

Polyimide Processing for PI-2600 series (not Photodefineable)

Standard Operating Procedure

Faculty Supervisor: Prof. Robert White, Mechanical Engineering (x72210)

Safety Office: Peter Nowak x73246 (Just dial this directly on any campus phone.)

(617)627-3246 (From off-campus or from a cell phone)

Tufts Emergency Medical Services are at x66911.

Revised: July 1, 2008

Goal:

Deposit and cure a non-photodefineable polyimide thin film.

Warnings:

The chemicals you are working with here are not acutely hazardous, however, the adhesion promoter (VM-651) is a severe skin, eye, and respiratory irritant. Thus, all processing (including hotplate bakes) must be done in the fume hood, as the solvent fumes released during processing have long-term health effects. The main thing is to not breathe any fumes, and not to get any polyimide or adhesion promoter in your eyes or on your skin.

1. Material Requirements:

1.1 Equipment and tools: Spin processor, photoresist oven, photoresist hotplates, wafer tweezers.

1.2 Chemicals: PI-2600 series polyimide, VM-651 adhesion promoter

1.2.1 PI-2600 series polyimides contain the solvent n-methyl-2-pyrrolidone (>60%) and the polyimide precursor s-biphenyldianhydride/p-phenylenediamine (10-30%)

1.2.2 VM-651 has proprietary contents.

1.2.3 Hazards associated with chemicals:

1.2.3.1 PI-2600 series polyimide: minor skin, eye and respiratory irritant. Not unusually flammable. Not a known carcinogen.

1.2.3.2 VM-651 adhesion promoter: Severe skin, eye, and respiratory irritant. Not unusually flammable. Not a known carcinogen.

1.3 Engineering Controls: Conduct procedures in the fume hood. Dispose of chemicals as described at the end of this document.

1.4 Personal Protective Equipment: Nitrile gloves and eye protection required for all procedures. When working in the fume hood, also wear trionic gloves, apron, and chemical goggles.

2.0 Procedure:

2.1 A small working quantity of polyimide should be transferred to an amber bottle for processing. The polyimide is kept in the freezer; you need to let it warm to room temperature, then pour a working quantity into a clean amber bottle for your use. The polyimide stock bottle should be returned to the freezer. The polyimide can be kept in the amber bottle at room temperature for about a month before properties will start to degrade. *Make sure you do this the day before so that air bubbles have time to come out of the polyimide!!* Make sure to label the bottle with name, date, and the material type.

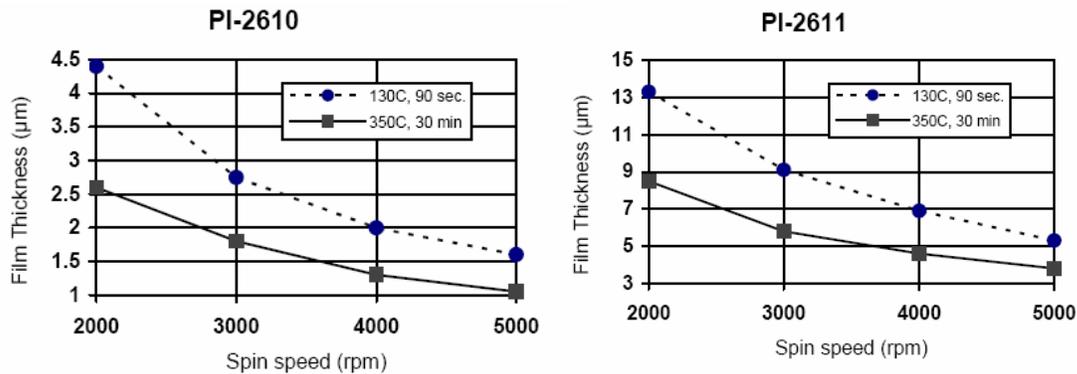
2.2 Similarly, a second amber bottle should be used to store a small working quantity of the adhesion promoter. Make sure to label the bottle with name, date, and VM-651.

2.3 Wafer should be clean and dry prior to starting processing. A Piranha clean (see Piranha clean SOP) is suggested. A dehydration bake at 200 °C in the hotplate or oven may also help.

2.4 Spin on the adhesion promoter. *Use the "dirty" spinner... the one designated for all materials.* (See Laurell Spinner SOP for instructions on using the spinner.):

2.4.1 In a small amber bottle, dilute the VM-651 adhesion promoter with DI water in a ratio of 100:1 by volume. This solution should not be stored, but should be made immediately before spinning on the polyimide.

- 2.4.2 Spin on the VM-651 adhesion promoter at 500 RPM for 4 seconds followed by 3000 RPM for 30 seconds.
- 2.4.3 Dispose of any excess adhesion promoter solution in the solvent waste bottle.
- 2.5 Spin on the polyimide. *Use the "dirty" spinner... the one designated for all materials.* (See Laurell Spinner SOP for instructions on using the spinner.)
 - 2.5.1 Pour a puddle of polyimide onto the wafer. Do not put too much on so it overflows and gets on the back of the wafer. Take care not to make any bubbles. Put a circular puddle in the center of the wafer.
 - 2.5.2 Allow the polyimide to settle for 10-30 seconds.
 - 2.5.3 Spread at 500 rpm for 5 sec
 - 2.5.4 Spin for 30 sec. Spin speed (RPM) needs to be determined based on which polyimide viscosity you have purchased, and what final thickness you require. Spin speed curves are available from the manufacturer. An example appears below (thickness is reported after soft bake and after cure).



- 2.6 Remove the wafer from the spinner using the wafer alignment tool.
- 2.7 Soft bake (should be conducted with the hotplates in the chemical hood). Bake at 200°C for 2 minutes.
- 2.8 Curing
 - 2.8.1 Curing must be performed in a nitrogen ambient on a hotplate or in a furnace.
 - 2.8.2 Ramp up 3°C/min to 350°C.
 - 2.8.3 Hold at 350°C for 30 minutes.
 - 2.8.4 Ramp down to room temperature at < 10°C/min.
 - 2.8.5 The furnace can be cleaned at 700 °C in air for 1 hour.
- 2.9 Stripping:
 - 2.9.1 Cured polyimide is difficult to strip.
 - 2.9.2 An O₂/CF₄ plasma (80/20% by flow rate), 300 mT, 400 W can remove the polyimide with etch rates on the order of 100 nm/min.
 - 2.9.3 A Piranha clean will attack the Polyimide but may not completely strip it.
- 2.10 Etching:
 - 2.10.1 Aluminum can be used as a hard mask for plasma etching the Polyimide.
 - 2.10.2 Sputter on the aluminum thin film on top of the cured polyimide (~ 1 micron thick) and pattern either by liftoff or by etching the aluminum.
 - 2.10.3 Etch the polyimide using a 20% CF₄, 80% O₂, 200 mT, 300 W plasma. Expect etch rates on the order of 100 nm/min.
 - 2.10.4 Strip the aluminum using a wet etch.

3.0 Cleanup:

- 3.1.1 Turn off the hotplates.
- 3.1.2 Dispose of any excess VM-651 adhesion promoter solution in the solvent waste bottle.
- 3.1.3 Clean any polyimide residue out of the spinner with fab wipes and dispose of in the photoresist/solvent waste bin.
- 3.1.4 Go through the Laurell Spinner clean procedure using a dummy wafer. See the Laurell SOP.

- 3.1.5 MAKE SURE NOT TO GET SOLVENT INTO THE VACUUM PATH OF THE SPINNER! ONLY SPRAY SOLVENT INTO THE SPINNER WHEN A WAFER IS ON THE CHUCK!
- 3.1.6 After the spinner bowl is all cleaned out (use fab wipes and solvent as needed), dump out the waste reservoir from the back of the spinner into the solvent waste bottle.
- 3.1.7 Clean off any residue from any tools or surfaces using acetone and isopropanol. Dispose of in the solvent/photoresist trash.
- 3.1.8 Remove the hotplates from the hood.

4.0 Storage:

- 4.1 VM-651 should be stored in the “solvent” cabinet.
- 4.2 The small amber “working” bottles of polyimide and VM-651 should be stored in the photoresist cabinet.
- 4.3 The larger stock bottle of polyimide should be stored in the freezer.

5.0 Waste Disposal:

- 5.1 Polyimide, and VM-651 waste:
 - 5.1.1 Solid waste should go in the solvent/photoresist trash.
 - 5.1.2 Liquid waste should go in the solvent/photoresist liquid waste bottle.

6.0 Accident Procedures:

- 6.1 Contact: Read MSDS prior to working with any chemical to familiarize yourself with the symptoms of exposure and recommendations for treatment.
 - 6.1.1 There are solvent fumes from the VM-651 and the polyimide. If you breath these fumes, you may feel dizzy. If this occurs, turn off the hotplates and leave everything in the hood. Leave the room and get some fresh air. If symptoms persist, contact Tufts health services and inform the lab directory and Tufts health and safety office.
 - 6.1.2 Skin contact from the chemicals may cause irritation and burning. For minor contact, exit the lab and rinse the affected area with water. For major contact (such as breaking the large bottle of VM-651 so it splashes all over you) get in the safety shower and remove the affected clothing. Have someone call emergency health services.
 - 6.1.3 Eye contact from either VM-651 or polyimide: Rinse well with water in eye wash for 15 minutes. **Contact Tufts Emergency Services x6-6911.**
 - 6.1.4 Ingestion: Do not induce vomiting. Immediately contact **Tufts Emergency Services x6-6911.**
- 6.2 Spill:
 - 6.2.1 If a small, contained spill occurs, such as inside the hood, wipe it up with chemical wipes and dispose of in the solvent trash container.
 - 6.2.2 If a large spill occurs that you are not comfortable cleaning up, such as breaking a bottle on the floor, evacuate the lab and contact Tufts emergency services (x66911).
- 6.3 Fire: There is low fire hazard associated with either the polyimide or the VM-651. Do not put them on a hotplate or in an oven when they are turned on. If a fire starts, remove any solvents from near the fire if it is safe to do so, and exit the lab. Do not try to fight the fire. Immediately contact Tufts emergency services once you are outside the lab at **Tufts Emergency Services at x66911.**

If at any time you feel a situation is dangerous, do not hesitate to call the safety office (x73246, Peter Nowak) or the faculty supervisor (x72210, Robert White). Please inform of the faculty supervisor and/or the health and safety office of any accident or health concern.

Report all accidents (injuries, major spills, fires) to the safety office at x73246 (Peter Nowak) and the faculty supervisor at x72210 (Robert White). For emergencies, call Tufts Emergency Services at x66911.