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IPC-TM-650
TEST METHODS MANUAL

Number	
2.3.26.2	
Subject	
Mobile Ion Content of Polymer Films	
Date	Revision
7/95	
Originating Task Group	
Deposited Dielectric Task Group (C-13a)	

1.0 Scope The purpose of this test is to determine the mobile ion content remaining in a polymeric dielectric film after curing.

2.0 Applicable Documents

None

3.0 Test Specimen

4.0 Apparatus or Material

- Spin (spray, extrusion, etc.) coating equipment
- Virgin Si monitor or production grade wafers (w/Au release coating if required for self-priming materials)
- Hot plate or convection oven drying apparatus
- Non-contact exposure tool (photosensitive compositions only)
- Curing furnace
- (4) 20ml capacity digestion bombs with PTFE liners
- Razor knife or blade
- Scissors
- ≥18 MR – CM deionized water
- Ion chromatograph

5.0 Procedure

5.1 General

5.1.1 This procedure assumes that the approximate weight of a cured coating of the material for a given wafer size and speed is known. If not, a spin speed curve and cured density or other cured film mass determination must be made. If the cured film mass in grams for the selected wafer size is mf, then N is (1/mf) rounded up to the nearest whole number.

5.1.2 The procedure further requires that films be prepared in such a way that they may be mechanically peeled from the wafer without excessive handling or damage. For materials that are not self-priming, this may simply mean omitting the use of an adhesion promoter. For self-priming materials, it may mean coating over a gold or other release layer on the surface of the wafer. Any release layer that cannot be guaranteed to be clean should not be used.

5.1.3 In order to obtain meaningful results, great care must be taken to assure that the films are not contaminated during their preparation. This requires that all wafers, etc., be handled with clean tweezers and gloves only and that all processing (coat, bake, exposure, cure) be done in a cleanroom that eliminates particulate accumulation on the film surface.

5.2 Test

5.2.1 Coat a layer of material onto 2xN wafer(s) at 2krpm for 30 seconds. This should be done in a cleanroom.

5.2.2 Softbake per the standard process for that material. This should be done in a cleanroom.

5.2.3 If the material is photosensitive and is negative working, blanket expose at a standard dose using a non-contact exposure method.

5.2.4 Cure all the wafers in a diffusion furnace or hot plate. This should be done in a cleanroom.

5.2.5 Tare two clean bomb assemblies to 0.01g (for bomb cleaning procedure see Section 6).

5.2.6 Peel the cured films from the wafers. Score the perimeter of the wafer with a clean razor blade if required. This should be done in a cleanroom.

5.2.7 Cut up the films using clean scissors and place N films in each of the bombs. This should be done in a cleanroom. Weigh the bombs to 0.01g and subtract the tares (see Section 4.5) to determine the exact sample mass in each bomb. Record these masses as ms1 and ms2.

5.2.8 Add approximately 10ml of DI water to the bombs and weigh the bombs to 0.01g. Subtract the weight from Section 4.7 to determine the mass of the water added. Record these masses as mw1 and mw2.

5.2.9 Seal the bombs and set aside.

5.2.10 Place approximately 10ml of DI water into each of two additional clean bombs. Seal the blank bombs.

5.2.11 Extract the films and the blank at 160°C for 20 hours in a box oven.

5.2.12 Remove the extract and analyze using an ion chromatograph. Calibrate and operate the instrument per the manufacturer's instruction and other procedures.

5.2.13 Adjust for the background as indicated by the blanks as follows:

$[I_e] = ([I_{s1}] + [I_{s2}]) - ([I_{b1}] + [I_{b2}])$

where:

$[I_e]$ = The average concentration of ion I in the extract due to the sample.

$[I_{s1}]$, $[I_{s2}]$ = The raw concentration of ion I in the extract from bombs 1 and 2.

$[I_{b1}]$, $[I_{b2}]$ = The raw concentration of ion I in the extract from blanks 1 and 2.

5.2.14 Correct the dilution during extraction to determine the levels of mobile ion species in the film:

$$[I_f] = [I_e] \times \frac{(m_{w1} + m_{w2})}{(m_{s1} + m_{s2})}$$

Where:

$[I_f]$ = The measured average concentration of ion I in the cured film.

5.3 Repeatability, Reproducibility, and Sensitivity

5.3.1 This method should give repeatability to ≤10% within a laboratory and reproducibility ≤20% between laboratories with proper calibration of instruments.

5.3.2 Ion chromatography should be run to give a nominal sensitivity of 0.01ppm in the extract at 0.1ppm in the film.

6.0 Notes — Bomb Cleaning Procedure

6.1 Method A

6.1.1 Add 40ml of acetone to the bomb. Replace the lid and shake by hand. This should loosen any residue. Rinse any remaining residue with additional acetone. After all the residue has been removed, rinse the container and lid an additional three times with acetone.

6.1.2 Rinse the container and lid three times with DI water.

6.1.3 Add 10ml to the container, seal, and extract at 160°C for 20 hours.

6.1.4 Empty the bomb and rinse three times with DI water.

6.1.5 Dry the container and lid for one hour in 100°C.

6.2 Method B

6.1.1 Add 40ml of acetone to the bomb. Replace the lid and shake by hand. This should loosen any residue. Rinse any remaining residue with additional acetone. After all the residue has been removed, rinse the container and lid an additional three times with acetone.

6.2.2 Fill vessel with 1:1 HNO_3 and let soak for several hours.

6.2.3 Empty vessel and rinse the container and lid three times with DI water.

6.2.4 Add 10ml to the container, seal, and extract at 160°C for 20 hours.

6.2.5 Empty the bomb and rinse three times with DI water.

6.2.6 Dry the container and lid for one hour in a 100°C oven.