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Test Methods and Acceptance Procedures for the Evaluation of Polymeric Materials

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Test Methods and Acceptance Procedures for the Evaluation of Polymeric Materials

Contents

TEST METHODS AND ACCEPTANCE PROCEDURES FOR THE EVALUATION OF POLYMERIC MATERIALS

(From Board Ballot JCB-18-04, formulated under the cognizance of the JC-13.5 Subcommittee on Hybrid, RF/Microwave, and MCM Technology.)

1 Scope

This Test Method covers the minimum requirements that should be in effect for the evaluation and acceptance of polymeric materials for use in industrial, military, space, and other special-condition products which may require capabilities beyond standard commercial microelectronics applications. It is not the intent of this Publication to specify a material, but to evaluate the material to assure that the quality and reliability of the microelectronic devices are not compromised. These materials shall be classified in two types as follows:

1) Type I being electrically conductive.
2) Type II being electrically insulative.

The user may elect to use test data from routine tests or evaluations for all or part of the user certification.

The test methods included as a part of this Publication are those which best address those physical, chemical, mechanical, and electrical properties of materials which can impact the reliability of these microelectronic devices. This Publication is not designed to be exhaustive of all techniques available as newer and more nearly absolute techniques are constantly being developed.

2 Applicable documents

The Standards and Publications listed, of current issue, form a part of this Test Method to the extent specified herein.

MIL-STD-883, *Test Methods and Procedures for Microelectronics*

MIL-PRF-38534, *General Specification Hybrid Microcircuits*

MIL-PRF-38535, *General Specification for Integrated Circuits (Microcircuits) Manufacturing*

FED-STD-406, *Federal Test Method Standard for Test of Adhesives*

ANSI/NCSL Z540-1, *General Requirements for Calibration Laboratories, Measuring and Test Equipment*

ASTM C177, *Steady State Heat Flux Measurement and Thermal Transmission Properties by Means of the Guarded Hot Plate Apparatus*

ASTM C518, *Steady State Heat Flux Measurement and Thermal Transmission Properties by Means of the Heat Flowmeter Apparatus*

ASTM D150, *AC Loss Characteristics and Dielectric Constant of Solid Electrical Insulating Materials*

2 Applicable documents (cont'd)

ASTM D257, *DC Resistance or Conductance of Insulating Materials*

ASTM D972, *Evaporation Loss of Lubricating Greases and Oils*

ASTM D1002, *Apparatus Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tensile Loading (Metal to Metal)*

ASTM D3574, *Flexible Cellular Materials-Slab, Bonded, and Molded Urethatne Forms*

ASTM D3386, *Coefficient of Linear Thermal Expansion of Electrical Insulating Materials by Thermogravimetric Method*

ASTM D3850, *Rapid Thermal Degradation of Solid Electrical Insulating Materials by Thermogravimetric Method (TGA)*

ASTM E1461, *Thermal Diffusivity of Solids by the Flash Method*

JEP114, *Guidelines for Particle Impact Noise Detection (PIND) Testing, Operator Training and Certification*

JESD22-A104, *Temperature Cycling*

JESD22-A106, *Thermal Shock*

JESD22-B103, *Variable Frequency Vibration*

JESD22-B104, *Mechanical Shock*

3 Requirements

3.1 Apparatus

Suitable measurement equipment necessary to determine compliance with the requirements of the applicable acquisition document and other apparatus as required in the referenced test methods.

3.2 Material acquisition specification

The microcircuit manufacturer shall prepare an acquisition specification describing the detailed electrical, mechanical, chemical, and thermal requirements for the polymeric material to be acquired. The requirements shall not be less stringent than those imposed by this method, but may be increased to reflect the specific parameters of a particular material or requirements of a particular application.

3.3 Certificate of compliance

The material supplier shall provide upon the user's request a certificate of compliance for each polymeric order. This certificate shall contain the actual test data for the supplier's testing as prescribed in this Publication.

3.4 Evaluation procedures

Evaluation procedures for polymeric materials shall be performed as specified in 4.1 through 4.18.

3.5 Properties of uncured materials

3.5.1 Materials

The components of a polymeric material and/or system shall be examined in accordance with Table 1 and 4.2 and shall be uniform in consistency and free of lumps or foreign matter when examined in film, liquid or other acceptable form. Any filler shall remain uniformly dispersed and suspended during the required pot life (see 4.4). The electrically conductive fillers used in type I materials shall be gold, silver, alloys of gold or silver, or other precious metals.

3.5.1.1 Encapsulating compounds

Encapsulating compounds are liquid material and are to be tested in accordance with the requirements in Table 1.

3.5.1.2 Molding compounds

Molding compounds as used in microelectronic devices are normally solid material and are to be tested in accordance with MIL-PRF-38535.

3.5 Properties of uncured materials (cont'd)

Table 1 — Requirements

Test or Condition	Test Method Paragraph	Adhesives				Absorbers				Film Dielectrics 1/				Particle Getters			
		Supplier		User		Supplier		User		Supplier		User		Supplier		User	
		A	C	A	C	A	C	A	C	A	C	A	C	A	C	A	C
Materials (3.5.1)	4.2	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Viscosity (3.5.2)	4.3	X	X			X	X			X	X						
Pot Life (3.5.3)	4.4	X	X			X	X			X	X						
Shelf Life (3.5.4)	4.5		X				X				X				X		
Thermogravimetric analysis (3.6.2)	4.6	X	X			X	X				X			X	X		
Outgassed materials (3.6.3)	4.7		X		X				X				X				X
Ionic impurities (3.6.4)	4.8	X	X			X	X				X				X		
Bond Strength (3.6.5)	4.9	X2/	X				X										
Coefficient of linear thermal expansion (3.6.6)	4.10		X														
Thermal conductivity (3.6.7)	4.11		X														
Volume resistivity (3.6.8)	4.12		X														
Type 1 materials		X2/	X														
Type 2 materials			X			X	X			X	X						
Dielectric constant (3.6.9)	4.13		X								X						
Dissipation factor (3.6.10)	4.14		X								X						
Sequential test environment (3.6.11)	4.15				X				X				X				
Density (3.6.12)	4.16																
Mechanical integrity (3.6.13)	4.17																X
Operating life test (3.6.14)	4.18																X

Test or Condition	Test Method Paragraph	Desiccants				Junction Coatings				Microwave Absorbers				Encapsulating Compounds			
		Supplier		User		Supplier		User		Supplier		User		Supplier		User	
		A	C	A	C	A	C	A	C	A	C	A	C	A	C	A	C
Materials (3.5.1)	4.2	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Viscosity (3.5.2)	4.3														X		
Pot Life (3.5.3)	4.4																
Shelf Life (3.5.4)	4.5		X				X								X		
Thermogravimetric analysis (3.6.2)	4.6	X	X			X	X								X		
Outgassed materials (3.6.3)	4.7				X				X				X				
Ionic impurities (3.6.4)	4.8	X	X				X							X	X		
Bond Strength (3.6.5)	4.9													X	X		
Coefficient of linear thermal expansion (3.6.6)	4.10														X		
Thermal conductivity (3.6.7)	4.11														X		
Volume resistivity (3.6.8)	4.12														X		
Type 1 materials																	
Type 2 materials						X	X								X		
Dielectric constant (3.6.9)	4.13														X		
Dissipation factor (3.6.10)	4.14														X		
Sequential test environment (3.6.11)	4.15				X				X								
Density (3.6.12)	4.16									X	X	X	X				
Mechanical integrity (3.6.13)	4.17																
Operating life test (3.6.14)	4.18				X												X

A = Performed at acceptance testing.

C = Performed at certification testing.

1/ Film dielectrics are defined as polymeric materials that are used in film form to act as either interlayer dielectrics, passivation layers, and/or circuit support films.

2/ Required at 25 °C test condition only. No high temperature storage required.

3.5 Properties of uncured materials (cont'd)

3.5.2 Viscosity

The viscosity of paste materials shall be determined in accordance with 4.3. The viscosity, including an acceptable range, shall be specified in the material acquisition document.

3.5.3 Pot life

The pot life when required shall be determined in accordance with 4.4 and shall be a minimum of 1 hour. The polymeric material shall be used within the pot life period after removal from the container, after mixing, or after thawing to room temperature in the case of premixed frozen polymers.

3.5.4 Shelf life

The shelf life, defined as the time that the polymeric material continues to meet the requirements of this Publication shall be determined in accordance with 4.5. This shelf life shall be a minimum of 12 months at –40 °C or below for one component system and a minimum of 12 months at room temperature (32 °C maximum) for two component systems unless the supplier certifies for some other period of time. The shelf life expiration date shall be affixed to the container by the supplier and no material shall be used after that date, unless it has been requalified.

3.5.4.1 Re-certification

Materials may be re-certified by the user once.

3.6 Properties of cured polymer materials

3.6.1 Curing of polymer materials

The material must be capable of meeting the requirements of this document when cured according to the supplier's instructions. The cure schedule for supplier tests shall be identical for all tests and shall be reported. The cure schedule for the user tests shall be the minimum cure schedule plus, as a minimum, the pre-seal bake specified in the user's assembly document and shall be reported. Deviation from the suppliers recommended cure schedule will require verification by the user of the materials performance.

3.6.2 Thermogravimetric analysis (TGA)

3.6.2.1 Thermal stability

The thermal stability of the cured material shall be determined in accordance with 4.6. Unless otherwise noted, the weight loss at 200 °C shall be less than or equal to 1.0 percent of the cured material weight.

3.6 Properties of cured polymer materials (cont'd)

3.6.2.2 Filler content

Polymeric materials using a filler to promote properties such as electrical and thermal conductivity shall be tested in accordance with 4.6 to determine the inorganic filler content. For acceptance testing, the percent filler content shall not differ from the filler content in the certified materials by more than ±2 percent.

3.6.3 Outgassed materials

Outgassing of the cured material shall be determined in accordance with 4.7. Outgassed moisture, as determined in 4.7.1, shall be less than or equal to 5,000 ppmv (0.5 percent V/V) for 3 packages (0 failures) or 5 packages (1 failure). Other gaseous species present in quantities greater than or equal to 100 ppmv (0.01 percent V/V) shall be reported in ppmv or percent V/V. The data obtained in 4.7.2 shall also be reported in the same manner but for information only. The outgassing of the cured getter shall be determined in accordance with 4.7. The vapor content of the package with getter shall not exceed 2000 ppmv after 24 hours at 150 °C and 3000 ppmv after 1000 hours at 150 °C.

3.6.4 Ionic impurities

The ionic impurity content shall be determined in accordance with 4.8 and shall meet the requirements specified in Table 2. Ionic content analysis shall be in triplicate for certification and single analysis for acceptance testing. Failure at acceptance shall require the passing of two additional samples.

Table 2 — Ionic impurity requirements

Total ionic content specified electrical conductance	**< 4.50 millisiemens/meter**
Hydrogen (pH)	4.0 < pH < 9.0
Chloride	< 200 ppm
Sodium	< 50 ppm
Potassium	< 50ppm
Fluoride	<50 ppm
NOTE Other ions present in quantities > 50 ppm shall be reported in ppm.	

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3.6.5 Bond strength

The bond strength of a polymeric material shall be determined in accordance with 4.9 at 25 °C, and 25 °C after 1,000 hours at 150 °C. The bond strength shall meet as a minimum the 1.0X requirement specified in figure 2019-4 of Method 2019 of MIL-STD-883 at each test condition. The manufacturer should test to shear or until twice the minimum 1.0X shear force is reached.

3.6 Properties of cured polymer materials (cont'd)

3.6.6 Coefficient of linear thermal expansion

The coefficient of linear thermal expansion shall be determined from -65°C to 150°C in accordance with 4.10. The coefficient of linear thermal expansion shall be ±10% of the value required in the users material specification or purchase order. Coefficient of thermal expansion should be measured in X, Y, and Z directions from mold gate to end of mold. This requirement shall apply to the material as it is configured for actual use. This requirement shall not apply to glass-supported polymeric films.

3.6.7 Thermal conductivity

The thermal conductivity shall be determined at 121 °C ±5 °C in accordance with 4.11. The thermal conductivity shall be greater than or equal to 1.5 watt/meter-K for type I polymers and greater than or equal to .15 watt/meter-K for type II polymers.

3.6.8 Volume resistivity

The volume resistivity shall be determined in accordance with 4.12. The volume resistivity of conductive materials at 25 °C, at 60 °C, at 150 °C, and at 25 °C after 1,000 hours at 150 °C shall be less than or equal to 5.0 microhm-meter for silver-filled polymers and less than or equal to 15.0 microhm-meter for gold-filled polymers. The volume resistivity of insulative materials shall be greater than or equal to 0.1 teraohm-meter at 25 °C and greater than or equal to 1.0 megohm-meter at 125 °C.

3.6.9 Dielectric constant

The dielectric constant of insulative polymeric materials shall be determined in accordance with 4.13 and shall be less than or equal to 6.0 at both 1 kHz and 1 MHz for this type of polymer but shall be less than or equal to 3.5 at 1 kHz and 1 MHz for materials used for dielectric layers.

3.6.10 Dissipation factor

The dissipation factor of insulative polymers shall be determined in accordance with 4.14 and shall be less than or equal to 0.03 at 1 kHz and less than or equal to 0.05 at 1 MHz.

3.6.11 Sequential test environment

The polymeric material shall withstand exposure to the test conditions specified in 4.15. After exposure to the complete sequence of environmental conditions, the test specimens shall show no evidence of mechanical degradation. For adhesives the measured bond strength of components shall meet as a minimum the 1.0X requirement specified on figure 2019-4 of method 2019 of MIL-STD-883.

3.6.12 Density

The density of microwave or RF absorbing materials shall be tested in accordance with 4.16. The acceptable value shall be that which is within ±10% of the value required on the user's material specification or purchase order.

3.6 Properties of cured polymer materials (cont'd)

3.6.13 Mechanical integrity

Particle getter integrity shall be verified after different levels of environmental stress as specified in 4.17.

3.6.13.1 Getter integrity (short term)

When tested in accordance with 4.17.1 all samples shall pass the criteria for PIND as defined in JEDEC Publication JEP114 or MIL-STD-883 method 2020.

3.6.13.2 Getter integrity (long term)

When tested in accordance with 4.17.2 all samples shall pass the criteria for PIND as defined in JEDEC Publication JEP114 or MIL-STD-883, method 2020, both initially and after storage at 150 °C for 1,000 hours. The salted particles shall remain attached to the getter material in the original position with no attachment and reattachment when viewed at 30X to 60X magnification.

3.6.13.3 Getter integrity (vibration)

When tested in accordance with 4.17.3 the sample shall pass PIND as defined in JEDEC Publication JEP114 or MIL-STD-883, method 2020, the salted particles shall remain attached to the getter material in the original position, with no detachment and re-attachment when viewed at 30X to 60X.

3.6.14 Operating life

When tested in accordance with 4.18, the comparison between initial and post test electrical data shall not indicate parametric shifts, which are unique to the test group containing getter material.

3.7 Responsibility for testing

The manufacturer and user are responsible for the performance of all tests as specified in Table 1 herein.

3.7.1 Test equipment and testing facilities

Test and measuring equipment and testing facilities of sufficient accuracy, quality, and quantity to permit performance of the required testing shall be established and maintained by the manufacturer and user. The establishment and maintenance of a calibration system to control the accuracy of the measuring and test equipment shall be in accordance with ANSI/NCSL Z540-1 or equivalent. The supplier and user may utilize a commercial laboratory for performing the required certification and acceptance testing.

3.7 Responsibility for testing (cont'd)

3.7.2 Testing conditions

Unless otherwise specified herein, all testing shall be performed in accordance with the test conditions specified herein.

3.8 Classification of testing

The test requirements specified herein are classified as certification testing and acceptance testing.

3.8.1 Certification testing

Certification testing shall be performed on the initial lot of material and for any major changes to the material thereafter and consist of all tests to determine conformance with all requirements specified herein. To ensure that both the polymeric material and the processes employing the material are controlled, both the supplier and the user of the material shall be responsible for performance of the tests as designated in Table 1.

3.8.1.1 Sample size

The number of samples to be subjected to each testing procedure shall be as specified in the individual test methods.

3.8.1.2 Failures

Failure of any polymeric material to meet the testing requirements shall be cause for refusal to grant certification approval.

3.8.1.3 Retention of data

All data generated for certification or re-certification shall be retained for a period of 7 years (this term coincides with Class K requirements).

3.8.2 Acceptance testing

Acceptance tests shall be performed on each lot and shall consist of tests as specified in Table 1.

3.8.2.1 Test lot

A test lot shall consist of all polymeric material manufactured under the same batch number, i.e., a batch number identifies those materials whose constituents can be traced to a single lot of raw materials.

3.8.2.2 Sample size

The number of samples to be subjected to each testing procedure shall be as specified in the individual test methods.

3.8.2 Acceptance testing (cont'd)

3.8.2.3 Failures

Failure of the samples to meet the testing requirements of a specific test shall be cause for rejection of the lot.

3.8.2.4 Retention of data

The data generated for acceptance testing shall be retained for a period of 5 years.

4 Procedure

4.1 Methods of examination and test

The following test criteria and analytical protocols shall be documented by the using activity prior to material certification.

4.2 Materials

The polymeric components or system or both shall be examined visually at a minimum magnification of 30X to ensure conformance with the requirements of 3.5.1. This can be accomplished with the aid of a stereomicroscope with the material thinly smeared on a glass microscope slide. The analyst should take into account that at high magnification, e.g., 30X, he can readily discern areas of resin and filler. The point to ascertain is, whether the filler particles are uniformly dispersed.

4.3 Viscosity

The material user and supplier shall define a mutually acceptable method for verifying the viscosity of fluid or paste materials. The supplier shall use the same method in performing the required certification and acceptance testing.

4.3.1 Viscosity test

Polymers can start very low in viscosity and become extremely viscous yet still be functional for a given application. Viscosity is best checked instrumentally wherein the material is sheared between a cone and plate assembly. Solvents and diluents as well as aging and heating can radically affect the viscosity. For these reasons viscosity of a process material should be closely controlled where it is shown to be a process parameter. Analyses are typically done at 25 °C ± 0.1 °C; however other temperatures can be used. An alternative, less expensive test for viscosity is given in 4.3.2.

4.3 Viscosity (cont'd)

4.3.2 Viscosity analysis

A determinable amount of material is extruded from a thermally equilibrated syringe in a given period of time via this procedure; subsequent calculations provide the viscosity dependent extrusion rate for the material. Fill a 3 cc LuerLok® syringe with the thermally equilibrated adhesive and stabilize it to room temperature, 25 °C ± 0.5 °C. Do not use the rubber follower. Preset the pressure on an adhesive dispenser to 60 ± 1 psig. Install a #21 needle (1/2 inch long) on the syringe and immediately place it into the adapter of the dispenser. Then extrude the material for exactly 30 seconds into a tared weighing dish. Reweigh and calculate the extrusion rate in grams/minute.

4.4 Pot life

The parameters to be used in the measurement of pot life (e.g., viscosity change, skin-over, loss of bond strength, etc.) are generally material dependent. The material supplier and user shall select the procedure to be used in establishing and testing the pot life.

4.5 Shelf life

Where applicable, an unopened container of material shall be stored under the condition specified in 3.5.4. As a minimum, the test methods and requirements specified in Table 3 shall be used to establish the shelf life.

Table 3 —Shelf life determination

Property	Requirement	Test method	Application/condition
Materials	3.5.1	4.2	All polymeric materials
Pot life	3.5.3	4.4	Adhesives; ∀ Absorbers; Junction coatings; Dielectrics
Bond strength	3.6.5	4.9	Adhesives; ∀ Absorbers; Junction coatings; 25 °C only
Volume resistivity 1/	3.6.8	4.12	Adhesives, type I, 25 °C only
1/ To be determined for materials where electrical conductivity is a design parameter.			

4.6 Thermogravimetric analysis (TGA)

The thermal stability of the polymeric system and its filler content (if any) shall be determined by testing samples of the cured system (see 3.6.1) in nitrogen using suitable TGA equipment or in accordance with ASTM D3850 or equivalent test method. Single point analyses are acceptable, however if the first sample fails, then two additional analyses must be performed. The average value of the three samples must then meet or exceed the minimum requirements.

4.6.1 Thermal stability

The thermal stability of the polymeric material shall be determined by heating the specimens from room temperature to not less than 210 °C, at a heating rate between 10 °C/minute and 20 °C/minute, in a nitrogen atmosphere with 20-30 milliliter/minute nitrogen flow. The weight loss at 200 °C shall be determined.

4.6.2 Filler content

The filler content of polymeric materials using a filler to promote properties such as electrical or thermal conductivity shall be determined by heating the specimen from room temperature to 600 °C, at a heating rate between 10 °C/minute and 20 °C/minute, in an air atmosphere with 20-30 milliliter/minute airflow. The temperature shall be maintained at 600 °C until constant weight is obtained. It is permitted to perform 4.6.1, followed by heating from 210 °C to 600 °C as detailed above. The filler content shall be reported as weight percent of the cured specimen.

4.6.3 Thermal conductivity

The ability to conduct heat is an important property of a material especially for adhesives and thermal greases. The group of materials can be unfilled, i.e., insulative, or filled with metal particles, alumina, or other conductors. Testing is rather intricate and requires large samples which become expensive to prepare when analyzing precious metal filled adhesives. These tests are best performed by the supplier of the materials or they can be subcontracted to any physical testing laboratory with this capability. The analyses should be done either to ASTM C177 for insulative materials or ASTM C518, for conductive materials, or ASTM E1461 using the flash method.

4.7 Outgassed materials

Ten test specimens shall be prepared using gold- or nickel-plated Kovar or ceramic packages, (dielectric materials may be prepared using aluminum coated silicon as the substrate). (The use of "leadless" packages is permitted to reduce moisture contributions due to package construction). The material shall be cured using the minimum cure schedule and shall receive the minimum pre-seal bake specified in the assembly document(s) (see 3.6.1). After a pre-seal bake, the packages shall be hermetically sealed. Only those packages that meet the fine and gross leak test requirements of test method 1014 of MIL-STD 883 shall be submitted for moisture content analysis. If less than 10 test specimens remain after hermetically testing, the failed packages shall be replaced by additional hermetical packages processed and tested in the same manner as the original group.

4.7 Outgassed materials (cont'd)

4.7.1 Testing for short term outgassing of moisture and other gaseous species

Five packages containing polymer prepared in accordance with 4.7 shall be heated in accordance with MIL-STD-883, method 1008, 24 hours at 150 °C. The packages shall then be immediately (less than or equal to 5 minutes) inserted into the ambient gas analysis apparatus. The packages shall be subjected to ambient gas analysis in accordance with MIL-STD-883, method 1018, procedure 1. In addition to moisture, other gaseous species present in quantities greater than or equal to 100 ppmv (0.01 percent V/V) shall be reported in ppmv or percent V/V.

All polymeric materials tested shall have quantities of material equivalent in mass and exposed surface area to that of the intended application. Gold plated Kovar tabs and alumina blanks may be used as facsimile device elements. Several polymeric materials of different application may be tested in combination with each other in this test, however their combined moisture content shall not exceed 5,000 ppmv.

4.7.2 Testing for long term outgassing of moisture and other gaseous species

Provided that the moisture requirement of 3.6.3 has been met by packages tested in 4.7.1, the remaining five devices containing polymer from the group prepared in accordance with 4.7 shall be heated in accordance with MIL-STD-883, method 1008 for 1,000 hours at 150 °C. The packages shall then be immediately (less than or equal to 5 minutes) inserted into the ambient gas analysis apparatus. The packages shall be subjected to ambient gas analysis in accordance with MIL-STD-883, method 1018, procedure 1. In addition to moisture, other gaseous species present in quantities greater than or equal to 100 ppmv (0.01 percent V/V) shall be reported in ppmv or percent V/V.

4.8 Ionic impurities

A water-extract analysis shall be performed to determine the level of ionic contamination in the cured polymeric material. The total ion content (specific electrical conductance) and the specific ionic content for the hydrogen (pH), chloride, sodium, fluoride and potassium ions shall be measured. Other ions present in quantities > 5 ppm shall also be reported in ppm. The methods of analysis submitted in the following paragraphs are suggested techniques. Federal Standard 406, Test Method 7071 may be used as a guide; however, shorter extraction periods at higher temperatures may be used providing only extractable ionics are being analyzed. Alternate methods of analysis may be selected where it can be shown that the techniques are equivalent and the method of analysis is approved by the using activity.

4.8.1 Sample preparation

Adequate material shall be cured to obtain 3 gram samples of polymer following grinding, for final preparation. The material shall be cured on teflon or other inert surface in a forced draft oven. When possible the cured specimen shall be removed from the curing substrate and ground to 60-100 mesh particles; polymeric film samples less than or equal to 0.025 cm thick shall be cured and cut into less than or equal to 0.25 cm^2 samples; gels or low modulus materials may be cast directly into the flat bottom of the sample flask for the extraction. Smaller sample sizes may be selected where it can be shown that the accuracy of the test method has not changed.

4.8 Ionic impurities (cont'd)

4.8.2 Extraction procedure

3 grams (equivalent resin) of the ground or cut equivalent polymer shall be added to a cleaned; tared, 250-ml flask made of pyrex, or equivalent. The weight of the cured material in each flask shall be recorded to the nearest milligram. 150.0 grams of deionized water with a measured specific conductance less than or equal to 0.1 millisiemens/meter (specific resistivity greater than or equal to 1.0 megohm-centimeter) shall be added to the flask. A blank shall be prepared by adding 150.0 grams of the deionized water and a boiling chip to a second 250-ml flask. The flask shall be refluxed for 20 hours.

NOTE 1.0 mho = 1.0 siemens; 1.0 mho/cm = 100.0 siemens/meter.

4.8.3 Measurement of ionic content

4.8.3.1 Total ionic content

The total extractable ionic content shall be determined by measuring the specific electrical conductance of the water-extract samples and the blank using a conductivity meter with an immersion conductivity cell having a cell constant of 0.01/centimeter (alternatively 0.1 cm^{-1} to adjust for proper analysis of the solution). The total ionic content, in millisiemens/meter, shall be obtained by subtracting the specific conductance of the blank from the specific conductance of the samples.

4.8.3.2 Hydrogen ion content (pH)

The pH of the water extract shall be determined using a pH meter with a standard combination electrode.

4.8.3.3 Specific ion analysis

Specific ion analysis of the water extract shall be conducted using ion chromatography or a demonstrated equivalent. The ion concentrations in the extract shall be converted to the sample extractable concentrations by multiplying the ratio of the deionized water weight (W) to polymer sample weight (S); that is, by (W/S). The chloride, sodium, fluoride and potassium ion levels and all other ions detected in quantities > 50 ppm shall be reported in ppm. Ionic impurity requirements shall be in accordance with 3.6.4, Table 2.

4.9 Bond strength

The bond strength of the polymeric material shall be determined in accordance with 4.9.1, 4.9.2 or 4.9.3. As a minimum, five elements shall be tested to failure at the following conditions:

a) At 25 °C.

b) At 25 °C after 1,000 hours at 150 °C in an air or nitrogen ambient. The average bond strength at each test condition shall be determined in kilograms (force).

4.9.1 Bond strength test procedure

The bond strength shall be determined in accordance with method 2019 of MIL-STD-883. A gold-metallized substrate or a gold- or nickel-plated package shall be used as the bonding surface for bond strength testing.

4.9.1.1 Type I materials

Suppliers shall use 0.08 inch-square (0.2 centimeter-square) gold-plated Kovar tabs.

4.9.1.2 Type II materials

Suppliers shall use 0.08 inch-square (0.2 centimeter-square) alumina chips.

4.9.2 Alternate bond strength procedure

The bond strength may be determined in accordance with ASTM D1002 as an alternative to test method 2019. If ASTM D1002 is used, the results must be correlated to assure that the bond strength of the adhesive is shown to be equivalent to the Method 2019 failure criteria.

4.9.3 Molding compounds or encapsulants

Molding compounds or encapsulants shall be tested in accordance with MIL-STD-883, test method 1034.

4.9.4 Reference guidance documents

Additional standard test methods can be found in literature such as:

ASTM D638, Tensile Properties of Plastics
ASTM D882, Tensile Properties of Thin Film Plastic Sheeting
ASTM D897, Tensile Properties of Adhesive Bonds
ASTM D1922, Propagation Tear Resistance of Plastic Film and Thin Sheeting by Pendulum Method

4.10 Coefficient of linear thermal expansion

The coefficient of linear thermal expansion shall be determined in accordance with ASTM D3386 over the temperature range of –65 °C to 150 °C. The glass transition temperature, coefficients, and temperature ranges corresponding to different slopes of the curve shall be noted.

4.11 Thermal conductivity

The thermal conductivity, in watt/meter-K, shall be determined at 121 °C ±5 °C in accordance with ASTM C177, ASTM C518, or ASTM E1461.

NOTE 1 cal/cm-s-K = 418.4 W/m-K.

4.12 Volume resistivity

4.12.1 Type I polymers

4.12.1.1 Paste materials

Test specimens shall be prepared using a standard 1 inch x 3 inch glass slide. A jig capable of holding this slide, with two scribed lines 100 mil apart and parallel to the length, shall be the guide for applying two strips of transparent tape. There shall be no wrinkles or bubbles in the tape. The slide shall be cleaned with alcohol and air dried. A drop of the type I material shall be placed between the two strips of tape. Using a single edge razor blade maintaining a 30° angle between the slide surface and the razor blade, the material shall be squeezed between the tape strips. The length of the applied strip shall be at least 2.5 inches. The tape shall be removed, and the material shall be cured according to 3.6.1. After cure, the test specimens shall be allowed to cool to room temperature.

4.12.1.2 Film materials

Test specimens shall be prepared using a standard 1 inch x 3 inch glass slide. The slide shall be cleaned with alcohol and air dried. A thin strip of the uncured film approximately 100 mil wide and at least 2.5 inches long shall be placed on the glass slide. The film shall be covered with a strip of copper foil or Teflon film and a second 1 inch x 3 inch glass slide shall be placed over the foil or Teflon film. Sufficient force (weight, clip, etc.), shall be applied to the assembly to compress the material during cure. The material shall be cured according to 3.6.1. After cure, the test specimen shall be allowed to cool to room temperature, and the top slide and foil or Teflon shall be removed. The exact width and thickness of each polymer strip shall be measured with a precision caliper and micrometer respectively. These measurements, after conversion to the appropriate units, shall be used to calculate the volume resistivity using the formula given in 4.12.1.3.

4.12.1 Type I polymers (cont'd)

4.12.1.3 Resistance measurements

Resistance measurements shall be made using a milliohm meter in conjunction with a special four-point probe test fixture. (This fixture can be made of an acrylic material with four spring-loaded contacts. The contacts must be set into the acrylic so that the current contacts are 2 inches apart, the voltage contacts are between the two current contacts, and the voltage contacts are separated from each current contact by 0.5 inch.) The four-point probe fixture shall be placed on the strip of conductive polymer and contact between each probe and the material shall be ensured. The measured resistance shall be recorded in ohms, and the resistivity shall be determined from the following formula:

$$P = R (w \times t) / l$$

where:

P = resistivity, ohm-m
R = measured resistance, ohms
W = width, (100 mil = 2.54 mm)
t = thickness, (micrometer reading of the material plus glass side) minus (micrometer reading of the glass slide)
l = length between inner pair of probes, (1 inch = 25.4 mm)

A minimum of three specimens shall be tested at 25 °C, at 60 °C, at 150 °C, and at 25 °C after 1,000 hours at 150 °C in an air or nitrogen ambient. The same specimens may be used for each test.

4.12.2 Type II polymer materials

Type II materials shall be tested in accordance with ASTM D257 at temperatures of 25 °C and 125 °C.

4.13 Dielectric constant

The dielectric constant of type II materials shall be determined as required in the user's material specification in accordance with ASTM D150 at frequencies of 1 kHz and 1 MHz at room temperature.

4.14 Dissipation factor

The dissipation factor of type II materials shall be determined as required in the user's material specification in accordance with ASTM D150 at frequencies of 1 kHz and 1 MHz at room temperature.

4.15 Sequential test environment

Testing shall be performed using either 4.15.1 or 4.15.2.

4.15.1 Sequential test environment

A minimum of five test specimens shall be subjected to the environmental conditions specified below. Specimens shall be prepared using the largest component/substrate/package combinations representative of end-use applications in backing material, attach surface, and size. Component types include resistor, capacitor, integrated circuit, and discrete semiconductor elements. Two components of each type shall be attached to the substrate with the adhesive (type I or II) proposed for use with that component type. The test specimens shall be subjected to the following environmental conditions in the sequence given:

a) Thermal shock (JESD22-A106, condition D, 15 cycles or MIL-STD-883, method 1011, condition C, 15 cycles).

b) Temperature cycling (JESD22-A104, condition C 100 cycles or MIL-STD-883, method 1010, condition C, 100 cycles).

c) Mechanical shock (JESD22-B104, condition B, Y1 only or MIL-STD-883, method 2002, condition B, Y1 only).

d) Variable frequency vibration (JESD22-B103, 20g peak acceleration, Y1 only or MIL-STD-883, method 2007, condition A, Y1 only).

e) Constant acceleration (MIL-STD-883, method 2001, condition B, Y1 only).

4.15.2 Alternate sequential testing

Alternative, testing in accordance with Qualification Testing (QML) sequences in accordance with MIL-PRF-38534, using maximum baseline limits may be performed. The user is still required to satisfy the requirements of 4.15.1 by completing the necessary supplemental testing, i.e., thermal shock and vibration.

Following the environmental exposures of 4.15.1 or 4.15.2, the test specimens shall be examined for possible degradation in accordance with MIL-STD-883, method 2017. For adhesives, one of each type of component from each sample shall be evaluated for die shear strength in accordance with MIL-STD-883, method 2019 and shall meet the strength requirements of figure 2019-4.

4.16 Density

The density of materials used as RF or microwave absorbers shall be determined in accordance with principles outlined in ASTM D972 or D3574. Those RF absorbers that are foamed in-place are to be foamed, cured, and cut to form the free standing material for this analysis.

4.17 Mechanical integrity

4.17.1 Getter integrity - short term

Samples shall be prepared using hermetically sealed packages representative of the maximum size and type which will incorporate the use of getter material. These samples will contain only "salted" particles and getter material. The getter material shall be applied to the package in the location and approximate volume as specified for a normal production part. The getter material coverage area shall be measured and recorded. The particles to be salted shall consist of the following unless otherwise agreed upon by the using activity.

1) Solder balls: 3-6 mils in diameter - 2 pieces required.
2) Aluminum ribbon: Approximate dimensions of 2 mil thick by 5 mil wide by 10 mil long - 1 required. A piece of aluminum wire 2 mil - 6 mil in diameter may be substituted for the ribbon.
3) Gold wire: 1 mil diameter by 15 mil - 20 mil in length - 1 piece required. Getter material application and cure shall take place in the sequence normally followed for production parts.

The samples shall be processed through the same environmental conditioning steps as a qualified production part. The samples shall be subjected to PIND test in accordance with JEDEC Publication JEP114 or MIL-STD-883, method 2020, condition A or B, which shall be repeated three times for a total of five cycles to verify the integrity of the getter material. During all PIND testing the samples shall be mounted on the tester such that the shock pulses integral with the test shall be in the direction most likely to dislodge the particles from the getter material. A minimum of three samples shall be evaluated and all shall pass the defined PIND criteria.

4.17.2 Getter integrity - long term

All of the conditions and requirements of 4.17.1 apply, except that the samples either newly prepared or as received from the short term test, shall be stored at 150 °C for 1,000 hours. The samples shall then be subjected to mechanical shock in accordance with JESD22-B104, condition B, Y1 or MIL-STD-883, method 2002, condition B, in the Y2 direction. Following mechanical shock the samples shall be PIND tested as specified above.

Following PIND, the samples shall be delidded and a visual inspection shall be performed to verify the following:

a) Determine if particles have separated from the getter material or have fallen into the package.
b) Determine if getter coverage has spread or bled out.
c) Check for any evidence of peeling from inside and/or getter becoming separated from package.

4.17 Mechanical integrity (cont'd)

4.17.3 Vibration

Samples shall be prepared as in 4.17.1 except that the lid shall be attached in such a manner that it may be removed for visual inspection. After particle salting and immobilization as in 4.17.1, visual inspection shall be done to verify entrapment of the salted particles. Location of the particles in the getter material shall be recorded for future reference.
The lid shall then be reattached to the package securely enough to withstand the testing that follows. After PIND testing in accordance with JEDEC Publication JEP114 or MIL-STD-883, method 2020, the samples shall be subjected to vibration in accordance with JESD22-B103, 20g or 50g peak acceleration or MIL-STD-883, method 2007, condition A or B. At the end of this test, the lids shall be removed from the package by whatever method is required. Location of the "salted" particles in the getter material shall be noted and compared with the location prior to vibration. Particles other than the original "salted" particles shall be ignored. A minimum of three samples shall be submitted for evaluation and all shall pass the defined PIND criteria initially and after vibration.

4.18 Operating life test

Ten electrically functioning samples shall be fabricated using hermetically sealed devices which have been processed through the same steps as a normally qualified production part as specified by the user's assembly drawing. Standard evaluation circuits may be substituted. All the samples shall meet the PIND test requirements in accordance with JEDEC Publication JEP114 or MIL-STD-883, method 2020, condition A or B. The samples shall be subjected to the life test in accordance with MIL-STD-883, method 1005, condition A, for 1,000 hours at 125°C. Electrical parameters shall be measured and recorded for the units initially and at the completion of the life test. Data taken from the samples shall be reviewed for evidence of device degradation due to the presence of getter material.

4.19 Test deviation

Additional, reduced or alternate testing, as may be dictated by the uniqueness of particular material and manufacturing construction techniques can be required or authorized by the using activity.

5 Summary

As a minimum, acquisition documents shall specify the following information:

a) Title, number, and revision letter of acquisition specification,

b) Size and number of containers required,

c) Manufacturer's product designation, and

d) Request for test data.

Annex A (informative) Differences between JESD72A and JESD72

This annex briefly describes most of the changes made to entries that appear in this publication, JESD72A, compared to its predecessor, JESD72 (June 2001). Some punctuation changes are not included.

Clause	Description of change
3.5.1	Added “be”.
4.8.3.3	3rd sentence: changed “…quantities > 5 ppm” to “…quantities > 50 ppm”.

Standard Improvement Form JEDEC _____

The purpose of this form is to provide the Technical Committees of JEDEC with input from the industry regarding usage of the subject standard. Individuals or companies are invited to submit comments to JEDEC. All comments will be collected and dispersed to the appropriate committee(s).

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1. I recommend changes to the following:

☐ Requirement, clause number _____

☐ Test method number _____ Clause number _____

The referenced clause number has proven to be:

☐ Unclear ☐ Too Rigid ☐ In Error

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