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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

IEC 60749-39:2021-11 RLV(en)

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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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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.

~~NOTE It is recommended that the moisture absorption parameters used in this standard be obtained from the material suppliers (such as the resin supplier).~~

2 Normative references

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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 ~~must~~ 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 ~~must~~ 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. ~~It is recommended that~~ 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 ~~may~~ can be sectioned and finely polished from larger specimens. ~~Care must be taken to maintain~~ 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 ~~Bake the sample at 125 °C for 24 h. Longer bake times may be required depending on the sample mass loss characteristics. The sample is considered dry when successive measurements result in less than 0,002 % difference between readings.~~

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 ~~Measure the mass of the sample according to 2.1 and record the mass as $M_{\mathrm{Comp,dry,1}}$~~

~~The mass gain/loss measurement shall be made within a few minutes after removal of the sample from the environmental chamber. Time delays longer than 5 min after removal from the environmental chambers could affect the resultant diffusivity measurements.~~

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

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.

~~It is suggested that~~ 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~~ in accordance with 4.2.3 and measure the sample mass~~ and measure the sample mass in accordance with 6.2.4. Record the mass as $M_{\mathrm{Comp,wet,t}}$.

~~Time intervals should be spaced so as to allow adequate measurement duration to capture the initial quick mass response and to provide a good spread in the data points during the later stages of the mass response curve. The total number of times the temperature/humidity chamber is disturbed for sample removal should be minimized.~~

~~Care should be taken that no condensed moisture from the chamber walls comes into contact with the sample during removal from the temperature/humidity chamber. If condensed water should contact the 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.~~

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 ($\mathrm{d}M/\mathrm{d}t$), as depicted in Figure 1.

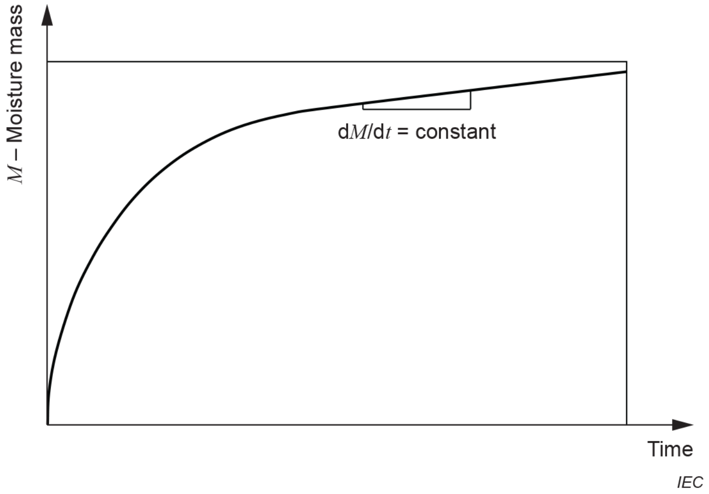

Figure 1 – Example of linearly increasing mass gain

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

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 $M_{\text{Comp,dry,2}}$

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

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.

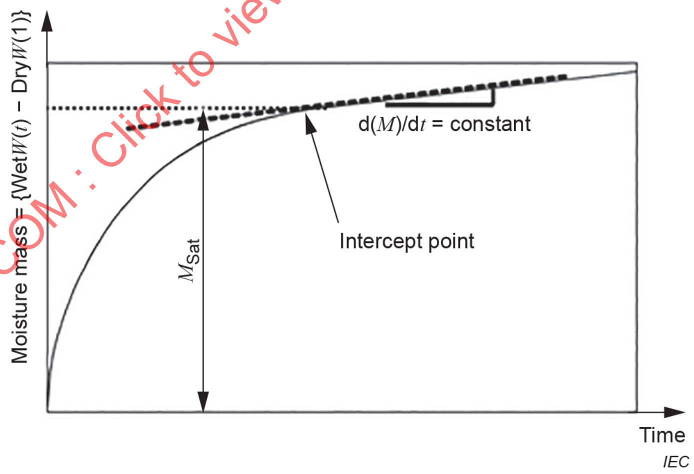

Figure 2 – Alternative intercept method to estimate the reversible Fickian moisture mass

6.3 Solubility and diffusivity calculation

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

$$C_{\text{sat}}(T, H_{\text{R}}) = \frac{M_{\text{Comp,wet,f}} - M_{\text{Comp,dry,2}}}{V} = \frac{M_{\text{sat}}(T, H_{\text{R}})}{V} \tag{3}$$

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

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

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 An alternate method for determining $D(T)$ is to use a best-fit curve-fitting approach of the experimental weight gain data. Equation (4) above is recognized as an approximation to the analytical closed form solution, however, it will provide an accurate approximation of an error of less than a few per cent. The value of $D(T)$ determined by a curve-fitting technique should be compared to the value determined by equation (4) as a reference check. Repeat the sorption measurements 4.2 to 4.3.3 using different temperature and humidity conditions. Suggested environmental conditions are 30 °C/60 % H_R, 60 °C/60 % H_R, and 85 °C/60 % H_R.~~

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 ~~place~~ 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.

~~It is suggested that the sample be transferred into a stainless-steel holder that has been preheated and stabilized at the set bake temperature.~~

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:

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

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.

6.4.7 Reset the bake oven to a higher bake temperature and repeat measurements following 6.4.1 to 6.4.6.

7 Calculation of activation energy for moisture diffusion

The activation energy for moisture diffusion is calculated from the slope of a plot of ln$\{D(T)\}$ versus $1/T$ where T is in degrees Kelvin. Report the best fit line as:

$$D(T) = D_0 \exp\left\{-\frac{E_a}{kT}\right\} \qquad (7)$$

where

D_0 is the pre-exponential factor for fitted line (in mm^2/s);

E_a is the activation energy (in eV);

k is Boltzman's constant, 8,617 × 10^{-5} eV/K.

~~NOTE 1~~ Diffusion of moisture ~~may~~ can show a dependency on the glass transition temperature (T_g) of the material. Measurements above the T_g of the material should be reported as a separate activation energy and pre-exponential factor.

NOTE ~~2~~ For accurate determination of the activation energy, ~~it is recommended that~~ a minimum of three temperatures differing by 20 °C to 30 °C for both above and below T_g is used.

8 Calculation of functional fit for solubility

The solubility can be fitted to the following formula:

$$C_{\mathrm{sat}}(T,P) = PS_0 \exp\left\{-\frac{E_{\mathrm{s}}}{\mathrm{k}T}\right\} \tag{8}$$

where

S_0 is the pre-exponential factor for fitted line (in mg^2 cm^{-3} Pa);

E_s is the activation energy (in eV);

k is Boltzman's constant, 8,617 × 10^{-5} eV/K.

9 Summary

The following information shall be tabulated:

a) mould compound identification (see Clause 5);
b) test temperature and relative humidities (see Clause 6);
c) D_0 (see Clause 7);
d) E_a, activation energy (eV) (see Clause 7);
e) $C_{\mathrm{sat}}(T,H_R)$, solubility at a given temperature and humidity (see 6.3);
f) $D(T)$, diffusivity at temperature (see 6.3).

Bibliography

JESD22-A120B, *Test Method for the Measurement of Moisture Diffusivity and Water Solubility in Organic Materials Used in Electronic Devices*

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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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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.

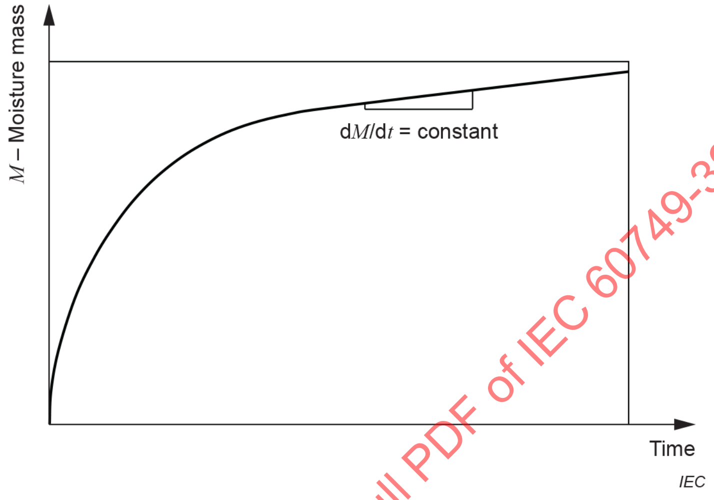

Figure 1 – Example of linearly increasing mass gain

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

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 $M_{\text{Comp,dry,2}}$.

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

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.

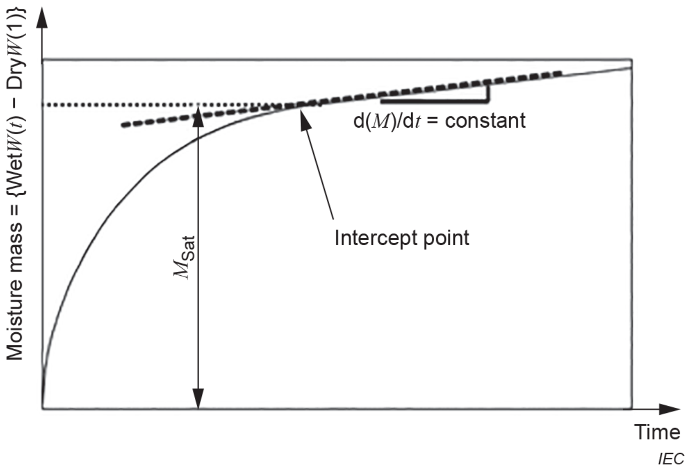

Figure 2 – Alternative intercept method to estimate the reversible Fickian moisture mass

6.3 Solubility and diffusivity calculation

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

$$C_{\text{sat}}(T, H_{\text{R}}) = \frac{M_{\text{Comp,wet,f}} - M_{\text{Comp,dry,2}}}{V} = \frac{M_{\text{sat}}(T, H_{\text{R}})}{V} \tag{3}$$

where

$C_{\text{sat}}(T,H_{\text{R}})$	is the moisture solubility at temperature T and H_{R} (in mg cm^{-3});
$M_{\text{Comp,wet,f}}$	is the final wet sample mass (in mg);
$M_{\text{Comp,dry,2}}$	is the final dry sample mass after the second bake (in mg);
V	is the sample volume (in cm^3);
$M_{\text{sat}}(T,H_{\text{R}})$	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 $M(t) - M_{\text{Comp,dry,1}}$

6.3.3 Using the plotted curve, calculate the moisture diffusivity from

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

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, $M_{\text{t}}/M_{\text{sat}}$ = 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:

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

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:

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

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.

6.4.7 Reset the bake oven to a higher bake temperature and repeat measurements following 6.4.1 to 6.4.6.

7 Calculation of activation energy for moisture diffusion

The activation energy for moisture diffusion is calculated from the slope of a plot of ln$\{D(T)\}$ versus 1/T where T is in degrees Kelvin. Report the best fit line as:

$$D(T) = D_0 \exp\left\{-\frac{E_\mathrm{a}}{\mathrm{k}T}\right\} \tag{7}$$

where

D_0 is the pre-exponential factor for fitted line (in mm^2/s);

E_a is the activation energy (in eV);

k is Boltzman's constant, 8,617 × 10^{-5} eV/K.

Diffusion of moisture can show a dependency on the glass transition temperature (T_g) of the material. Measurements above the T_g of the material should be reported as a separate activation energy and pre-exponential factor.

NOTE For accurate determination of the activation energy, a minimum of three temperatures differing by 20 °C to 30 °C for both above and below T_g is used.

8 Calculation of functional fit for solubility

The solubility can be fitted to the following formula:

$$C_\mathrm{sat}(T,P) = PS_0 \exp\left\{-\frac{E_\mathrm{s}}{\mathrm{k}T}\right\} \tag{8}$$

where

S_0 is the pre-exponential factor for fitted line (in mg^2 cm^{-3} Pa);

E_s is the activation energy (in eV);

k is Boltzman's constant, 8,617 × 10^{-5} eV/K.

9 Summary

The following information shall be tabulated:

a) mould compound identification (see Clause 5);

b) test temperature and relative humidities (see Clause 6);

c) D_0 (see Clause 7);

d) E_a, activation energy (eV) (see Clause 7);

e) $C_\mathrm{sat}(T,H_\mathrm{R})$, solubility at a given temperature and humidity (see 6.3);

f) $D(T)$, diffusivity at temperature (see 6.3).

Bibliography

JESD22-A120B, *Test Method for the Measurement of Moisture Diffusivity and Water Solubility in Organic Materials Used in Electronic Devices*

SOMMAIRE

COMMISSION ÉLECTROTECHNIQUE INTERNATIONALE

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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L'IEC 60749-39 a été établie par le comité d'études 47 de l'IEC: Dispositifs à semiconducteurs. Il s'agit d'une Norme internationale.

Cette deuxième édition, fondée sur le document JESD22-A120B du JEDEC, annule et remplace la première édition parue en 2006. Ce document est utilisé avec la permission du propriétaire des droits d'auteur qui est la JEDEC Solid State Technology Association. Cette édition constitue une révision technique.

Cette édition inclut les modifications techniques majeures suivantes par rapport à l'édition précédente:

a) mise à jour du mode opératoire relatif à la détermination du "poids sec".

Le texte de cette Norme internationale est issu des documents suivants:

Projet	Rapport de vote
47/2652/CDV	47/2725/RVC

Le rapport de vote indiqué dans le tableau ci-dessus donne toute information sur le vote ayant abouti à son approbation.

La langue employée pour l'élaboration de cette Norme internationale est l'anglais.

Ce document a été rédigé selon les Directives ISO/IEC, Partie 2, il a été développé selon les Directives ISO/IEC, Partie 1 et les Directives ISO/IEC, Supplément IEC, disponibles sous www.iec.ch/members_experts/refdocs. Les principaux types de documents développés par l'IEC sont décrits plus en détail sous www.iec.ch/standardsdev/publications.

Une liste de toutes les parties de la série IEC 60749, publiées sous le titre général *Dispositifs à semiconducteurs – Méthodes d'essais mécaniques et climatiques*, se trouve sur le site web de l'IEC.

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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

1 Domaine d’application

La présente partie de l’IEC 60749 détaille les modes opératoires pour la mesure des propriétés caractéristiques de la diffusivité d’humidité et de l’hydrosolubilité dans les matériaux organiques utilisés dans l’encapsulation des composants à semiconducteurs.

Ces deux propriétés des matériaux sont des paramètres importants pour la performance de fiabilité réelle des semiconducteurs sous boîtier en plastique après exposition à l’humidité et qui sont soumis à une refusion à température élevée au moment du brasage.

2 Références normatives

Les documents suivants sont cités dans le texte de sorte qu’ils constituent, pour tout ou partie de leur contenu, des exigences du présent document. Pour les références datées, seule l’édition citée s’applique. Pour les références non datées, la dernière édition du document de référence s'applique (y compris les éventuels amendements).

IEC 60749-20, *Dispositifs à semiconducteurs – Méthodes d’essais mécaniques et climatiques – Partie 20: Résistance des CMS à boîtier plastique à l'effet combiné de l'humidité et de la chaleur de brasage*

3 Termes et définitions

Aucun terme n'est défini dans le présent document.

L'ISO et l'IEC tiennent à jour des bases de données terminologiques destinées à être utilisées en normalisation, consultables aux adresses suivantes:

- IEC Electropedia: disponible à l'adresse http://www.electropedia.org/
- ISO Online browsing platform: disponible à l'adresse http://www.iso.org/obp

4 Appareillage

4.1 Balance analytique permettant d’obtenir une résolution de 0,000 01 g ou 0,001 % de la masse de l’échantillon.

4.2 Étuve à haute température permettant de maintenir des températures uniformes de 100 °C à 250 °C ± 2 °C.

4.3 Chambres de simulation de température et d’humidité permettant de maintenir des températures dans une plage comprise entre 30 °C et 85 °C et des humidités relatives (H_R) dans une plage comprise entre 60 % H_R et 85 % H_R. Dans la zone de travail de la chambre, la tolérance de température doit être de ± 2 °C et la tolérance de H_R doit être de ± 3 % H_R.

4.4 Plateaux en acier inoxydable perforés ou paniers maillés en acier inoxydable utilisés pour maintenir les échantillons et pour le positionnement dans les étuves.

4.5 Plaque ou disque en aluminium de grande taille utilisé pour dissiper la chaleur.

4.6 Dessiccateur pour maintenir les échantillons secs.

5 Echantillons

Les échantillons des mélanges de moulage doivent être des disques ou des éprouvettes plats à côtés parallèles. Les dimensions linéaires doivent être mesurées précisément à ± 0,02 mm.

Pour approcher un comportement de diffusion unidimensionnel avec des effets de bord limités à moins de 5 % de la reprise de masse d'humidité diffusée totale, la zone de surface libre dans la dimension de l'épaisseur doit être inférieure à 5 % de la zone de surface libre à côtés plats de l'échantillon. Pour un disque de rayon, r, et d'épaisseur, h, la relation suivante doit être satisfaite:

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

pour une éprouvette de longueur, L, et de largeur, W,

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

Il convient que l'épaisseur d'échantillon recommandée se situe dans la plage comprise entre 0,3 mm et 1,0 mm. Il convient que l'épaisseur maximale d'échantillon ne dépasse pas 1,0 mm, car le temps nécessaire pour obtenir une saturation d'humidité à des températures inférieures à 60 °C est excessivement long pour les mélanges à diffusivité lente.

Les paramètres d'absorption d'humidité utilisés dans la présente norme peuvent être obtenus auprès des fournisseurs de matériaux (les fournisseurs de résine par exemple).

6 Mode opératoire

6.1 Préparation de l'échantillon

6.1.1 Traiter et prendre en compte les échantillons en utilisant des conditions de traitement recommandées, conformément à la spécification du fabricant.

6.1.2 Afin d'obtenir l'épaisseur d'échantillon appropriée, telle qu'elle est donnée par les Formules (1) ou (2), les échantillons peuvent être prélevés et polis finement à partir d'échantillons plus grands. La planéité et le parallélisme des deux bords doivent être maintenus pour les échantillons préparés de cette façon.

Il convient de vérifier la présence éventuelle de vides dans les échantillons préparés, à la fois à l'intérieur et en surface au moyen d'un microscope acoustique ou à rayons X. Dans l'idéal, il convient que les échantillons ne présentent quasiment aucun vide.

6.2 Mesures d'absorption au-dessous de 100 °C

6.2.1 Mesurer les dimensions linéaires de l'échantillon préparé à ± 0,02 mm près. Consigner l'épaisseur de l'échantillon, h, et en calculer le volume, V, en utilisant la relation géométrique appropriée à sa forme.

NOTE Le calcul du volume par mesure des dimensions linéaires n'est jamais exact. L'erreur est plus faible quand l'échantillon est de grande dimension. Une façon de déterminer le volume exact est d'utiliser le principe d'Archimède qui consiste à mesurer le poids de l'échantillon dans l'air et immergé dans un liquide dont la densité est connue (alcool éthylique, alcool isopropylique, etc.). Ainsi, le volume d'un échantillon de forme irrégulière peut également être déterminé.

6.2.2 Le poids sec de l'échantillon doit être déterminé conformément à l'IEC 60749-20, d'abord en étuvant l'échantillon à 125 +5/-0 °C pendant 24 h et en continuant à étuver et à peser l'échantillon toutes les 12 h jusqu'à ce qu'aucune perte de poids supplémentaire ne soit observée afin de garantir que l'échantillon ou les échantillons sont secs. Le poids sec est déterminé lorsqu'aucune perte de poids supplémentaire d'une différence inférieure à 0,002 % n'est observée après deux mesures consécutives avec un intervalle d'étuvage minimal de 12 h. Dans les 30 minutes qui suivent le retrait de l'étuve, peser l'échantillon ou les échantillons au moyen de la balance analytique spécifiée en 4.1 et déterminer le poids sec conformément au 6.2.4.

Conformément à l'IEC 60749-20, il convient de peser l'échantillon ou les échantillons de petits dispositifs (d'une hauteur totale inférieure à 1,5 mm) dans les 30 minutes qui suivent le retrait de l'étuve.

6.2.3 Retirer l'échantillon de l'étuve et le refroidir immédiatement en le mettant en contact avec le dissipateur thermique prévu en 4.5.

Dans le cas où plus d'un échantillon doit être mesuré, il convient de placer les échantillons et le dissipateur thermique dans un dessiccateur afin de limiter la reprise d'humidité au cours des mesures de masse.

6.2.4 Peser l'échantillon au moyen de la balance décrite en 4.1 et consigner la masse comme suit: $M_{\text{Comp,dry,1}}$

Points de lecture: au point de lecture souhaité, retirer l'échantillon ou les échantillons de l'étuve. Dans les 30 minutes qui suivent leur retrait de l'étuve, retirer l'échantillon ou les échantillons du conteneur et déterminer leur poids à l'aide de la balance analytique spécifiée en 4.1. Dans les 30 minutes qui suivent leur pesée, disposer les échantillons dans un conteneur propre, sec et peu profond sans que les corps des échantillons se touchent. Remettre l'échantillon ou les échantillons dans l'étuve pendant la durée souhaitée. Poursuivre jusqu'à ce que l'échantillon ou les échantillons aient perdu toute leur humidité comme cela est déterminé par le poids sec en 6.2.2.

6.2.5 Placer l'échantillon ou les échantillons dans un support en acier inoxydable, puis transférer dans une chambre de simulation de température et d'humidité, stabilisée à une température et une humidité préétablies.

Il convient de transférer l'échantillon dans un support en acier inoxydable qui a été préchauffé et stabilisé à la température établie de la chambre.

6.2.6 Périodiquement, retirer l'échantillon de la chambre de simulation de température et d'humidité, refroidir et mesurer la masse de l'échantillon conformément à 6.2.4. Consigner la masse comme suit: $M_{\text{Comp,wet,t}}$.

6.2.7 Points de lecture: les points de lecture sur l'axe X (temps), conformément à l'IEC 60749-20, sont sélectionnés afin de tracer la courbe d'absorption. Pour les premières valeurs lues, il convient que les points soient notés sur une période relativement courte (24 h