A Guide to the Spin-Coaters and Photo-Chemicals in FAB.nano

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Abstract— **We accommodate a wide range of spin-coated materials at FAB.nano, from standard photo- and electron-beam lithography resists to non-photosensitive polymers. This document summarizes the spin-coating capabilities at FAB.nano, and provides guidance to researchers on which spin-coaters to choose for a given type of material. We also provide a summary of lab-supplied photo-chemicals and describe the process of user-specific specialty chemicals for situations when the needs go beyond what is standard lab supplied.**

I. INTRODUCTION

SPIN-COATING is the primary mechanism by which to apply photoresists and non-photosensitive polymers as thin-film onto substrates. The choice of material depends on the process needs: film thickness, exposure mechanism, etch selectivity, minimum feature-size, materials compatibility of developer, ease of removal, or whether the film is part of the final-device structure. The details of spin-coating and material-specific recipes will be discussed in a separate document. Here, we focus on choosing a compatible spin-coater and photo-chemicals for a given lithography process.

It is important to choose the correct coater for a specific material to coat. Residues of incompatible materials can contaminate other researchers' samples, and improper cleaning or incompatible waste streams may precipitate out sticky insoluble materials (e.g. when mixing *PMGI* and acetone). Such byproducts contaminate the coater bowl and will clog up the internal waste lines of the spin-coater. This leads to accelerated degradation and coater damage; improper cleaning can also lead to resist seeping into mechanical components, resulting in poor vacuum, damage to axes/motors, and a vacuum chuck that is very difficult to remove.

By making the right choices and following these guidelines, we can contribute to the longevity of the spin-coaters and minimize avoidable downtime caused by misuse.

We tried to minimize MIT and lab-specific references, and only included it where it was needed for clarity. The spinner names we refer to are of course FAB.nano specific/internal, and necessary to be included. However, other information, particularly on choices between different materials or chemicals, is universal and of use beyond FAB.nano.

II. SUBSTRATE PREPARATION

Proper substrate preparation is an important requirement for successful thin-film spinning and patterning, and requires the removal of particles and contaminants, as well as modifying the substrate's surface to help improve resist application.

A. *Substrate Cleaning*

Contamination with larger particles is easily noticed through "comet-streaks" in the spin-coated resist (especially in thinner resists). These streaks are often too thick to properly expose or develop. Contamination with smaller particles cannot be as readily observed, but will cause defects by interfering with the exposure or development around the contaminant.

To remove contamination from substrates, corrosive cleans (such as RCA or piranha) are the preferred procedure [1], [2]. However, the substrate materials must be able to withstand the corrosive chemicals. If the substrate is contaminated with organic thin film residues (e.g. from previous lithography steps), both the RCA and piranha cleans offer an effective approach for removal. RCA and piranha cleaning can either be done in the general-purpose acid hoods, or in dedicated wet benches with standing baths. Oxygen plasma ashing (*Asher-Barrel-Thierry*) is a good alternative for removing organic residues. However, ashing does not remove particles. Ashing steps may require optimization and inspection of the substrate when developing a process (e.g. with FTIR, ellipsometry or contact angle measurements).

In situations where the substrate materials are unable to withstand the corrosive RCA or piranha cleaning chemicals, a solvent clean (often using a sonicator) can be an alternative for particle or organic film removal, although it is often less effective. The order of solvents used is particularly important. For example, acetone leaves residue contamination, and should not be used as the final solvent in a cleaning sequence.

A common mistake is to solvent-clean brand-new wafers (fresh out of the box), which typically have neither particle nor organic film contamination – and which are likely to pick up contamination and residues from the solvent clean.

Megasonic cleaning with DI water offers an additional alternative for removing particle contaminants [3]. We provide a DI megasonic cleaning tank in the *Solvent-Clean-U06* hood.

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TABLE I
FAB.NANO OVERVIEW OF AVAILABLE SPIN-COATERS AND THEIR PROPERTIES

MIT.nano Tool Name	Location & Associated Hood	Alternative	Waste Stream / Compatibility	Intended for…
Spinner-Resist-U12	12U *General-Spinner-Hood*	*Spinner-Resist-L10*	Acetone	Standard photoresists, EBL resists, approved personal resists
Spinner-Resist-L10	10L *PhotoPrep-L10*	*Spinner-Resist-U12*	Acetone	Standard photoresists, EBL resists, approved personal resists
Spinner-Resist-Dispense-U10	10U *Resist-Spinner-Hood*	*Spinner-Resist-U12*	Acetone EBR	Fab.nano baseline/standard photoresists only (AZ3312, AZ nLOF, AZ 10XT)
CoatDevelop-picoTrack	8L *Dedicated enclosure*	*Spinner-Resist-Dispense-U10*	Acetone EBR	Automated coater/developer track for wafers and standard thin resists (AZ3312, AZ nLOF). 6” and 8” wafers only.
Spinner-PMGI-U12	12U *General-Spinner-Hood*	*Spinner-Polymer*	Foil Lining (1)	Only spinner for PMGI or LOR
Spinner-EBL	6U (white light) *EBL-Spinner-Hood*	*Spinner-Resist-L10*	Acetone	Electron beam lithography (EBL) resists that are not photosensitive (e.g. PMMA, HSQ)
Spinner-Polymer	4U (white light) *TBD*	-	Bowl Liner (2)	Coating polymers that are not photosensitive (e.g. PI2500 series polyimide, PU, BCB) and approved nanoparticle suspensions
Spinner-SU8	24U *SU8-Spinner-Hood*	-	PGMEA	All negative epoxy resists (e.g. SU8, mr-DWL) and soft lithography

(1): This spinner has no plumbed waste collection system, and Al foil is used to line the bowl and collect waste. Cleaning the lid and bowl can be done with EBR and fab-wipes. Refer to the system SOP for details.
(2): Cleaning procedure is determined on a case-by-case basis, depending on the polymer being spun. A bowl-liner is used to collect and then transfer the waste, following by cleaning of the bowl with a compatible solvent (again, determined on a case-by-case basis). Refer to the system SOP for details.

TABLE II - PART 1 OF 2
SPIN-COATERS AND PHOTORESIST COMPATIBILITIES, FOR STANDARD AND E-BEAM LITHOGRAPHY RESISTS

MIT.nano Tool Name	AZ3312	XT-10	nLOF	PMGI / LOR (2)	Approved User Photoresists (3)	HSQ	PMMA, ZEP, CSAR	ma-N	ESpacer DisCharge	Approved User EBeam Resists
Spinner-Resist-U12	Yes	Yes	Yes	-	Yes (2)	Yes	Yes	Yes	Yes	Yes (2)
Spinner-Resist-L10	Yes	Yes	Yes	-	Yes (2)	Yes	Yes	Yes	Yes	Yes (2)
Spinner-Resist-Dispense-U10	Yes	Yes	Yes	-	Yes (2)	-	-	-	-	-
CoatDevelop-picoTrack	Yes	-	Yes	-	Yes (2)(4)	-	-	-	-	-
Spinner-EBL	-	-	-	-	-	Yes (5)	Yes	-	Yes	Yes (2)
Spinner-PMGI-U12	-	-	-	Yes	-	-	-	-	-	-
Spinner-SU8	(1)	(1)	(1)	-	(1)(2)	-	-	-	-	-
Spinner-Polymer	-	-	-	Yes	-	-	-	-	-	-

TABLE II - PART 2 OF 2
SPIN-COATERS AND PHOTORESIST COMPATIBILITIES, FOR STANDARD AND E-BEAM LITHOGRAPHY RESISTS

MIT.nano Tool Name	SU8 Series	mr-DWL	Approved User SU8-like Resists	Approved User Nanomaterials (5)	Polyimide (Non-Photo)	Polyimide (Photosensitive)	BCB	Approved User Polymers (5)
Spinner-Resist-U12	-	-	-	-	-	-	-	-
Spinner-Resist-L10	-	-	-	-	-	-	-	-
Spinner-Resist-Dispense-U10	-	-	-	-	-	-	-	-
CoatDevelop-picoTrack	-	-	-	-	-	-	-	-
Spinner-EBL	-	-	-	-	-	-	-	-
Spinner-PMGI-U12	-	-	-	-	Yes	Yes	-	-
Spinner-SU8	Yes	Yes	Yes	-	-	Yes (2)	-	-
Spinner-Polymer	-	-	-	Yes	Yes	-	Yes	Yes

(1): Only for specifically approved cases where regular resist must be spun on top of SU8 (and the proximity is important to the work).
(2): With adequate precautions to avoid incompatible waste-stream and clogging of system. See staff for training.
(3): The PTC approval will specify the coater to be used for your resist
(4): With syringe dispense system, see staff for training
(5): Preferred option

B. *Surface Preparation*

Once the substrate is clean, its surface needs to be made compatible (primed) for the resist. The goal of priming is to temporarily enhance the compatibility of the substrate for a coated resist. Different resist materials may require different priming approaches. Incompatibilities between surfaces and resist can cause peeling and resist failure during develop or subsequent etches. A primed surface's properties are time sensitive, and therefore priming should take place shortly before spin-coating.

The standard procedure for hydrophilic Si and SiO_2 surfaces is to start with a de-hydration bake, followed by the application of *HMDS* to create a partially hydrophobic surface that acts as a barrier to polar liquids at the surface-resist interface, thereby preventing resist undercut during development [4]. The *HMDS* oven (*HMDS-Yes-U10*) accomplishes both the de-hydration and hydrophobic modification at the same time. Surface conditions can be checked by observing the contact angle of a water drop (a hydrophobic surface is desired for coating resist).

The benefit of *HMDS* may not extend well to non-Si materials or non-standard photoresists, and the combination of resist and substrate materials has to be carefully considered when choosing a surface preparation method. For example, to improve adhesion to III-V semiconductor surfaces, *SurPass 3000* is used with *HSQ*, while other resists on III-V surfaces may use *SurPass 4000* [5].

III. SPIN-COATERS

A. *Equipment Overview*

The spin-coaters in FAB.nano are summarized in Table I with their capabilities and intended uses. With the exception of the automated coater-track and the *PMGI* spinner, all of the spinners are Brewer CBX or CEE Apogee manual spin-coaters that share similar basic hardware and user interfaces, though some spinners have added features such as automatic dispense. Having multiple spin-coaters provides backup capabilities and helps reduce bottlenecks for users. It also allows us to separate materials that may have incompatible waste streams or cleanliness requirements.

Table II summarizes the types of resists and maps them onto the spin-coaters they can be used on. We differentiate primarily by waste-stream compatibility, with the main differentiation between "normal" photoresists and often more viscous epoxy-negative resists (e.g. *SU8*, *mr-DWL*), polyimide, and "general" or "exploratory" polymers and nanomaterials. More details about each resist are provided in the chemical section.

B. *General Resist Spin-Coaters*

The broadest-use spin-coaters are the manual "***Spinner-Resist-U12***" and "***Spinner-Resist-L10***". They do not have auto-dispense mechanisms, and can accommodate pieces to 8" wafers. Photoresist is pipetted onto the sample, with our baseline resists stored in the hood. Approved user resists are stored in the cabinet below the hood.

Waste is collected and routed to a carboy behind the hood. We use acetone to clean the bowl from resist residues. Although acetone has a greater fire hazard than other solvents, for manual cleaning it is preferred due to a lower health hazard. Any resist used must therefore be compatible with an acetone waste-stream. As summarized in Table II: *PMGI*, thick polymers (e.g. polyimide, *BCB*), custom polymers, and negative epoxies (e.g. *SU8* or *mr-DWL*) are not permitted. They have dedicated spin-coater choices, instead.

Electron-beam lithography (EBL) resists may be spun on the *AllPurpose* spin-coaters, though the dedicated *Spinner-EBL* is our preferred spin-coater for some EBL resists (see Table III).

C. *Automated Dispense Spin-Coaters*

For large samples (particularly wafers), we offer automated dispense spin-coating of three baseline resists: thin, positive ***AZ 3312***; negative, liftoff ***AZ nLOF 2020***; and thick, positive ***AZ 10XT 520cP***. Automated resist-dispense and develop can help improve spin homogeneity, repeatability and film-quality on wafers, reduce chemical waste, minimize direct handling of chemicals, and provide faster throughput for multiple wafers.

The ***Spinner-Resist-Dispense-U10*** is a manual spin-coater with these three resists plumbed for automatic dispense controlled by the software of the spin-coater. This also simplifies dynamic dispense (the application of photoresist while wafer is spinning at a low speed). The spinner's purpose and strength is to apply the three standard resists (Table III) onto wafers. For pieces or user-specific resists, one of the manual spin-coaters should be used.

Spin-coating produces excellent film uniformity except at the very edge of the sample, where resist can form a thicker "edge bead". Resist can also seep to the backside, and splashes from the coater bowl can end up on the backside as well. Both the edge bead and backside contamination can be a problem in subsequent process steps (contaminating hotplates, creating flatness errors during exposure). Backside edge-bead removal (EBR) sprays a solvent (e.g. *EBR 70/30*) from the bottom, to eliminate resist residues on the wafer backside. Because the EBR solvent can seep around to the wafer frontside near the edge, it also reduces the edge-bead. Resist quality assessment using profilometry (e.g. stylus Dektak, AFM) or optical methods (reflectometry or ellipsometry) can help characterize the resist profile and surface topography, and quantify the edge-bead.

The ***CoatDevelop-picoTrack*** is a fully automated coat-develop system, restricted to 6" and 8" wafers because of the robot handling and automation. Its main strength is high throughput and process repeatability (particularly when developing). We provide the two thin baseline resists: thin, positive *AZ 3312*; and negative, liftoff *AZ nLOF 2020*. A syringe-based manual dispense option is available for approved user-resists and coatings. Thick resist coating (*AZ 10XT 520cP*) is currently not supported, because the viscosity is too high for the system's resist pump. Even when running a small number of wafers, the *CoatDevelop-picoTrack* offers throughput advantages, because it contains in-situ vapor-prime modules for HMDS application.

TABLE III

Fab.nano Overview of Lab-Supplied Resist Materials and Their Properties

Name	Thickness Range in µm (1)	Type	Carrier Solvent	Recommended Removal (2)	Developer	Application
Standard UV Photoresists						
AZ 3312	1.5	Positive	PGMEA	Acetone	0.26N TMAH (AZ300/726MIF)	Baseline "Thin Resist"
AZ nLOF 2020	2.0	Negative	PGMEA	Acetone	0.26N TMAH (AZ300/726MIF)	Baseline "Liftoff Resist"
AZ nLOF 2035	3.5					
AZ 10XT 520cP	12	Positive	PGMEA	Acetone	0.35N TMAH (AZ 435MIF)	Baseline "Thick Resist"
Non-Photosensitive Films						
PMGI SF5	1.5	Not Photosensitive	Cyclopentanone	NMP	0.26N TMAH (AZ300/726MIF)	Used for bilayer liftoff Incompatible with acetone
Thick Negative Epoxy Resists for Soft Lithography						
SU8 2000.5	0.5	Negative	Cyclopentanone	PGMEA	PGMEA and IPA rinse	Thick epoxy resist for soft lithography. On MLA-150 use 375 nm laser. Can also be exposed using e-beam lithography.
SU8 2002	2					
SU8 2005	5					
SU8 2010	10					
SU8 2025	25					
SU8 2050	50					
SU8 2100	100					
SU8 2150	150					
mr-DWL5	5	Negative	GBL	PGMEA	PGMEA and IPA rinse	Similar to SU8 but more expensive Formulated for MLA-150 at 405 nm
mr-DWL40	40					
Electron Beam Lithography Resists						
PMMA 950A2	50 – 100 nm	Positive	Anisole	Acetone	3:1 IPA:MIBK	
PMMA 950A4	200 – 400 nm					
PMMA 950A6	300 – 600 nm					
PMMA 950A8	400 – 1000 nm					
PMMA 950A11	2 – 5 µm					
PMMA 495A4	200 – 400 nm	Positive	Anisole	Acetone	3:1 IPA:MIBK	Used for bilayer liftoff
ZEP 520A	300 – 700 nm	Positive	Anisole	NMP	o-Xylene	Much more expensive than PMMA, may have improved RIE performance
CSAR-62 AR-P 6200.04	80 nm	Positive	Anisole	NMP	o-Xylene	Lower-cost alternative to ZEP, to provide improved RIE performance
CSAR-62 AR-P 6200.09	200 nm					
CSAR-62 AR-P 6200.18	800 nm					
HSQ 2%	30 – 50 nm	Negative	MIBK	Hydrofluoric Acid	25% TMAH, Salty Developer	Dry etching and very fine features
HSQ 4%	60 – 80 nm					
HSQ 6%	100 – 130 nm					
ma-N 2401	100 nm	Negative	Cyclopentanone & Anisole	Acetone	0.26N TMAH (AZ300/726MIF)	Faster and lower cost than HSQ
ma-N 2403	300 nm					
Electra AR-PC 5090.02	42 nm	Anti-Charging	IPA	Water	Water	Add-on film for EBeam Lithography to reduce substrate charging

(1): Nominal thickness or thickness range based on data sheet, or (if in *italic*) as measured on our manual spin coaters at 1000 rpm and 4000 rpm.

(2): While acetone removes resist, an IPA rinse is necessary because resist residues remain in the acetone and can remain on the sample if only acetone is used. The IPA (or sometimes Methanol before IPA) rinse prevents that. More details will be covered in a lab insight paper on sample cleaning.

Chemical Acronyms: PGMEA also known as PM Acetate (Propylene glycol methyl ether acetate, or 1-Methoxy-2-propanol acetate), NMP (1-Methyl-2-pyrrolidone), TMAH (Tetramethylammonium hydroxide), IPA (Isopropyl alcohol, 2-propanol, or isopropanol), PMGI (polydimethylglutarimide), MIBK (Methyl isobutyl ketone), GBL (Gamma Butyrolactone)

D. Automated Developers

In addition to manual development, we offer two automated developer systems. The ***CoatDevelop-picoTrack*** can process 6” or 8” wafers, with spray and puddle recipes and metal-ion-free (MIF) developers. Thick resist can be developed with an appropriate puddle recipe. For more flexibility of substrates (e.g. large pieces or other wafers sizes), the ***Develop-Brewer-200CBX*** is an automated dispense/rinse system to develop photoresists with our standard 0.26N MIF developer. Small pieces or wafers with backside patterns should not be developed on this tool because the developer can seep around the backside and into the vacuum line, causing tool problems and damage.

E. Specialty: Electron-Beam Lithography Spin-Coater

Resists for electron-beam lithography can be spun on the ***Spinner-EBL***, a dedicated spin-coater for electron beam resists that are not light sensitive (e.g. *HSQ*, *PMMA*). As a dedicated spinner for those resists, the Spinner-EBL has the advantage of being readily available to EBL users. We recommend the *Spinner-EBL* for *HSQ* because the *HSQ* freezer is immediately next to it. Because *HSQ* can lead to SiO_2 residue/particulates, and the carrier solvent MIBK evaporates very quickly, the spinner bowl should always be lined with Al foil when spinning *HSQ*.

F. Specialty: PMGI & LOR Spin-Coater

An alternative to liftoff using *nLOF* negative resist is a dual-layer processes with ***PMGI*** or ***LOR*** as the bottom layer. The benefit of the dual-layer approach is that the top layer can be standard positive thin resist (*AZ 3312*), while the bottom layer is not photosensitive and formulated with different solvents so that there is no cross-dissolution when spinning the top layer. The bottom layer then is “wet-etched” during the develop step, with the undercut profile determined by the length of time the *PMGI / LOR* is exposed to developer.

One important consideration is that *PMGI / LOR* are incompatible with the acetone waste stream generated in our regular spin-coaters. Mixing of *PMGI / LOR* with acetone results in a sticky precipitate, which clogs up the waste lines and quickly damages the spin-coaters (Fig. 1). Care must be taken to separate *PMGI* and *LOR* from the *Spinner-Resist-U12* and its Acetone waste stream.

Fig. 1: Photographs showing a clogged spin-coater drain line. PMGI was mixed with acetone and precipitated, fully clogging the drain. This problem occurred in just a few weeks.

Therefore, all *PMGI* and *LOR* films must be spun in the ***Spinner-PMGI-U12***, co-located in the same hood in U12 as the *Spinner-Resist-U12*. The *Spinner-PMGI-U12* helps us separate the waste-streams, avoid incompatibilities, and prevents equipment damage. Waste is collected by lining the spinner bowl with foil and EBR can be used to clean the lid and bowl as needed. Co-locating it allows users to more conveniently spin both layers, switching coaters without having to switch hoods.

For 8” wafers, or as an alternative, the *Spinner-Polymer* is the back-up spinner for *PMGI/LOR*. The *Spinner-Polymer* uses a bowl-liner to help collect waste for each run.

G. SU8 and Soft-Lithography

The 24U lab space provides a full soft-lithography workflow to create *SU8* molds and *PDMS* replicas. The ***Spinner-SU8*** is dedicated for *SU8* and similar photosensitive viscous polymers compatible with the *SU8/PGMEA/Isopropyl Alcohol* waste-stream. *Acetone* should not be used in the spinner, to avoid incompatibilities and formation of precipitates that can clog the waste lines.

The *Spinner-SU8* has plumbed *PGMEA* developer and Isopropyl Alcohol topside rinse for *SU8* processing. The spinner has additional auto-dispense capabilities for a low-viscosity *SU8* (e.g. up to 5 μm thickness) that can be deployed when demand requires.

H. General Polymers and Nanomaterials

The ***Spinner-Polymer*** is a dedicated spin-coater for non-photosensitive polymers, nanomaterials, and other non-standard FAB.nano materials. In general, spinnable polymers such as *PEDOT:PSS, BCB, Polyimide, PDMS, Polyurethane* and other approved materials (e.g. *PZT*) can be spun on the *Spinner-Polymer*. Furthermore, materials with nanoparticles in suspension can be spun in the *Spinner-Polymer*. *PMGI* and *LOR* resists can also be used in *Spinner-Polymer*, particularly for 8” wafers. Because of the range of different materials spun on this coater the bowl is lined and waste is locally collected by the users after each use. The wide range of processes applied at this spin-coater may lead to cross-contamination. Careful cleaning of the bowl liner after each use, and attention to the waste stream are important steps to avoid creating insoluble precipitates in the coater, and assuring process safety and repeatability.

Many of the non-standard FAB.nano materials may require refrigeration during storage. Failure to let the solution warm up can lead to temperature mismatches between the substrate and the solution. These mismatches can result in particle aggregation or thermal cracking upon annealing and subsequent delamination of the spin-coated layer.

Because the *Spinner-Polymer* accommodates the broadest range of chemicals and solvents, and because of the potential presence of nanomaterials from previous users, there are unique safety concerns that need to be addressed. A strong understanding of the chemical safety for the material to be used on the *Spinner-Polymer* is very important, and users should discuss with staff the specific safety procedures on this coater to ensure cleaning and waste handling are properly addressed for their materials.

IV. LAB-SUPPLIED PHOTO-CHEMICALS

This section provides a more in-depth overview of all the FAB.nano supplied resists. Photo-chemicals that are not provided by FAB.nano can be requested as user-supplied chemicals, outlined in Section V.

Table III shows all of the photoresists that FAB.nano supplies. There are four broad categories: standard (baseline) resists, resists with acetone-incompatible waste-streams (*PMGI*), Soft-Lithography/*SU8*, and electron-beam lithography (EBL) resists.

A. Standard Resists

Our thin baseline resists, ***AZ 3312***, is a positive photoresist that spins films of about 1 to 2 µm thickness. The preferred wavelength for MLA-150 exposure is 405 nm (375 nm is an alternative, but that laser has lower power, and the MLA optics are optimized for 405 nm). Exposed resist can be developed in either *AZ 300 MIF* and *AZ 726 MIF* developers (see section IV on developers). *AZ 3312* resist is easily removed by acetone, and is the recommended resist to protect wafers during die-sawing.

If thicker films are required (e.g. for long dry etching or for DRIE), ***AZ 10XT 520cP*** enables 8 to 15 µm thick positive photoresist. Similar to the thin *AZ 3312* resist, *AZ 10XT* is exposed using 405 nm on the MLA-150, with higher dosages required (due to the thickness). Development time in the standard 0.26N MIF developers can exceed 10 minutes, so a higher concentration developer such as *AZ 435 MIF* is recommended.

B. Single-Layer Liftoff Resists

An undercut resist profile is important to enable liftoff deposition. Standard positive resist cannot create an undercut, and instead the inverse image of what a standard positive resist can deliver is required. With a single layer of resist, this can be achieved in two ways: either by using a negative resist, or through an image reversal technique [6].

We offer a chemically amplified negative resists, ***AZ nLOF 2020 and AZ nLOF 2035***. They offer a range of ~1 to 4 µm (*nLOF 2020*) and ~3 to 6 µm (*nLOF 2035*) in thickness and are suitable for liftoff of thicker films, because significant undercut profiles can be achieved. However, as a chemically amplified resist, process conditions for *nLOF* are more stringent, and less forgiving than standard positive resists. For example, a post-exposure bake is required for *nLOF* to work, and dose/defocus conditions for MLA exposure can have a significant impact. Running a dose-exposure matrix, and (if using pieces) choosing the optical autofocus on the MLA, are important for successful results. For *nLOF*, we recommend the 375 nm laser if using the MLA-150. Development of *nLOF* is similar to *AZ 3312*, using either *AZ 300 MIF* or *AZ 726 MIF*.

Image reversal achieves a similar outcome as negative resists, but instead uses a positive resist and adds an image reversal step: The photoactive compound (PAC) of positive resist transitions, upon UV exposure, from an insoluble into a soluble chemical. The image reversal step then further modifies this chemical (and without changing the unexposed PAC). Once modified, a flood exposure is conducted, which renders all unexposed resist soluble. The previously exposed and image reversed resist, however, remains insoluble.

Dedicated image reversal resist contains thermally activated additives, but image reversal can also be achieved with standard positive resists such as ***AZ 3312***, because an ammonia (NH_3) atmosphere results in the desired chemical steps, and generates an image reversal behavior. The *HMDS* oven (*HMDS-Yes-U10*) is able to carry out this image reversal NH_3-bake procedure. Development is the same as for regular *AZ 3312*, using either *AZ 300 MIF* or *AZ 726 MIF*.

C. Dual-Layer Liftoff Resists

While single-layer liftoff resists provide a good choice for many applications, on substrates with a greater degree of variability, users often prefer to avoid careful process calibrations and instead use a dual-layer liftoff process using a stack of *PMGI* and *AZ 3312*: a thin layer of ***PMGI SF5*** is first spun onto the substrate and baked, followed by a layer of *AZ 3312* (or any other thin photoresist). *PMGI* is not photosensitive, but slowly dissolved (“etched”) in *AZ 300 MIF* or *AZ 726 MIF* developer. Therefore, after exposing and developing the *AZ 3312* top layer resist, any additional time in the developer will remove the *PMGI* and create the desired undercut profile for liftoff. The amount of undercut is determined by time.

Because of waste-stream incompatibilities, *PMGI* (or its thicker variety, *LOR*) cannot be spun on the same coaters as the regular photoresist. Only the *Spinner-PMGI-U12* or the *Spinner-Polymer* can be used for coating of *PMGI* and *LOR*.

D. SU8 and Soft-Lithography

The primary use of ***SU8*** is in soft-lithography applications, where *PDMS* is poured over an *SU8* mold to create a replica that forms microfluidic channels. We supply a broad range of dilutions, for thicknesses spanning from 0.5 to 150 µm.

A benefit of hard-baked *SU8* is that it is much harder to remove than regular photoresist. Thus, *SU8* occasionally finds use as a mask in regular processes, particularly when it is not meant to be removed and instead is meant to be part of the final device.

Exposure of *SU8* on the MLA-150 requires the 375 nm laser as it is insensitive at 405 nm. However, the *mr-DWL* series of resist offers an *SU8*-like product that is designed to be used with the MLA-150’s 405 nm laser so that a much lower exposure dosage is required.

SU8 can also be exposed with electron-beam lithography, and is very sensitive to electron exposure (i.e. low dosage). While not a very common EBL resist, it finds niche applications particularly in situations where it is meant to be part of the final device (often by overcoating it with another material).

A challenge when processing thicker *SU8* films is its high viscosity, which makes it more difficult to cleanly dispense and pick up wafers after coating. Extreme care must be taken to avoid contaminated gloves from subsequently contaminating the rest of the lab. Changing gloves after dispensing or picking up wafer after coating is an important precaution to take. Uncured *SU8* on the back-side of a wafer is a common mode of contamination, as well as a source of poor vacuum and exposure difficulties on the MLA-150. Ensuring a clean wafer backside (using *PGMEA* to dissolve contaminants, or a razor blade to scrape off baked on *SU8*, or applying blue-tape as a protective film on the backside) is important before proceeding to other parts of the lab. We use the dedicated *Spinner-SU8* to help further reduce the chances of cross-contamination.

Many of these precautions also apply to other user-supplied high-viscosity materials (e.g. polyimide).

E. Electron-Beam Lithography Resists

We provide several common electron-beam lithography (EBL) resists for dry etching and liftoff. ***PMMA*** is a positive EBL resist that is not sensitive to white light. The 950 molecular weight series of *PMMA* allows film thicknesses of 100 nm to about 1 µm. Even thinner films can be achieved by diluting the *950A2 PMMA* with the solvent *anisole*.

PMMA is commonly developed in *3:1 IPA:MIBK* and we stock a pre-mixed ready-to-use solution. Because mixing IPA and MIBK is endothermic, a pre-mixed solution eliminates the wait time for temperature stabilization. It also helps reduce waste caused by mixing up too much. For PMMA, a range of development approaches exist, aiming at improving contrast and/or reducing exposure dose. For example, a mixture of *IPA:DI Water* offers high-contrast development of PMMA [7].

For liftoff, a dual-layer can help provide a better undercut profile. As the bottom layer, the lab-supplied 495 molecular weight *PMMA* (***495A4***) is used, while the top layer is from the ***950 PMMA*** series. The benefit is that for a given dose, the *495 PMMA*'s shorter chains mean that it is more exposed than it's *950 PMMA* counterpart. Thus, the bottom layer of *495 PMMA* will develop out more than the top *950 PMMA* layer, creating the desired liftoff undercut.

PMMA may not offer enough chemical resistance for selectivity in dry etching (especially in chlorine chemistry). Instead, ***ZEP 520A*** provides a more chemically resistant EBL resist, along with higher sensitivity (i.e. lower dose time). However, the high cost of ZEP is unfortunate, and we also provide ***CSAR-62 AR-P 6200.04*** as a somewhat more economical alternative to *ZEP*. Both *ZEP* and *CSAR* are developed in *o-Xylene* solvent.

HSQ is an electron-sensitive spin-on-glass. It is a negative-tone inorganic EBL resist capable of sub 10-nm feature sizes, and it's SiO_2 properties make it an excellent mask choice for both wet and dry etching. However, it is a very expensive resist and has a limited shelf-life that requires careful storage and management of inventory. *HSQ* is developed in 25% *TMAH* in

TABLE IV

OVERVIEW OF FAB.NANO LAB-SUPPLIED DEVELOPERS & SOLVENTS

Name	Type	Additional Chemical Detail (1)	To be used in	Disposal	Primary Application
AZ 300 MIF	Developer	0.26N (0.238%) TMAH	Developer Hoods	Aspirate/AWN	Standard resist developer without surfactant
AZ 726 MIF	Developer	0.26N (0.238%) TMAH	Developer Hoods	Aspirate/AWN	Standard resist developer with surfactant
AZ 435 MIF	Developer	0.35N (0.32%) TMAH	Developer Hoods	Aspirate/AWN	For thick resist developing
HMDS	Adhesion Promoter	-	Spin-Coaters	Solvent Waste	Adhesion promotor for spin-coating, can be spin-coated but recommend HMDS oven
SurPass 3000 SurPass 4000	Adhesion Promoter for EBL	mildly acidic mildly alkaline	Spin-Coaters	Solvent Waste	Can help improve resist adhesion to substrates such as III-V's for electron beam lithography
Isopropyl Alcohol (IPA)	Solvent	-	Solvent Hoods	Solvent Waste	Cleaning / Rinsing
Methanol (MeOH)	Solvent	-	Solvent Hoods	Solvent Waste	Cleaning / Rinsing
Acetone	Solvent	-	Solvent Hoods	Solvent Waste	Cleaning / Rinsing / Liftoff
PGMEA	Solvent/Developer	-	Solvent Hoods	Solvent Waste	SU8 developer
EBR 70/30	Solvent	70% PGME / 30% PGMEA	Solvent Hoods	Solvent Waste	Cleaning / EBR
NMP	Solvent	-	Solvent Hoods	Solvent Waste	Cleaning / Rinsing / Liftoff
MIBK	Solvent	-	Solvent Hoods	Solvent Waste	Developing PMMA, diluting HSQ
Anisole	Carrier Solvent	-	Solvent Hoods	Solvent Waste	Diluting PMMA and ZEP
o-Xylene	Solvent/Developer	-	Solvent Hoods	Solvent Waste	Developing ZEP and CSAR
3:1 IPA:MIBK	Solvent/Developer	-	Solvent Hoods	Solvent Waste	Developing PMMA (preferred)
25% TMAH	Base	Particularly hazardous. Must use in acid hood, with corrosive PPE & physical buddy	Acid Hood	Aspirate/AWN	Developing HSQ
Salty Developer	Base	NaOH / NaCl	Dedicated Hood	Aspirate/AWN	Safer developer for HSQ

(1): Please refer to Safety Data Sheets (SDS), PubChem, and/or CAMEO Chemicals for each chemical for a more in-depth overview of their hazards, incompatibilities, and proper handling procedures & PPE.

Chemical Acronyms: HMDS (Hexamethyldisilazane), EBR (edge bead remover), PGMEA or PM Acetate (1-Methoxy-2-propanol acetate, aka Propylene glycol methyl ether acetate), PGME (1-Methoxy-2-propanol), NMP (N-Methyl-2-pyrrolidone), TMAH (Tetramethylammonium hydroxide), IPA (Isopropyl alcohol), MIBK (Methyl isobutyl ketone), GBL (Gamma Butyrolactone), Anisole (Methoxybenzene), AWN (acid-waste neutralization system) an acid-hood. Because 25% *TMAH* is particularly hazardous [8], a safer and higher resolution alternative is "salty developer", which is a *NaOH/NaCl* based developer [9]. A dedicated hood is used for Na^+ and K^+ developer products to reduce the risk of ionic contamination and to provide special cleaning procedures before samples join the rest of the lab. The only practical removal method of *HSQ* is by using hydrofluoric acid.

A lower-cost alternative to *HSQ* is provided by ***ma-N***, a negative-tone organic EBL resist. A benefit of *ma-N* over *HSQ* is that, being an organic resist, it can be removed with solvents (instead of hydrofluoric acid).

Many EBL resists are not UV light sensitive. However, some (e.g. *PMMA, ma-N*) are sensitive in the far-UV below 240 nm [10]. The far-UV sensitivity opens up interesting applications of PMMA as a protective or sacrificial layer, because flood-exposure of far-UV using a cross-linker UV-source allows for easy removal or undercuts, e.g. in situations where a substrate is sensitive to the standard *AZ* developers.

V. LAB-SUPPLIED PHOTO SOLVENTS & DEVELOPERS

A list of lab-supplied solvents and developers used in lithography is summarized in Table IV. Standard photoresists develop in a variety of basic solutions. Photoresist developers are often low-concentration solutions of *KOH* or *NaOH* in water, and can have added surfactant to assist improve surface-wetting. In many electronic devices, including silicon CMOS chips, the mobile potassium (K^+) and sodium (Na^+) can be detrimental to device performance. Because of this, the microelectronics industry switched to a "metal ion free" developer (***MIF***) which is a solution of low-concentration *TMAH*. FAB.nano supplies 3 varieties of *MIF* developer and we isolate the use of K^+ and Na^+ developers (including the "salty developer") to a dedicated hood to avoid device contamination throughout the lab.

A. Standard TMAH Developers

The ***AZ 300 MIF*** and ***AZ 726 MIF*** developers have identical 0.26N (2.38%) *TMAH* concentrations, but the 726 has added surfactants to facilitate surface wetting and a more even development. Our automated dispense developers will use the 726. For immersion development, both the 300 and 726 are suitable, and in many cases expected to be indistinguishable. For thicker resists, the ***AZ 435 MIF*** provides a higher concentration and faster development time. All three developers are basic and disposed into the acid-waste neutralization system by aspirating or disposing into the developer bench sink.

B. Corrosive Developers

The development of *HSQ* for high contrast commonly uses concentrated ***25% TMAH***. This is a particularly hazardous chemical [8], and must only be used in an acid-hood with the correct corrosive personal protective equipment (PPE). A safer and higher contrast alternative is the use of the "***salty developer***", a *NaOH / NaCl* mix [9], which is used in a dedicated hood to reduce Na^+ cross-contamination.

C. Solvent Developers

If needed, ***MIBK***, ***Anisole*** and ***NMP*** can be used to dilute resists down (depending on the resist's carrier solvent) for immediate use. However, the standard solvents we supply are not sufficiently high purity to allow diluting resists for longer-term storage. ***O-Xylene*** and the ***3:1 IPA:MIBK*** pre-mix are developers for *ZEP* and *PMMA*, respectively. ***PGMEA*** (also referred to as ***PM-Acetate***) is the developer for *SU8*.

D. Solvents for Cleaning

The solvents in Table IV are mainly used to clean substrates (e.g. ***Acetone***, ***Methanol***, ***Isopropyl Alcohol***) or develop certain types of resist (mainly EBL resists and *SU8*). ***NMP*** is mainly used for liftoff or resist removal.

Acetone evaporates quickly, and can leave residues on the substrate surface. When using acetone to dissolve resist, and the acetone dries out, the particles and resist residues left on the dried-out surface are very challenging to remove. Therefore, the acetone is typically rinsed off using a different solvent – for example isopropyl alcohol, or methanol followed by isopropyl alcohol.

While all solvents are flammable, the flash-point and health hazards vary significantly between them, and it is important not to assume that the solvents are all equal in those respects. This is also why we are using acetone for cleaning the resist spin-coaters (instead of e.g. *NMP*, which has considerable health hazards compared to acetone [11]).

VI. PERSONAL-CHEMICAL REQUESTS

We expect that our lab-supplied chemicals are adequate for well over 90% of the user needs. However, in some situation a user may require very specific chemicals that we do not stock, and which do not have equivalents (e.g. polyimide). In these situations, the user can fill out a chemical request form on the MIT.nano website [12], requesting permission to supply a user-specific chemical. Upon approval, the user will coordinate with MIT.nano staff, who will receive the chemical, inventory and tag it, and bring it into the lab.

Users are not permitted to bring any chemicals through the gowning area, and building code prohibits the transport of chemicals through public elevators, stairways, and hallways in the building. Any exceptions are handled on a case-by-case basis.

The overview in Table II merely provides some guidance for where user-chemicals may be used. Once a user-chemical is approved, FAB.nano staff will provide further guidance for the spin-coater that should be used for that chemical (based on the specifics of the chemistry and the application).

Users are not permitted to bring any chemicals through the gowning area, and building code forbids the transport of chemicals through public elevators, stairways, and hallways in the building.

If, over time, a chemical turns out to be needed by many users from many research groups, we will consider adding it to the lab-supplied chemical list. Similarly, for chemicals that fail to see any significant demand over time, we may eventually remove it from the lab-supplied list and any future need would be accommodated as a user-chemical.

VII. CONCLUSION

This paper provided an overview of the spin-coating capabilities at FAB.nano. We summarized the different spin-coaters (Table I), and how they map onto specific resist choices (Table II). Details about the lab-supplied resists (Table III) as well as supplementary chemicals for cleaning/developing (Table IV) were provided.

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