Negative Resist Processing for E-Beam Lithography using ma-N 2403

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.

For more information: www.microchem.com
The negative tone photoresist ma-N 2403 is used in both optical DUV lithography and electron beam lithography. ma-N 2403 exhibits - robust process latitudes, not chemically amplified, high structural resolution capability up to μm- and nm-range, no swelling in aqueous alkaline developers, excellent etch resistance to acidic and alkaline etch solutions when used as a resistant etch mask, high resistance to acidic and alkaline plating baths. Proper process optimization guarantees highly reproducible results

This SOP assumes you are already familiar with contact lithography.

Revised: January 30, 2018

1. Material Requirements:

1.1 Equipment: Spin coater, hotplate or oven, tweezers, several glass beakers, disposable pipets
1.2 Chemicals: ma-N 2403 resist (solution of 1-Methyl-2pyrrolidine, n-Butyl acetate & cyclohexanone), ma-D 532 developer (3% Tetramethylammonium hydroxide), Acetone
   Hazards associated with chemicals:
   ma-N 2403 resist is a combustible liquid and vapor. It causes irritation to eyes, nose, and respiratory tract. Prolonged, repeated contact, inhalation, ingestion, or absorption through the skin, may cause toxic effects to internal organ systems.
   ma-D 532 developer contains 3% Tetramethylammonium hydroxide (TMAH). Deaths have occurred due to skin exposure to 25% TMAH. At this concentration TMAH is corrosive and can cause burns, eye damage and target organ toxicity.
   Acetone is volatile, flammable solvents. Avoid heat sources. Do not breathe fumes. Conduct processing in the fume hood. Keep away from sparks and flames. In case of fire, use water spray, alcohol foam, dry chemical, or carbon dioxide.
   Engineering Controls: Conduct procedure in the fume hood. Dispose of chemicals as described in the end of this document.
1.3 Personal Protective Equipment: Nitrile gloves and eye protection for all procedures.
2.0 Procedure:

2.1 Wafer or substrate preparation
A preclean may be helpful - piranha (see Piranha clean SOP) or ultrasound acetone and IPA. Recall that substrates must be conductive so Si is recommended.

2.2 Dehydration bake your wafers at 200 C.
   2.2.1 Perform dehydration back on an aluminum foil topped hotplate (5 min) or in the convection oven (30 min).

2.3 Spin coating – Conduct in “Dirty” Spinner
   2.3.1 Using a disposable pipet, dispense resist (up to 8mL for a 100mm wafer)
   2.3.2 A 5 step spin is recommended
       4 sec ramp to 500rpm
       8 sec hold at 500rpm
       4 sec ramp to final speed
       45 sec hold at final speed
       4 sec ramp to 0rpm
   2.3.3 To determine final spin speed, consult MicroChem datasheets.

2.4 Pre Bake
   Hot plate: 90C for 60 - 90 seconds

2.5 Exposure:
   Using electron beam exposure (standard: 20 keV electrons) the dose D₀ amounts to 80 µC/cm². The usual exposure dose is 1.5 x D₀ for submicron patterning. Aspect ratio of 3 – 5 can be attained. ma-N 2400 shows a contrast of 3.0.

2.6 Development:
   Place sample in ma-D 532 for 45-60 seconds. A small amount of developer should be sufficient.
   Immediately transfer to water for 3-5min
   Dry with nitrogen gun

2.7 Remove resist
   Acetone or O₂-plasma are effective. Using appropriate glassware, rinse your substrates in DI water and blow dry.

3.0 Storage:
   3.1 Store the resist upright in original containers in the photoresist storage cabinet above 10C (50F). Do not refrigerate. Keep away from sources of ignition, light, heat, oxidants, acids, and reducers. Shelf life is 6 months from date of manufacture.
   3.2 Acetone is found in the solvent storage cabinet.
4.0 Waste Disposal:

**4.1 Solvent waste (Acetone, ma-N 2403 resist)**

4.1.1 Wipes are disposed of in the solvent trash can

4.1.2 Liquid waste and rinse water are collected in the solvent waste bottle.

**4.2 Alkaline/Developer waste (ma-D 532 developer)**

4.2.1 Wipes are disposed of in the Acid/Base trash can

4.2.2 Liquid waste and rinse water are collected in the alkaline/developer waste bottle.

5.0 Accident Procedures:

**5.1 Contact:** Read MSDS prior to working with any chemical to familiarize yourself with the symptoms of exposure and recommendations for treatment.

5.1.1 Skin contact: Rinse for 15 minutes with water. Contact Tufts health services and inform the lab directory and Tufts health and safety office.

5.1.2 Eye contact: Rinse for 15 minutes with water. Contact Tufts health services and inform the lab directory and Tufts health and safety office.

5.1.3 Ingestion: Do not induce vomiting, gently wipe or rinse inside of mouth with water. Contact Tufts health services and inform the lab directory and Tufts health and safety office.

5.1.4 Inhalation: Remove to fresh air. Contact Tufts health services and inform the lab directory and Tufts health and safety office.

**5.2 Spill:**

5.2.1 If a small, contained spill occurs, such as inside the hood, wipe it up with chemical wipes and dispose of in the appropriate trash container.

5.2.2 If a large spill occurs that you are not comfortable cleaning up: evacuate the lab and contact Tufts emergency services (x66911). Notify the faculty advisor.

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).

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.