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Test Method for the Measurement of Moisture Diffusivity and Water Solubility in Organic Materials Used in Integrated Circuits

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TEST METHOD FOR THE MEASUREMENT OF MOISTURE DIFFUSIVITY AND WATER SOLUBILITY IN ORGANIC MATERIALS USED IN INTEGRATED CIRCUITS

FOREWORD

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IEC-PAS 62307 was submitted by the JEDEC and has been processed by IEC technical committee 47: Semiconductor devices.

The text of this PAS is based on the following document:

This PAS was approved for publication by the P-members of the committee concerned as indicated in the following document:

Draft PAS	Report on voting
47/1595/PAS	47/1608/RVD

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NOTE DIN 53495 has been withdrawn and replaced by DIN EN ISO 62:1999-08.

Standard Test Method for the Measurement of Moisture Diffusivity and Water Solubility in Organic Materials Used in Integrated Circuits

(From JEDEC Board Ballot JCB-00-62, formulated under the cognizance of the JC-14.1 Subcommittee on Reliability Test Methods for Packaged Devices)

1 Scope

This specification details the procedures for the measurement of characteristic bulk material properties of moisture diffusivity and water solubility in organic materials used in the packaging of IC components. These two material properties are important parameters for the effective reliability performance of plastic packaged ICs after exposure to moisture and subjected to high temperature solder reflow.

This test method outlines the requirements necessary for the measurement of water sorption properties of organic materials used in the packaging of IC components.

2 Reference documents

ASTM standard D570-98, “Standard Test Method for Water Absorption of Plastics”.

SEMI G66-96, “Test Method for the Measurement of Water Absorption Characteristics for Semiconductor Plastic Molding Compounds”.

DIN 53495, “Determination of water absorption”.

3 Apparatus

3.1 Analytical balance capable of a resolution of either 0.00001g or 0.001% of sample weight.

3.2 High temperature oven capable of maintaining uniform temperatures from 100 °C – 250 °C ±2 °C.

3.3 Temperature / humidity chamber(s) capable of maintaining temperatures of 30 °C - 85 °C and relative humidities from 60%RH – 85%RH. Within the chamber working area, temperature tolerance must be ±2 °C and the RH tolerance must be ±3%RH.

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

3.5 Large aluminum plate or disk used for heat sink capability.

3.6 Desiccator for holding dry samples.

4 Test samples

4.1 Test samples must be flat parallel-sided discs or coupons. The linear dimensions must be accurately measured to within ±0.02mm.

4.1.1 To approximate one-dimensional diffusion behavior with edge effects limited to less than 5% of the total diffusional moisture mass uptake, the free surface area in the thickness dimension must 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 must be met:

$$h < 0.05r \quad (1)$$

for a coupon of length L, and width W,

$$h < \frac{0.05(W \bullet L)}{(W + L)} \quad (2)$$

Recommended sample thickness should be in the range from 0.3 – 1.0mm. It is recommended that the maximum sample thickness not exceed 1.0mm, because the time to achieve moisture saturation at temperatures below 60 °C will be excessively long for slow diffusing compounds.

5 Procedure

5.1 Sample preparation

5.1.1 Prepare samples by using proper processing parameters as recommended by the material supplier.

5.1.2 Process and cure the samples using recommended processing conditions per the manufacturer's specification.

5.1.3 To obtain the appropriate sample thickness as given by relations (1) or (2), samples may be sectioned and finely polished from larger bulk specimens. Care must be taken to maintain near-parallel sided flatness for samples prepared in this manner.

The prepared samples should be inspected for voids, both internal and surface. The ideal samples should be nearly void free.

5 Procedure (cont'd)

5.2 Absorption measurements below 100 °C.

5.2.1 Measure the linear dimensions of the prepared sample to the nearest ±0.02mm. Record the sample thickness, h, and calculate the sample volume, Vol, using the appropriate geometric relationship based on the sample shape.

5.2.2 Bake the sample at 125 °C for 24 hours. Longer bake times may be required depending on the sample weight loss characteristics. The sample is considered dry when successive measurements result in less than 0.002% difference between readings.

5.2.3 Remove the sample from the bake oven and immediately cool by placing in contact with the heat sink of 3.5.

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 weight measurements.

5.2.4 Weigh the sample according to 3.1 and record weight as DryWt(1).

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

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

5.2.6 At accumulative times, remove the sample from the temperature / humidity chamber, cool as per 5.2.3, and measure the sample weight.

Time intervals should be spaced as to allow adequate measurement duration to capture the initial quick weight response and to provide a good spread in the data points during the later stages of the weight 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.

5 Procedure (cont'd)

5.2 Absorption measurements below 100 °C (cont'd)

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

a) Additional weight gain after a 24 hour period is less than 0.002% from the previous measurement.

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

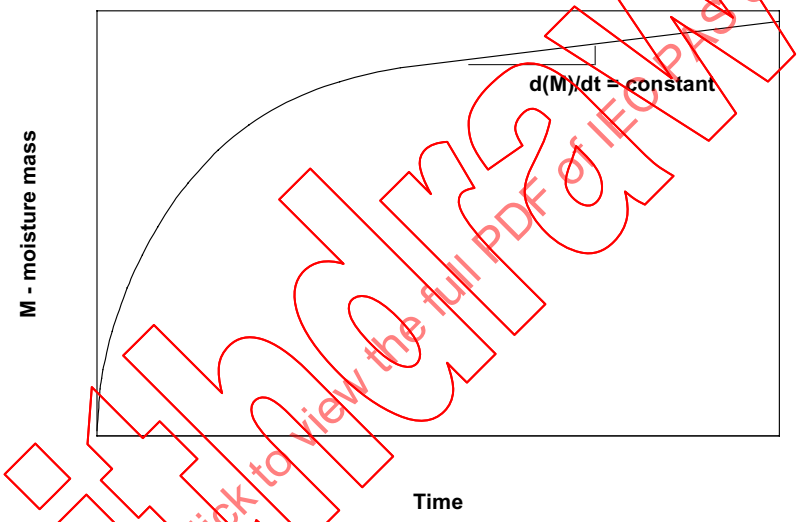

Figure 1 — Example of linearly increasing weight gain.

5.2.8 Record the final wet weight of the sample as, WetWt(f).

5.2.9 Bake the sample again at 125 °C until dry as determined per 5.2.2.

5.2.10 Record the second final dry weight as, DryWt(2).

5 Procedure (cont'd)

5.3 Solubility and diffusivity calculation

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

$$C_{Sat}(T,\%RH) = \frac{WetWt(f) - DryWt(2)}{Vol} = \frac{M_{Sat}(T,\%RH)}{Vol} \quad (3)$$

Where: $C_{Sat}(T,\%RH)$ = the moisture solubility at temperature T and %RH (mg/cm^3).

WetWt(f) = the final wet sample weight (mg).

DryWt(2) = final dry sample weight after second bake (mg).

Vol = sample volume (cm^3).

$M_{Sat}(T,\%RH)$ = saturated moisture content at temperature T and %RH (mg)

5.3.2 Plot weight gain curve verses time using change in weight as: Wt(t) – DryWt(1).

5.3.3 Using the plotted curve, calculate the moisture diffusivity from:

$$D(T) = \frac{0.04919h^2}{t_{0.5}} \quad (4)$$

Where: D(T) = the diffusivity at temperature T (mm^2/s)

h = sample thickness (mm)

$t_{0.5}$ = the sorption half-time defined as the time at which the sorbed mass of moisture is equal to one-half the saturated mass, e.g., $M_t/M_{Sat} = 0.5$.

NOTE 1 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 to less than a few percent error. 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 sorption measurements 5.2 to 5.3.3 using different temperature and humidity conditions. Suggested environmental conditions are 30 °C/60%RH, 60 °C/60%RH, and 85 °C/60%RH.

5 Procedure (cont'd)

5.4 Desorption measurements above 100 °C

5.4.1 Place sample into a chamber maintained at 85 °C/60%RH or 85 °C/85%RH for 168hrs or until M_{Sat} is achieved as determined by a calculation using a previously determined diffusivity at 85 °C.

5.4.2 Remove the sample from the temperature/humidity chamber, cool as per 5.2.3, and record the saturated sample weight, M_{Sat}.

5.4.3 Immediately place the sample into 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.

5.4.4 Remove the sample after a recorded elapsed period of time, immediately cool per 5.2.3, and measure the sample weight per 5.2.4.

5.4.5 Repeat steps 5.4.3 and 5.4.4 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 Arrehenius fit, see 6.1, of the absorption diffusivities determined in 5.3.3.

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

$$\frac{M_t}{M_{Sat}} = 1 - \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp\left\{-\frac{(2n+1)^2\pi^2 Dt}{h^2}\right\} \qquad (5)$$

5.4.6 Calculate D(T) using equation (4), where $t_{0.5}$ is now defined as the time at which the desorbed mass of moisture is equal to one-half the saturated mass.

5.4.7 Reset bake oven to a higher bake temperature and repeat measurements following 5.4.1 to 5.4.6.