to all modifications of graphene materials. Even if this document focuses on graphene materials, an analogue systematics might be useable for other 2D materials. Nevertheless, as it is expected that failure mechanisms are material dependent, a direct comparison of time-to-failure values is not recommended.

As such, the results of this reliability screening test give guidance to optimize graphene material fabrication processes and the use of graphene materials in a product design.

In some cases, it may be required that prior to the tests the environmental chamber be evacuated to vacuum so the intrinsic KCCs are measured (without influence from other environmental contaminants).

It is the intent of this document to be compatible with and work in conjunction with the performance standards defined in the IEC 62607 series.

NANOMANUFACTURING – RELIABILITY ASSESSMENT –

Part 3-1: Graphene-based material – Stability: Temperature and humidity test

1 Scope

This part of IEC TS 62876 establishes a standardized method to determine the

- stability

of films of graphene-based material by a

- temperature and humidity test.

It establishes a general methodology for reliability stress screening (RSS) to qualify the use of graphene-based material in its subsequent product value stage. The intention is to prepare test samples undergoing the same or similar failure mechanisms as the graphene-based material in the final product.

- Selected stress factors in this test to drive the failure mechanisms are low temperature, high temperature, change of temperature and damp heat with respect to test severity, test sequence, sample quantities and acceptance criteria.
- The RSS test procedure defined here supports reliability assessment in the design phase of a product by providing guidelines for selecting appropriate measurement methods and how to define specific pass/fail criteria.
- The described methodology does not replace traditional reliability tests on the final product level and will not provide full reliability data which allow the estimation of product lifetimes. If the samples pass the test, the design is called qualified in accordance with IEC 62876-3-1.

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.

IEC 60068-2-1, *Environmental testing – Part 2-1: Tests – Test A: Cold*

IEC 60068-2-2, *Environmental testing – Part 2-2: Tests – Test B: Dry heat*

IEC 60068-2-14, *Environmental testing – Part 2-14: Tests – Test N: Change of temperature*

IEC 60068-2-78, *Environmental testing – Part 2-78: Tests – Test Cab: Damp heat, steady state*

3 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 General terms

3.1.1
key control characteristic
KCC
key performance indicator
material property or intermediate product characteristic which can affect safety or compliance with regulations, fit, function, performance, quality, reliability or subsequent processing of the final product

Note 1 to entry: The measurement of a key control characteristic is described in a standardized measurement procedure with known accuracy and precision.

Note 2 to entry: It is possible to define more than one measurement method for a key control characteristic if the correlation of the results is well-defined and known.

3.1.2
graphene
graphene layer
single-layer graphene
monolayer graphene
single layer of carbon atoms with each atom bound to three neighbours in a honeycomb structure

Note 1 to entry: It is an important building block of many carbon nano-objects.

Note 2 to entry: As graphene is a single layer, it is also sometimes called monolayer graphene or single-layer graphene and abbreviated as 1LG to distinguish it from bilayer graphene (2LG) and few-layer graphene (FLG).

Note 3 to entry: Graphene has edges and can have defects and grain boundaries where the bonding is disrupted.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.1]

3.1.3
graphene-based material
GBM
graphene material
grouping of carbon-based 2D materials that include one or more of graphene, bilayer graphene, few-layer graphene, graphene nanoplate and functionalized variations thereof as well as graphene oxide and reduced graphene oxide

Note 1 to entry: "Graphene material" is a short name for graphene-based material.

3.2 Terms regarding reliability

3.2.1
end of life
life cycle stage of a product starting when it is removed from its intended use stage

Note 1 to entry: In the context of IEC TS 62876-3-1, "end of life" is reached when a failure is observed.

3.2.2
failure
loss of ability to perform as required

Note 1 to entry: A failure of an item is an event that results in a *fault* (IEV 192-04-01) of that item.

Note 2 to entry: Qualifiers, such as catastrophic, critical, major, minor, marginal and insignificant, can be used to categorize failures according to the severity of consequences, the choice and definitions of severity criteria depending upon the field of application.

Note 3 to entry: Qualifiers, such as misuse, mishandling and weakness, can be used to categorize failures according to the cause of failure.

[SOURCE: IEC 60050-192:2015, 192-03-01]

3.2.3
failure criterion
pre-defined condition for acceptance as conclusive evidence of failure

Note 1 to entry: In the context of IEC TS 62876-3-1, the failure criterion is a pre-defined change of a key control characteristic (KCC) which is tested during reliability stress screening.

Note 2 to entry: If more than one KCC is observed during the test, end of life is reached if any of the KCCs reaches the failure criterion.

[SOURCE: IEC 60050-192:2015, 192-03-03, modified – The term KCC has been integrated into Note 1 to entry in the explanation of the failure criterion in this specific case. Note 2 to entry has been added.]

3.2.4
lot tolerance percent defective
LTPD
percent defective units that the sampling plan will reject 90 % of the time

Note 1 to entry: A single sampling plan for attributes consists of a sample of size n and an acceptance number c. The procedure operates as follows: select n items at random from the lot. If the number of defective items in the sample set is less than c, the lot is accepted. Otherwise, the lot is rejected.

3.2.5
mean time to failure
MTTF
expectation of the time to failure under stress

Note 1 to entry: In the case of non-repairable items with an exponential distribution of operating times to failure (i.e. a constant failure rate), the MTTF is numerically equal to the reciprocal of the failure rate. This is also true for repairable items if after restoration they can be considered to be "as-good-as-new".

Note 2 to entry: Operating time is the time accumulated from the first use, or from restoration, until failure.

Note 3 to entry: For the estimation of the MTTF from test results, the MTTF is calculated as the arithmetic mean value of the measured times to failure. If not all samples have failed, the behaviour of the remaining units might be extrapolated to the time when the sample under test falls below the failure criterion.

[SOURCE: IEC 60050-192:2015, 192-05-11, modified – In the definition, "under stress" has been added after the word "failure" and Note 3 to entry has been added.]

3.2.6
reliability
ability to perform as required, without failure, for a given time interval, under given conditions

Note 1 to entry: The time interval duration may be expressed in units appropriate to the item concerned, e.g. calendar time, operating cycles, distance run, etc., and the units should always be clearly stated.

Note 2 to entry: Given conditions include aspects that affect reliability, such as: mode of operation, stress levels, environmental conditions, and maintenance.

[SOURCE: IEC 60050-192:2015, 192-01-24, modified – Note 3 to entry has been omitted.]

3.2.7
reliability stress screening
RSS
process for detecting flaws by applying environmental and/or operational stresses to precipitate them as detectable failures.

Note 1 to entry: In the context of IEC TS 62876-3-1, the flaws are related to changes in the key control characteristics of the graphene layers for a given application.

[SOURCE: IEC 60050-192:2015, 192-09-19]

3.2.8
sample under test
SUT
manufactured product or clearly defined part of a product undergoing testing

3.2.9
time to failure
TTF
time accumulated from the first application of stress until failure

Note 1 to entry: The functionality of SUT is tested before and after the application of stress.

Note 2 to entry: SUT need not be functionally operating under stress test. Nevertheless, it is mentioned in the test report if the SUT was operating or not.

[SOURCE: IEC 60050-192:2015, 192-05-01, modified – In the term and definition, "operating time" has been replaced by "time". In the definition, "accumulated from the first use, or from restoration," has been replaced by "accumulated from the first application of stress" to render the definition more appropriate for the scope. The original Note 1 to entry has been replaced and Note 2 to entry has been added to take into account the specific application and scope.]

4 General

4.1 Sample under test

The sample under test (SUT) shall be clearly defined. In the content of this document it is always a substrate covered with a layer of a graphene material. Typical examples for substrates are the following:

– silica on silicon (SiO_2 on Si) – typical values of the thickness of the SiO_2 layer are 90 nm and 300 nm;
– SiC (0001);
– copper (Cu);
– polyethylene terephthalate (PET);
– quartz.

The graphene material layer may consist of, for example,

a) graphene,
b) few-layer graphene, or
c) graphene oxide,

which might be

1) grown on the substrate,
2) transferred from another substrate to the substrate, or
3) printed on the substrate as an ink.