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Edition 2.0 2021-11

INTERNATIONAL STANDARD

NORME INTERNATIONALE

Semiconductor devices – Mechanical and climatic test methods – Part 39: Measurement of moisture diffusivity and water solubility in organic materials used for semiconductor components

Dispositifs à semiconducteurs – Méthodes d'essais mécaniques et climatiques – Partie 39: Mesure de la diffusivité d'humidité et de l'hydrosolubilité dans les matériaux organiques utilisés dans les composants à semiconducteurs

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CONTENTS

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SEMICONDUCTOR DEVICES – MECHANICAL AND CLIMATIC TEST METHODS –

Part 39: Measurement of moisture diffusivity and water solubility in organic materials used for semiconductor components

FOREWORD

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This edition includes the following significant technical changes with respect to the previous edition:

a) updated procedure for "dry weight" determination.

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

Draft	Report on voting
47/2652/CDV	47/2725/RVC

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 International Standard 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 the parts of the IEC 60749 series, under the general title *Semiconductor devices – Mechanical and climatic test methods*, 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- withdrawn,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.

SEMICONDUCTOR DEVICES – MECHANICAL AND CLIMATIC TEST METHODS –

Part 39: Measurement of moisture diffusivity and water solubility in organic materials used for semiconductor components

1 Scope

This part of IEC 60749 details the procedures for the measurement of the characteristic properties of moisture diffusivity and water solubility in organic materials used in the packaging of semiconductor components.

These two material properties are important parameters for the effective reliability performance of plastic packaged semiconductors after exposure to moisture and being subjected to high-temperature solder reflow.

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 60749-20, *Semiconductor devices – Mechanical and climatic test methods – Part 20: Resistance of plastic encapsulated SMDs to the combined effect of moisture and soldering heat*

3 Terms and definitions

No terms and definitions are listed in this document.

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

4 Apparatus

4.1 Analytical balance capable of a resolution of either 0,000 01 g or 0,001 % of sample mass.

4.2 High-temperature oven capable of maintaining uniform temperatures from 100 °C to 250 °C ± 2 °C.

4.3 Temperature/humidity chamber(s) capable of maintaining temperatures in a range from 30 °C to 85 °C and relative humidities (H_R) in a range from 60 % H_R to 85 % H_R. Within the chamber working area, temperature tolerance shall be ±2 °C and the H_R tolerance shall be ±3 % H_R.

4.4 Perforated stainless steel trays or stainless steel wire mesh baskets used for holding samples and for placement into ovens.

4.5 Large aluminium plate or disk used for heat sink capability.

4.6 Desiccator for holding dry samples.

5 Samples

Samples of mould compound shall be flat parallel-sided discs or coupons. The linear dimensions shall be accurately measured to within ±0,02 mm.

To approximate one-dimensional diffusion behaviour with edge effects limited to less than 5 % of the total diffusional moisture mass uptake, the free surface area in the thickness dimension shall be less than 5 % of the flat-sided free surface area of the sample. For a disc of radius, r, and thickness, h, the following relation shall be met:

$$h < 0{,}05r \tag{1}$$

for a coupon of length, L, and width, W,

$$h = \frac{0{,}05(WL)}{(W+L)} \tag{2}$$

Recommended sample thickness should be in the range from 0,3 mm to 1,0 mm. The maximum sample thickness should not exceed 1,0 mm, because the time to achieve moisture saturation at temperatures below 60 °C will be excessively long for compounds with slow diffusivity.

The moisture absorption parameters used in this standard can be obtained from the material suppliers (such as the resin supplier).

6 Procedure

6.1 Sample preparation

6.1.1 Process and cure the samples using recommended processing conditions in accordance with the manufacturer's specification.

6.1.2 To obtain the appropriate sample thickness as given by Formulae (1) or (2), samples can be sectioned and finely polished from larger specimens. Near parallel-sided flatness shall be maintained for samples prepared in this manner.

The prepared samples should be inspected for voids, both internal and surface, using acoustic microscopy or x-ray. The ideal samples should be nearly void-free.

6.2 Absorption measurements below 100 °C

6.2.1 Measure the linear dimensions of the prepared sample to the nearest ±0,02 mm. Record the sample thickness, h, and calculate the sample volume, V, using the appropriate geometric relationship based on the sample shape.

NOTE Calculating the volume by measuring the linear dimensions is never accurate. The error is smaller when the sample is big. One accurate way of determining the volume is to use Archimedes's principle, which is to measure the sample weight in air and immersed in a liquid with known density (ethyl alcohol, IPA, etc.). In this way, the volume of a sample with irregular shape can also be determined.

6.2.2 The dry weight of the sample shall be determined, in accordance with IEC 60749-20, firstly by baking the sample for 24 h at 125 +5/-0 °C and continuing to bake and weigh the sample every 12 hours until no further weight loss is observed to ensure that the sample(s) are dry. The dry weight is determined when no further weight loss is observed, less than 0,002 % difference, after two consecutive measurements with a minimum baking interval of 12 h. Within 30 minutes after removal from the oven, weigh the sample(s) using the analytical balance equipment described in 4.1 and determine the dry weight in accordance with 6.2.4.

In accordance with IEC 60749-20, small sample(s) (less than 1,5 mm total height), devices should be weighed within 30 minutes after removal from oven.

6.2.3 Remove the sample from the bake oven and immediately cool by placing in contact with the heat sink of 4.5.

If more than one sample is to be measured, the samples and heat sink should be placed into a desiccator to limit moisture uptake during the mass measurements.

6.2.4 Weigh the sample using the balance described in 4.1 and record the mass as $M_{\text{Comp,dry,1}}$.

Read points: At the desired read point, remove the sample(s) from the bake oven. Within 30 minutes after removal of the sample(s) from the bake oven, remove the sample(s) from the container and determine their weight using the analytical balance equipment in 4.1. Within 30 minutes after weighing the samples, place them in a clean, dry, shallow container so that the sample bodies do not touch each other. Return the sample(s) to the bake oven for the desired time. Continue until the sample(s) have lost all their moisture as determined by the dry weight in 6.2.2.

6.2.5 Place the sample(s) into a stainless steel holder and transfer to a temperature/humidity chamber stabilized at a pre-set temperature and humidity.

The sample should be transferred into a stainless-steel holder that has been preheated and stabilized to the set chamber temperature.

6.2.6 At accumulative times, remove the sample from the temperature/humidity chamber, cool and measure the sample mass in accordance with 6.2.4. Record the mass as $M_{\text{Comp,wet,t}}$.

6.2.7 Read Points: The X-axis (time) read points, in accordance with IEC 60749-20, are selected for plotting the absorption curve. For the early readings, points should be relatively short (24 h or less) because the curve will have a steep initial slope. Later readings can be spread out further (10 days or more) as the curve becomes asymptotic. The Y-axis (weight gain) should start with ''0''and increase to the saturated weight gain. Most sample(s) will reach saturation between 0,3 % and 0,4 % when stored at 85 °C/85 % RH. Devices shall be kept at room ambient between removal from the oven or chamber and weighing and subsequent reinsertion into the oven or chamber.

Ensure that no condensed moisture from the chamber walls comes into contact with a sample during removal from the temperature/humidity chamber. If condensed water should contact a sample, immediately dry the sample using nitrogen or dry air. The sample should then be returned to the chamber for re-equilibration and another data point taken at a later time.

The sample weight measurement shall be made within a few minutes after removal of the sample from the temperature/humidity chamber. Time delays longer than 5 minutes after removal from the temperature/humidity chamber could affect the sample weight measurements.

Within 30 minutes after weighing the samples, place them in a clean, dry, shallow container so that the sample bodies do not touch each other. Return the sample(s) to the temperature/humidity chamber for the desired time.

6.2.8 Place the sample back into the temperature/humidity chamber and continue mass measurements until either of the following conditions are met:

a) additional weight gain after a 24 h period is less than 0,002 % from the previous measurement;

b) a plot of the weight gain versus time shows a linearly increasing weight gain after an initial decreasing change in mass with time (dM/dt), as depicted in Figure 1.

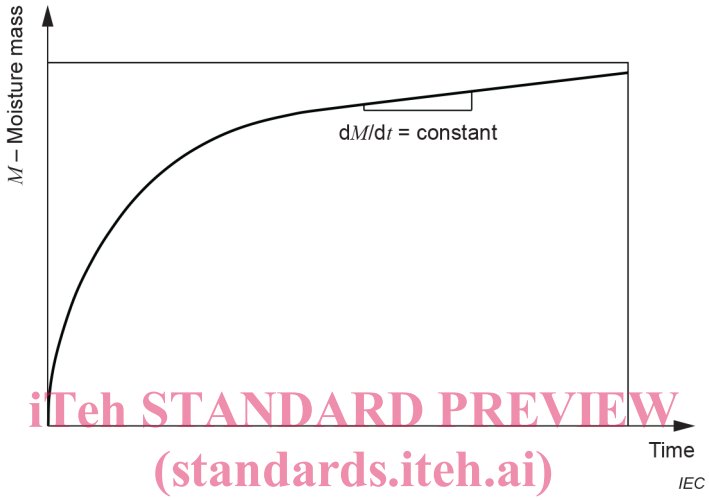

Figure 1 – Example of linearly increasing mass gain

6.2.9 Record the final wet mass of the sample as $M_{Comp,wet,f}$.

6.2.10 Bake the sample again at 125 °C until dry as determined by 6.2.2.

6.2.11 Record the second final dry mass as $M_{Comp,dry,2}$.

6.2.12 Record the saturated moisture mass as, $M_{Sat} = M_{Comp,wet,f} - M_{Comp,dry,2}$.

NOTE An alternative method to estimate the reversible saturated moisture mass can be determined by an intercept approach as shown in Figure 2. Using this method the intercept point between the weight gain curve and a linear extrapolation of the linear varying portion of the weight gain curve can be used to estimate the reversible Fickian moisture weight gain response.

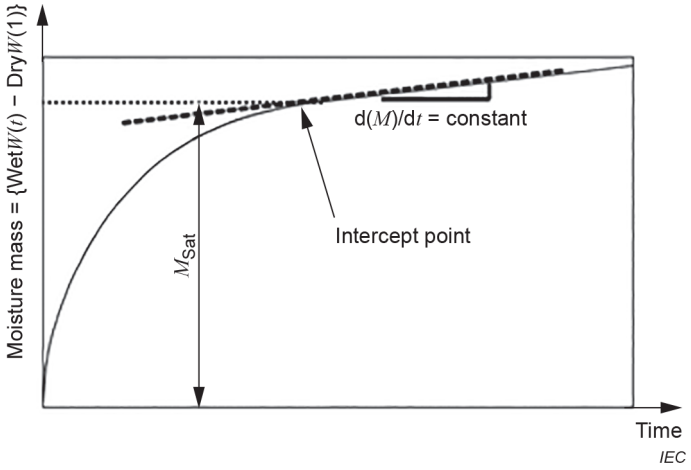

Figure 2 – Alternative intercept method to estimate the reversible Fickian moisture mass

6.3 Solubility and diffusivity calculation

6.3.1 Calculate the solubility at the given temperature and humidity by using:

$$C_{sat}(T,H_R) = \frac{M_{Comp,wet,f} - M_{Comp,dry,2}}{V} = \frac{M_{sat}(T,H_R)}{V} \quad (3)$$

where

$C_{sat}(T,H_R)$ is the moisture solubility at temperature T and H_R (in mg cm^{-3});

$M_{Comp,wet,f}$ is the final wet sample mass (in mg);

$M_{Comp,dry,2}$ is the final dry sample mass after the second bake (in mg);

V is the sample volume (in cm^3);

$M_{sat}(T,H_R)$ is the reversible saturated moisture content at temperature T and H_R (in mg).

6.3.2 Plot mass gain curve versus time using change in mass as $M(t) - M_{Comp,dry,1}$

6.3.3 Using the plotted curve, calculate the moisture diffusivity from

$$D(T) = \frac{0{,}049\ 19\ h^2}{t_{0,5}} \quad (4)$$

where

$D(T)$ is the diffusivity at temperature T (in mm^2 s^{-1});

H is the sample thickness (in mm);

$t_{0,5}$ is the absorption half-time defined as the time at which the absorbed mass of moisture is equal to one-half the saturated mass, for example, M_t/M_{sat} = 0,5;

M_t is the mass of moisture at time t.

NOTE Formula (4) is recognized as an approximation to the analytical closed form solution, however, it will provide an accurate approximation to less than a few percent error. An alternate method for determining $D(T)$ is to use a best fit curve fitting approach of the experimental weight gain data. The following solution for rectangular or square samples can be used:

$$\frac{M_t}{M_{sat}} = 1 - \frac{512}{\pi^6} \sum_{l=0}^{\infty} \sum_{m=0}^{\infty} \sum_{n=0}^{\infty} \frac{\exp(-D\,t\,L_{eqv})}{(2l+1)^2\,(2m+1)^2(2n+1)^2} \tag{5}$$

where $L_{eqv} = \left(\left\{ \frac{(2l+1)}{x_0} \right\}^2 + \left\{ \frac{(2m+1)}{y_0} \right\}^2 + \left\{ \frac{(2n+1)}{z_0} \right\}^2 \right)$

Here, x_o, y_o, and z_o are the width, length, and thickness of the sample, respectively. l, m and n represent integers relating to calculation of diffusion/concentration steps solved by iterative calculations in each principal direction. The value of $D(T)$) determined by a curve fitting technique using Formula (5) should be compared to the value determined by Formula (4) as a reference check.

6.3.4 Repeat the absorption measurements 6.2 to 6.3.3 using different temperature and humidity conditions. The following environmental conditions shall be used: 30 °C/60 % H_R, 60 °C/60 % H_R, and 85 °C/60 % H_R.

6.4 Desorption measurements above 100 °C

6.4.1 Place the sample in a chamber maintained at 85 °C/60 % H_R or 85 °C/85 % H_R for 168 h or until M_{sat} is achieved as determined by a calculation using a previously determined diffusivity at 85 °C.

6.4.2 Remove the sample from the temperature/humidity chamber, cool in accordance with 6.2.3 and record the saturated sample weight, M_{sat}.

6.4.3 Immediately transfer the sample into a stainless-steel holder that has been preheated and stabilized at the set bake temperature and place in a bake oven stabilized at a temperature greater than 100 °C.

6.4.4 Remove the sample after a recorded elapsed period of time, immediately cool in accordance with 6.2.3 and measure the sample weight in accordance with 6.2.4.

6.4.5 Repeat steps 6.4.3 and 6.4.4 until the sample is dry.

Appropriate times for recording weight losses can be determined by using a first-order extrapolation of the value for the diffusivity by using an Arrhenius fit (see Clause 7 and Clause 8) of the absorption diffusivities determined in 6.3.3.

Estimated weight losses can be assessed by using the following equation:

$$\frac{M_t}{M_{sat}} = 1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left\{ -\frac{(2n+1)^2 \pi^2 D\,t}{h^2} \right\} \tag{6}$$

where

D is the diffusivity;

t is the time.

6.4.6 Calculate $D(T)$ using Formula (4), where $t_{0,5}$ is now defined as the time at which the desorbed mass of moisture is equal to one-half of the saturated mass.