IEC 62788-1-6

Edition 1.0 2020-05

INTERNATIONAL STANDARD

NORME INTERNATIONALE

AMENDMENT 1
AMENDEMENT 1

Measurement procedures for materials used in photovoltaic modules – Part 1-6: Encapsulants – Test methods for determining the degree of cure in Ethylene-Vinyl Acetate

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Procédures de mesure des matériaux utilisés dans les modules photovoltaïques – Partie 1-6: Encapsulants – Méthodes d'essai pour déterminer le degré de durcissement dans l'éthylène-acétate de vinyle

IEC 62788-1-6:2017-01/AMD1:2020-05(en-fr)

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

COMMISSION ELECTROTECHNIQUE INTERNATIONALE

ICS 27.160 ISBN 978-2-8322-8209-0

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FOREWORD

This amendment has been prepared by IEC technical committee 82:Solar photovoltaic energy systems.

The text of this amendment is based on the following documents:

FDIS	Report on voting
82/1691/FDIS	82/1720/RVD

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

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

- reconfirmed,
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- replaced by a revised edition, or
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2 Normative references

Add the following new references:

ISO 6721-1, *Plastics – Determination of dynamic mechanical properties – Part 1: General principles*

ISO 14577-1, *Metallic materials – Instrumented indentation test for hardness and materials parameters – Part 1: Test method*

3 Terms and definitions

Add the following new term:

3.6
degree of cure
G_i
<indentation method> parameter that correlates with the extent of cross-linking within the EVA using the indentation method

Note 1 to entry: Unit: dimensionless.

5.2.1 Sampling and storage

Replace the existing subclause 5.2.1.1 with the following:

5.2.1.1

Because the results for the secondary method may depend on the make of EVA, test results may only be directly compared for the same formulation of EVA. Therefore, test specimens should come from the same manufacturer(s) for the same fabrication lay-up configuration (backsheet/EVA combination). Changes in the encapsulant that affect the curing process, including but not limited to a change of the material supplier, would require validating the correlation between $G_{\%}$ and the degree of cure. For example, if the percentage vinyl acetate content in the EVA resin changes, a new correlation between $G_{\%}$ and the degree of cure (from a secondary method) should be obtained because the percentage vinyl acetate content is known to significantly affect the viscoelastic-dependent cure characteristics of the encapsulant.

7 Test report

Replace the existing item h) with the following:

h) identification of test method used and test instrument and other equipment used, including the laminator and the temperature, pressure, and time settings used, when applicable. In the case of the indentation secondary method (per correlation or usual use of method), the test temperature, tip material, tip geometry, and tip size, maximum indentation load, maximum indentation depth, and frequency of modulation (if applicable) shall also be reported;

Replace the existing item i) with the following:

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i) reference to sampling procedure, where relevant, including the number of tests per specimen;

Replace the existing item k) with the following:

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k) measurements (associated and their uncertainty), examinations and derived results supported by tables, graphs, sketches and photographs as appropriate including degree of cure, specimen mass, measured enthalpy, graphs of the enthalpy/temperature data, graphs of the crystallization peaks, and gel content;

Add the following new clause:

8 Indentation secondary method

8.1 General

An alternative secondary method, using indentation to characterize the degree of cure of EVA, has emerged from the PV industry. The method is presently being used by module manufacturers and has been demonstrated in in-line application. A general description of the principle, related equipment, and use of the method is given herein.

G_i is obtained using an indentation instrument, where the result may follow from the analysis of the applied load, penetration depth, use of a modulated applied load (such as harmonic stiffness, storage modulus, loss modulus, or tan[δ]), and/or the specimen viscoelastic response (including the relaxation or recovery response). G_i may be obtained from a more complicated dimensionless fit, including a fit applied to the specimen's viscoelastic response (e.g., using a Maxwell model) or a combination of characteristics monitored during indentation. G_i may be obtained from a dimensionless fit of the response of the specimen (for example

$$G_i = \frac{C_\mathrm{t} - C_\mathrm{n}}{C_\mathrm{m} - C_\mathrm{n}}$$

where

C is the characteristic of interest,

C_t is the test specimen;

C_m is the reference specimen with the greatest thermal history (“maximum correlated”, e.g., most-cross-linked); and

C_n is the reference specimen with minimal or no thermal history (“minimum correlated”, i.e., not-laminated or not-cured).

NOTE A suitable apparatus is supplied by LayTec AG[1] in Germany. For this presently available commercial equipment, G_i can range from 0,3 to 2,5..

The terminology and definitions related to dynamic indentation, including harmonic stiffness, storage modulus, loss modulus may be found in ISO 14577-1. The terminology and definitions related to tan[δ] may be found in ISO 6721-1.

8.2 Principle

The indentation method probes the mechanical response (viscoelastic characteristics) of cured EVA layers, which change due to the cross-linking of the EVA during the curing process. In the method, a probe tip (connected to a force transducer) is pressed against the flexible back-sheet affecting the EVA. The corresponding reaction force acting on the tip during indentation and subsequent relaxation is recorded by the force transducer and analyzed, giving a figure of merit (G_i) describing the viscoelastic properties of the material. The principle of the indentation method is described in the related references in the bibliography.

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A correlation between the degree of cure and the gel content can be established by a series of measurements on samples, each with a different degree of cure. Indentation can thus be used as a secondary method to quantify the degree of cure. To enable more widespread comparison, the same set of sample materials shall be characterized using the gel content method (Clause 6 of IEC 62788-1-6) after indentation to establish a correlation between gel content and the degree of cure. The indentation method is valid for the specific combination of EVA and back-sheet examined.

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This test procedure may also be applied to cross-linking ethylenic co-polymers other than EVA. The maximum tip displacement, maximum applied load, and temperatures identified for the indentation measurements in this procedure have been optimized for EVA. For other materials, the optimum temperature depends on the stack of materials subject to indentation, including the encapsulant and backsheet.

The method and instrumentation is designed for non-destructive examination of PV modules with a flexible back-sheet for the purpose of manufacturing process control. The method may not be applied to modules with a glass/glass laminated construction. The indentation method may be used for quality control in production lines. The variation of the method (one standard deviation) is typically ≤ ±3 % (see Kunath et al.).

8.3 Instrument and equipment for the indentation method

Equivalent instruments that contain the following components and can be shown to provide repeatability and reproducibility of ≤ ±5 % for two standard deviations for correlation of G_i as defined in Section 3.6 may be used.

The instrument consists of:

[1] This information is given for the convenience of users of this International Standard and does not constitute an endorsement by IEC of the product named.

– a rigid probe;

– a force transducer connected to the probe, measuring the reaction force acting on the probe during indentation;

– a temperature regulation system, to maintain the test specimen (module) at a designated temperature;

– a mechanically rigid linear stage that may be used to move the probe and force transducer for site-specific indentation.

8.4 Instrument calibration

The tip, force transducer, and temperature regulation system shall be verified and calibrated regularly according to the specifications of the equipment vendor.

8.5 Correlation of the degree of cure

For EVA, to establish a correlation between the figure of merit (G_i) given by the instrument and the gel content, a set of specimens with different cross-linked gel content ($G_{\%}$) including the range of the manufacturer's specification limits (e.g., between 70 % and 93 %) shall be used. Specimens for correlation, usually prepared by varying the lamination time or temperature, shall first be examined using the indentation tester. The correlation specimens shall be subsequently analysed using the gel content method (Clause 6 of IEC 62788-1-6) so that an empirical correlation (best fit or series of best fits through the range of correlation) between $G_{\%}$ and G_i is established. Figure 5 (similar to Lux et al.) shows an example of a correlation for an EVA material. The figure shows an example of correlation data for a single material rather than the application of indentation to multiple module specimens, therefore no error bars are given. Because of the nonlinear nature of the curing process, the results of the secondary methods (including indentation) should not be extrapolated beyond the range of $G_{\%}$ and G_i of the correlation specimens.

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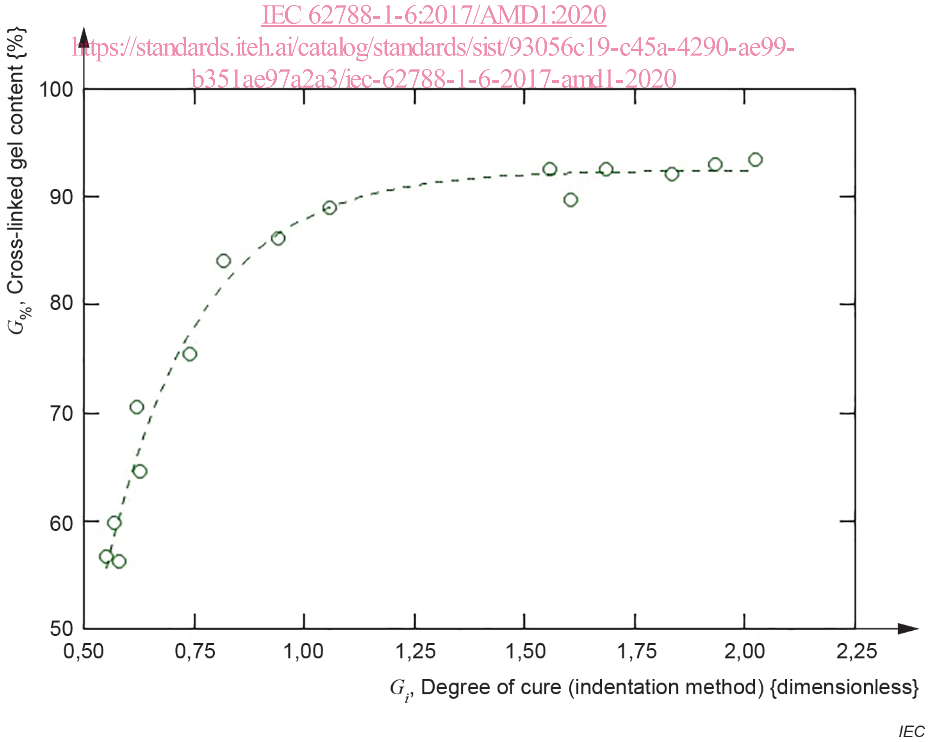

An example of the empirical correlation (dashed line for a first order exponential fit) is shown for the correlation data (circles) for a representative combination of encapsulant and backsheet.

Figure 5 – Example of the correlation applied between G_i (indentation) and $G_{\%}$ (gel content)

8.6 Specimen preparation for the indentation method

The indentation method does not require sample specific preparation. Test specimens may consist of full-size modules or mini-modules. Specimens shall be placed in the test device with the backsheet-side facing the probe tip. Measurements shall only be conducted at locations within the specimen where just a cell is present (i.e., not between cells or at a location where the interconnect ribbon is present) and at least 10 mm away from the edge of a cell. The measurement locations should be clean and without defects or damage in the specimen materials.

If measurements are performed for the purpose of correlation between $G_{\%}$ and G_i, subsequent preparation requirements for the gel content method may apply.

8.7 Test procedure for the indentation method

A module specimen is placed under the indentation test probe, with both the specimen and probe being held rigidly in place. The specimen is heated to a temperature above the melt transition temperature of the encapsulant (for example, within the range of 85 °C and 95 °C for EVA) and maintained constant at the designated temperature ±1 °C for all measurements. For example, heating can be achieved from below with a heater, such as a halogen light source that irradiates the front (glass side) of the PV module. When the designated test temperature is achieved and stabilized, the probe is pressed into the backsheet/encapsulant stack and the measurement is performed. For contemporary backsheet materials laminated on EVA encapsulant, the stabilization time of at least 45 s is recommended; however, shorter stabilization times may be used if validated for indentation. For contemporary backsheet materials laminated on EVA encapsulant, the maximum indentation depth may be in the order of 120 µm and the applied load may be in the order of 15 N. The optimum measurement temperature range for crosslinking encapsulant materials other than EVA has not yet been established. If the test procedure is applied to other encapsulation materials, the range of the test temperatures may have to be adjusted.

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The measurement for the indentation method gives the figure of merit, G_i, characterizing the degree of cure based on the viscoelastic response of the specimen.

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