ROUND-ROBIN VERIFICATION AND FINAL DEVELOPMENT OF THE IEC 62788-1-5 ENCAPSULATION SIZE CHANGE TEST

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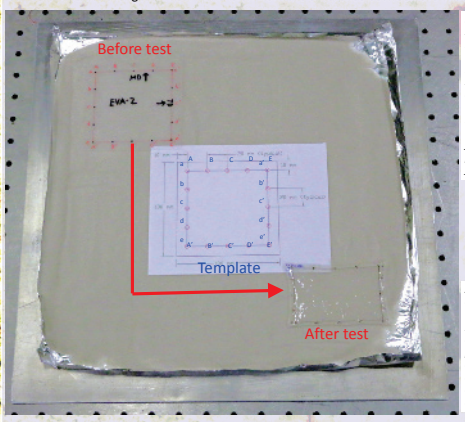
Introduction

Polymeric encapsulation materials may a change size when processed at typical module lamination temperatures. The relief of residual strain, trapped during the manufacture of encapsulation sheet, can affect module performance and reliability. For example, displaced cells and interconnects threaten: cell fracture; broken interconnects (open circuits and ground faults); delamination at interfaces; and void formation. The IEC 62788-1-5 standard quantifies the maximum change in linear dimensions that may occur to allow for process control of size change. Developments incorporated into the Committee Draft (CD) of the standard as well as the assessment of the repeatability and reproducibility of the test method are described here. No pass/fail criteria are given in the standard, rather a repeatable protocol to quantify the change in dimension is provided to aid those working with encapsulation.

Test Protocol: Method, Equipment, and Specimen Sampling

The test procedure generally follows as:

1. Size, mark, and measure the specimen(s) size (to ±0.5 mm) in their unprocessed state.
2. Place specimen(s) in a oven on a preheated substrate consisting of handle/aluminum foil/"graded" sand layers.
3. Maintain the specimen(s) in the oven for 5 minutes.
4. Remove the specimen(s) from the oven, allow them to cool to ambient, and measure their final size.
5. Calculate the change in linear dimension from the difference between the initial and final measurements.



LEFT: *Photograph of representative EVA specimens (top left) before and (bottom right) after performing the test. A template (made to scale) was used to mark the test specimens. In the test, specimens (100 mm squares) are placed on a layer of ASTM C788 "graded" sand (2-4 mm thick, to minimize friction) which is located on an aluminum foil (20-25 μm thick, to render uniform temperature). The foil, at least 300 x 300 mm in size, is placed here on an aluminum sheet, used for support an ease of handling.*

$$\Delta L = 100 \times \frac{L_f - L_i}{L_i}$$

ABOVE: *The change in linear dimension is calculated as a percentage. A negative value indicates the encapsulant has shrunk; a positive value indicates the encapsulant has increased in size. In practice all magnitudes and signs of size change have been observed. The results are distinguished between the machine extrusion direction **(MD)** and transverse direction **(TD)** for rolls of encapsulation.*

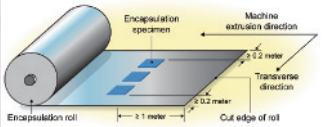
Standardized measurement locations were defined between the New Work Item Proposal (NWIP) and CD versions of the standard. The effects of the location within the specimen or at the edge of the specimen were quantified in discovery experiments at the time the NWIP was submitted. From that, a set of (5) measurements for the MD and (5) for the TD should be obtained 1 cm from the specimen periphery. The measurement grid is meant to minimize bias between measurements at the corners relative to the middle of the specimen. In practice, some materials were more prone to size-change at the corners (*e.g.*, the line aa'), while other materials were more affected at their middle (*e.g.*, the line cc'). For 6 specimens, the maximum and difference (maximum minus minimum) should be reported to identify ***greatest*** change in size that may occur for unconstrained material. The average and standard deviation should also be reported to give a more ***representative*** sense of the size change that may occur during the lamination of PV modules.

ABOVE: *A leveling tool may be used to grade the sand substrate. In the example here, a "C" shaped object is used to flatten the sand. The tool shown is recessed (out of plane to the photo) in its center (between the handles at the edges). The tool shown will level the sand between the handles when dragged over the sand in the direction towards the top or bottom of the photo. In practice, any straight-edge (such as a blade, rigid applicator, ruler, roller, or similar object) may be used to grade the sand.*

ABOVE: *A circulating closed-loop controlled oven is required for the test. The differences in the duration and magnitude of radiation present are readily distinguished in the figure. If an aluminum foil substrate at least 300 mm x 300 mm in size is used, multiple specimens may be tested simultaneously in the oven. If space permits, sand used in subsequent tests may be equilibrated in the oven, e.g., in a jar or other container, while it is being used for the size change test.*

Test equipment was better defined after the NWIP and interlaboratory study. A straight-edge or leveling tool may be used to flatten the "graded" sand substrate to the thickness of 2 mm to 4 mm. (The use of talc, kraft paper, glass, stainless steel, and liquid water as substrate materials was explored previously, see references). A convection oven (accurate to ±2.5 °C) was specifically identified for the standard upon review of the initial work. Circulating air will aid the specimen(s) to more quickly achieve and stabilize at the test temperature. Convection is expected to improve the temperature uniformity within the oven and the repeatability of the applied temperature between tests. Circulating ovens were used in the round-robin test described below. From the round-robin, it may be necessary to baffle the fan, *e.g.*, through strategic placement of shelves, in order to prevent blowing sand or inadvertent specimen movement during the test.

Material samples are to be obtained at the beginning, middle and end of at least two separate rolls of encapsulation. Each sample set consists of a specimen obtained from the interior in addition to two specimens obtained closer to the edges of the roll (as shown in the schematic). A minimum of 18 specimens would therefore be examined for datasheet reporting. Additional sampling may be performed for the purpose of quality control and manufacturing process control. The quantities and protocol for sampling are not strictly specified in this case, rather these should be consistent with the manufacturer's own procedures.



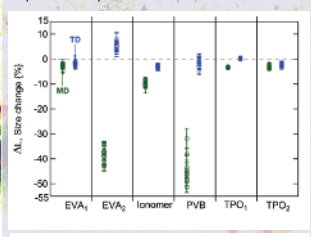
ABOVE: *Schematic identifying the designated sampling locations at the ends of a roll of encapsulation. The requirements for specimen location relative to the periphery of the roll are shown, such that the cutting and handling of the roll should not greatly affect the specimens prior to examination.*

Experimental Results

Round-robin verification of the test was performed using (6) representative materials, including: poly(ethylene-co-vinyl acetate) (EVA); poly(ethylene-co-methacrylic acid metal salt) ("ionomer"); polyvinyl butyral (PVB); thermoplastic polyethylene/polyoctene copolymer (TPO, aka polyolefin); and thermoplastic polyurethane (TPU). Two types of EVA were examined: one that was processed to reduce size change ("EVA_1"), and one that was not necessarily manufactured to reduce size change ("EVA_2"). Two TPO materials, obtained from different manufacturers, were examined.

The goal of the round-robin was to quantify the repeatability (variability within each laboratory) and reproducibility (the variability between the laboratories in addition to the repeatability). The round-robin was conducted for the CD version of the standard as in ISO 5725-2 and ASTM E691. ***From the round-robin, the repeatability and reproducibility are no better than the order of 1%.*** A correlation was observed between the variation and magnitude of the measurements.

During its development, including: discovery experiments; an interlaboratory experiment; CD version; and subsequent publication, the standard was improved significantly from its original implementation as a manufacturer's test protocol. For example, two materials examined in the round-robin were significantly improved from previous versions examined in the interlaboratory study.



ABOVE: *Box-plot of the round-robin data. The results are given first for the machine extrusion direction **(MD)** in green and then the transverse direction **(TD)** in blue. The average and standard deviation of the 6 specimens examined is shown for each of the 12 participating laboratories. Outliers have been removed from the box-plot and summary tables. Outliers were identified using Mandel's h and k statistics to evaluate variation between and within laboratories, respectively. Outliers were then confirmed using Cochran's test (for h) or Grubb's test (for k).*

ΔL Results: Machine Extrusion Direction (%)

Material	MAX	DIFF	x	s_x	s_r	s_R	r	R
EVA_1	1.30	-7.35	-2.59	0.97	0.54	1.04	2.73	2.91
EVA_2	-31.83	14.62	-38.72	6.62	1.01	6.13	18.55	17.17
Ionomer	-7.00	9.00	-9.18	1.30	2.26	2.56	3.64	7.15
PVB	-27.23	26.94	-44.78	3.44	5.53	6.36	9.64	17.81
TPO_1	-2.74	1.21	-3.25	0.20	0.18	0.26	0.56	0.72
TPO_2	-1.63	3.14	-3.17	0.49	0.64	0.78	1.36	2.19

ΔL Results: Transverse Direction (%)

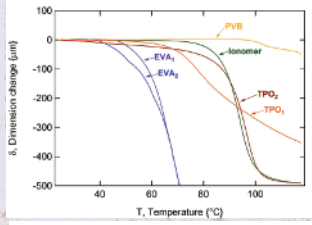
Material	MAX	DIFF	x	s_x	s_r	s_R	r	R
EVA1	0.84	-5.72	-2.01	0.76	0.59	0.91	2.12	2.55
EVA2	13.38	-12.87	5.63	2.56	0.95	2.52	7.16	7.06
Ionomer	-1.55	3.85	-3.51	0.73	2.09	2.19	2.05	6.14
PVB	3.21	-9.57	-1.26	1.43	1.73	2.17	4.01	6.08
TPO1	0.75	-1.00	0.21	0.19	0.15	0.23	0.54	0.66
TPO2	-0.51	3.48	-2.41	0.57	0.60	0.79	1.60	2.22

ABOVE: *Summary of the round-robin experiment. Quantities include: the maximum size change observed at each laboratory (MAX, as reported in the standard); the difference (DIFF, maximum minus minimum, as reported in the standard); the average (x, as reported in the standard); the standard deviation (s_x, as reported in the standard); the repeatability of the standard deviation (s_r); the reproducibility of the standard deviation (s_R); the repeatability (r); and the reproducibility (R). All values apply to a percent change in the linear dimension of the test specimens.*

Materials characterizations, including thermomechanical analysis (TMA) and differential scanning calorimetry (DSC), were performed on the round-robin materials to examine their mechanical response relative to their phase transition temperatures. These characterizations clarify the physical state and response for each material relative to the processing temperature used during the test. The temperatures in the round-robin were recommended by the material manufacturer. DSC identifies that all of the samples were examined in their melt state (note other PVB formulations may not exhibit a melt transition). TMA identifies that the materials may be affected by a mechanical load at the test temperatures used in the round-robin, confirming their propensity to strain relieve during the test. EVA may exhibit melt transitions up to 80 °C. EVA typically begins to cross-link above 120 °C, limiting strain relief. It is therefore recommended to examine EVA at 100 °C. Strain relief is not limited in materials that do not cross-link with temperature; therefore, they should be examined in their melt or glassy states, as recommended by their manufacturer.

MATERIAL	TEST TEMPERATURE IN R-R (°C)	DSC $T_g = T_{\alpha}$ (°C)	DSC T_c (°C)	DSC T_{m1} (°C)	DSC T_{m2} (°C)	TMA T_s (°C)
EVA 1 (balanced)	150	<-25	37	48	65	58
EVA 2 (unbalanced)	150	<-25	37	44	65	58
Ionomer	140	<-25	58	55	86	87
PVB	165	42	86	97	146	99
TPO 1	150	<-25	50	49	65	69
TPO 2	150	<-25	78	46	95	88

ABOVE: *Summary of: test temperatures used in the round-robin; the glass transition (alpha-relaxation) temperature, T_g; the crystallization (freezing) temperature, T_c; the melt temperature, T_m; and the softening point temperature, T_s. DSC was performed using a Q2000 (TA Instruments, Waters LLC) to execute a single heating and cooling cycle up to 220 °C at 10°C·min^{-1}. For all materials, two melt events were observed upon initial heating, as indicated with the subscripts -1 and -2. "< -25" is used to indicate when T_g could not be identified within the lower limit of the DSC scan.*



ABOVE: *Data profiles for the TMA characterization. The softening point is determined from the profile, when a measurable change in dimension may be invoked as the temperature is increased. Penetration probe measurements were performed using a Q400 (TA Instruments, Waters LLC) to perform a single heating up to 120 °C at 10°C·min^{-1}. while applying a load of 0.1 N.*

Summary

A standardized test for the characterization of change in linear dimensions of encapsulation sheet has been developed and verified.

The round-robin experiment described here identified that the repeatability and reproducibility of measurements is on the order of 1%.

Recent refinements to the test procedure to improve repeatability and reproducibility include:

- The use of a convection oven to improve the thermal equilibration time constant and its uniformity
- Well-defined measurement locations reduce the effects of sampling size -and location- relative to the specimen edges
- A standardized sand substrate may be readily obtained to reduce friction that would otherwise complicate the results
- Specimen sampling is defined, so that material is examined at known sites across the width and length of rolls
- Encapsulation should be examined at the manufacturer's recommended processing temperature, except when a cross-linking reaction may limit the size change. EVA, for example, should be examined 100 °C, between its melt transition (occurring up to 80 °C) and the onset of cross-linking (often at 100 °C).

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For More Information, refer to the publications:

D.C. Miller, L. Ji, G. Kelly, X.H. Gu, N. Nickel, P. Norum, T. Shioda, G. Tamizhmani, J.H. Wohlgemuth, "Examination of Size-Change Test for PV Encapsulation Materials", Proc. SPIE, 2012, 8472-29. http://www.nrel.gov/docs/fy12osti/54186.pdf http://www.nrel.gov/docs/fy12osti/56320.pdf

IEC 62788-5 – Measurement Procedures for Materials Used in Photovoltaic Modules: Part 1: Encapsulants. Part 5 – Measurement of Change in Linear Dimensions of Sheet Encapsulation Material Resulting From Applied Thermal Conditions," International Electrotechnical Commission: Geneva, (submitted).

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