E-beam resist recipes

Here we can keep a list of resist recipes used in the group.

Contents

External pages

Kavli Nanolab page:

https://www.tudelft.nl/en/faculty-of-applied-sciences/about-faculty/departments/quantum-nanoscience/kavli-nanolab-delft/equipment/process-recipes/resist-recipes/

A brief ebeam resist comparson: Media:Ebeam_resist_compare.pdf

Allresist page for all of their resists: http://www.allresist.com/downloads/

Microchem page (PMMA / PMGI / LOR / MAA-MMA copolymer): http://microchem.com/Products.htm

Microchemicals page (HMDS and TiPrime adhesion promotors):
https://www.microchemicals.com/products/adhesion_promotion/hmds.html

(note confusing name similarity of microchem and microchemicals...)

Resist adhesion

Depending on the exact resist, substrate, and substrate cleanliness, you might experience severe problems with resist adhesion. These usually manifest themselves in the form of cracks, either at the chip edges, or around sharp corners of your patterns. It's therefore recommended to use filleting and round corners as much as possible. The best way to deal with adhesion problems is the following methods:

- Pre-bake your substrates at 120C or higher (no upper limit really) for 5min or longer, to evaporate any water from the surface
- Apply the resist to your sample, but instead of spinning right away, let it sit for 60s so the resist can adhere and spread out
- **Use adhesion promoters**
- **such as TI Prime**
    - contains titanium, so remember to descum in case of lift-off, and think about if Ti is bad for your sample
    - spin at 4000rpm, bake 2min 110C
    - subsequently apply other resist layers as usual
- Alternative adhesion promoter: **HMDS**
    - not intended for spin-coating
    - There's a machine in the old MED module that does the surface prep for you, the DeltaRC80 (manual on cleanroom database). Recommended procedure: 6min preback at 150C, then run recipe 3
    - if you want to spincoat, recommended procedure is 4000rpm, and 2min bake at 200C (Eugene). But: NEVER Spincoat!!! The manufacturer also tells you NEVER to spin coat (https://www.microchemicals.com/products/adhesion_promotion/hmds.html)!!! Don't do it!

PMGI / PMMA Dolan Bridge (Sal's qubit recipe)

Sal's qubit recipe

pattern for resist bridges 100nm x 400nm

1. Layer 1: PMGI SF7 @ 4500 rpm, bake at 180C for 15 min (320nm)
2. Layer 2: PMMA 950K A3 @ 5500 rpm, bake at 175C for 15 min (120nm)
3. Exposure: 2200 uC/cm2
4. Develop:
    1. PMMA layer: MIBK 1:3 IPA 70s, IPA 70s, N2 blowdry
    2. PMGI layer: MF321 10s, H2O I 10s, H2O II 15s, IPA >20s, N2 blowdry
5. Cleaning:
    1. Tepla clean: 200 W 200 sccm O2, 30 sec
    2. HF dip: BOE 20 sec

1-2-2017 - (Mark) Tried today and worked but used 60s to develop PMMA layer. Used 2200 uC/cm2 on EBPG5000+ and used smallest beam (2nm spot size, 24NM_441PA_400UM_100KV) with no PEC. Bridge size is designed to be 1 um across and 160 nm wide. The substrate was MoRe no sapphire but the bridge itself was patterned in a 100x80 micron hole in the metallization (directly on sapphire). No electra or aquasave was used.

7-2-2017

1. Evaporation: 45nm / 90nm and 35 degree angle, should produce 225 nm overlap.
2. Lift-off: NMP at 80 C for 20 minutes.

Marios's qubit recipe

Note: Mario uses a tilt angle of 11° during evaporation! Byoung-moo uses 35°

Junctions	preheat 110C 1 min
bilayer spin	PMGI SF7 1500 rpm Bake @ 175 C for 5' 570nm
	cool for about 2'
	PMMA A3 950k 2500 rpm. Bake @ 175 C for 10' >175nm Total thickness: 720 nm
	This second layer can be thicker to have smaller JJ and larger play
e-beam	
	dose 1850 for junctions layer, Bss 2.5nm,NEW EBEAM: 5nm 23nm_4963pA_400nm_100kV
Developing	
	MIBK : IPA, 1:3 60"
	Ethanol : IPA, 1:3 10"
	IPA, 10"
	Blowdry N2
	PMGI develop MF321 undiluted 12", water rinse
	Check the result with the microscope before proceeding
	Evaporate fairy dust
pre cleaning	Plasma cleaning in F3:
	Oxygen 10.0 (sscm?) (pressure 10 mbar?), power 195W, 45" plasma
	HF dip BufferedHF : H2O (20:20) 30sec dip
Al Evaporation	Titanium evaporation: 48nm, 0.1 nm/s Liquid nitrogen cooling
	Aluminium layer: 30nm, 0.1 nm/s, M3 191° M2 -175° (35degree, it's 11degree right?-b.ann) **Warning: see note before this table!**
	Oxidation layer: P = 0.5 mbar, 8', 0°
	Aluminium layer:50nm, 0.1nm/s, M2 169°
	Oxidation layer: P = 0.5 mbar, 8', 0°
Lift off	NMP @ 88 C with stirring 20'
	IPA rinse

Copolymer / PMMA Dolan Bridge

Thicknesses estimated from datasheet Media:PMMA_Data_Sheet.pdf

1. Copolymer (MMA) 8.5% EL13 3000rpm for 60s, HP bake 175°C for 15 min (around 700 nm, extrapolated)

2. PMMA 950 A4 3000rpm, HP bake 175°C for 15 min (about 220-240 nm)
3. Expose at dose 1200 uC/cm2 with PEC (probably works without PEC, too), (Mark: I had to use 900-1000 μC dose)
4. Develop in MIBK+IPA 1:3 for 60 seconds and stop in IPA, 30 sec.

With +- 11° evaporation, ~200nm overlap (Doesn't this depend on the bridge size??)

Can be used for marker lift off, as well

Note on overlap
Using simple trigonometry:

$$\text{Overlap} \simeq 2(t_{\text{copoly}} - t_{\text{Al}}) \tan\theta - w_{\text{bridge}}$$

According to this, for a 160 nm wide bridge, 15° evaporation angle angle, and a 50 nm Al layer, the overlap should be about 180-190 nm

Standard 495K / 950K PMMA for liftoff (double layer)

1. Bottom layer: 495 A4 @ 4000rpm, bake at 175C for 10 min (180 nm)
2. Top layer: 950 A3 @ 4000rpm, bake at 175C for 10 min (120-140 nm)
3. Exposure: 1200 uC/cm2 (depending on feature size?)
4. Develop: MIBK:IPA (1:3) for 60 sec, stop in IPA 60 sec

- Note: thicknesses estimated from Media:PMMA_Data_Sheet.pdf

- According to Gary, this is very important to let the sample fully cool down before spinning the 2nd layer; otherwise the two layers may mix and give random total thickness.

Standard 950K / 495K / 950K PMMA for liftoff (triple layer)

1. Layer 1: 950 A7 @ 3000rpm, bake at 180C for 4 min (450 nm)
2. Layer 2: 495 A4 @ 3000rpm, bake at 180C for 4 min (190 nm)
3. Layer 3: 950 A7 @ 3000rpm, bake at 180C for 4 min (450 nm)
4. Exposure: 1200-2400 uC/cm2
5. Develop: MIBK:IPA (1:3) for 60 sec, stop in IPA 60 sec

- Resist stripping: At least 1 hour in PRS3000
- Note: thicknesses estimated from Media:PMMA_Data_Sheet.pdf
- Likewise the bilayer case, this is very important to let the sample fully cool down after each baking step and before spinning the next layer; otherwise the layers may mix and give random total thickness.

Thick PMMA for etching

1. Resist: 950 A8 @ 2000rpm, bake 15min at 175C. **Note: 5 min also works!**
2. Exposure: 1500uC/cm2
3. Develop: MIBK:IPA 1:3 60sec, IPA 60sec

- Resist about 1.1um thick

S1813 / W / PMMA Three layer mask for dry etching

1. spin S1813 at 3000rpm + 110°C for 2 min + 180°C for 30min --- 1.4um thickness
2. sputter W recipe 150WRFVC3, 150W, pressure 0.02 for 15s --- ~9nm thickness
3. spin PMMA 950 A6 at 3000rpm + 180°C for 2min
4. ebeam expose with dose 1200uC/cm2
5. develop with MIBK 1:3 IPA for 70s and stop with IPA

for negative resist change steps 3.-5. for this:

- spin AR-N-7700-18 at 1500rpm + 85° for 1:30min
- ebeam expose with dose 110uC/cm2 and after bake at 110°C for 2min
- develop with MF321 for 90s and stop with Water

ZEP

Discontinued resist? Used to be used as a PMMA alternative with better dry etch resistance

AR-P 6200 (CSAR 62)

http://www.allresist.com/csar-62-ar-p-6200/

Recommended by Charles and Marco as an e-beam positive mask with good dry etch resistance and less teflonization than S1813.

1. SxAR-P 6200.09 at 4000 rpm, bake 3 min on 150 C hotplate (200 nm)
2. Exposure: 350 uC/cm2 or higher
3. Develop: Pentylacetate 60 sec, stop with MIBK 1:1 IPA for 60 sec

Update 14-4-2016 (Mark): Using somewhat lower doses (210 uC/cm2) appears to improve pattern reproducibility.

Update 02-05-2017 (Felix): Same dose for AR-P 6200.13, 400nm @ 4000 rpm. Clearance dose 260 uC/cm2. This resistis better suited for etching MoRe/NbTiN for more than 2 min, i.e. 100nm thick layers

- Spin coating:

SX AR-P6200/13	1000 - 8000rpm: 750nm - 250nm
SX AR-P6200.2 or P6200/9:	1000 – 8000rpm: 390 – 175nm
Diluted SX AR-P6200.2 (1:1):	1000 – 8000rpm: 150 – 50nm
SX AR-P6200/04	1000 - 8000rpm: 250 - 60nm
SX AR-P6200/13	4000rpm: 400nm
SX AR-P6200/09	4000rpm: 200nm
SX AR-P6200/04	4000rpm: 80nm

QuTech recipe for lift-off of 100nm wide sputtered SiN structures

- Spincoating: CSAR.04 @ 4000rpm, bake 3min on 150C hotplate
- Exposure: 450μC/cm^2, beam: 21NM_897PA_400UM_100KV.beam_100
- Development: Pentylacetate 60sec, MIBK:IPA (1:1) 60sec, IPA 60sec

- Sputter 50 nm SiN (100% Ar) for ~650 sec
- Liftoff in NMP for 4 hours (@80C)
- Finish liftoff using a spraying syringe
- 1 min in Acetone, 1 min in IPA
- Dry with N2 gun

AR-N 7700

http://www.allresist.com/ebeamresists-ar-n-7700-7720/

Negative ebeam resist

Note: For circumventing the stripping issues of AR-N after dry-etching PMMA below **is not necessary** (Felix, July 2018). Instead, you can do an in-situ oxygen cleaning step. The recipe for the Leybold F3 is: 50sccm O2, 30W, 290Vb, anywhere between 30-90s. Take care that you set up this one before the SF6/O2 (or CHF3/O3, or whatever else), as venting the chamber in between and only then attempting to do O2 cleaning does not work!

Note 2: Recently (late 2017) there have been a lot of problems in stripping AR-N after dry-etching. A solution to this is to add a PMMA layer below which, after development of the AR-N, can be removed by using an oxygen plasma (see Mario's recipe) **This is not necessary anymore, see above!!!**

Note 3: Recently (June 2019) stripping problem AR-N after dry-etching shows up again even after oxygen ashing. I (bann) could remove remaining ARN using Tepla and stripping again with very hot PRS or NMP (more than 80 degree). Please be careful with stripping and do not forget to put the sample in ultrasonic bath. I has been found important. Probably more power in-situ oxygen cleaning step is recommended.

Anja's original recipe:

1. spincoating: 5s @500rpm, 55s @6000rpm: film thickness of about 320nm
2. prebake: 85C on hotplate for 1 minute
3. EBPG: dose 117uC/cm2
4. post-exposure bake: hotplate at 105C, for 2 minutes
5. development: MF321, 1 minute (hold sample vertically)
6. rinse: MF321:H2O=1:10 15s (2x/ second time in fresh bath) + H2O (2x/ second time in fresh bath) (sample always vertical)

Daniel's recipe:

1. Spin at 3000 rpm 1 minute
2. Bake at 90C for 1 minute on hot plate
3. Expose at dose 180-200 uC/cm2
4. Post Exposure bake at 105C for 2 minutes on hot plate
5. Develop in MF321 for 1 min and then stop by rinsing in MF321:(DI H20) 1:10 twice and once more in DI water
6. Strip with warm (80C) PRS3000

Mario's recipe for Al patterning

1. Spin PMMA 950K A3, 4000 rpm, bake 90s 185C (130nm)
2. Spin ARN 7700/18, 8000 rpm, bake 90s 85C (200nm)
3. Expose with 100 uC/cm2 (I use biggest beam with a 65nm resolution, and can pattern 250nm lines with high accuracy)

4. Post-bake 2min at 110C
5. Develop in MF321 for ? min and then stop by rinsing in MF321:(DI H20) 1:10 twice and once more in DI water
6. Ashing of PMMA: F1/2 using the O2 cleaning recipe at 50W

HSQ (negative resist)

A general word about using HSQ: It's a fairly delicate resist, especially compared to PMMA.

- HSQ is stored in the refrigerator. It is important to let the resist warm up to room temperature prior to spin coating
- HSQ is **NOT** poured from the big bottles, but from the small plastic ones. If the one you want to use is (close to) empty, talk to Eugene to refill it.
- DO NOT directly pour onto sample, but use syringe and filter! (Lots of particles otherwise).
    - DO NOT use glass beaker, but either directly pour into syringe or use teflon beaker and clean with BOE (Anja)
- Spin curves can be found at TU Delft webpage (https://www.tudelft.nl/en/faculty-of-applied-sciences/about-faculty/departments/quantum-nanoscience/kavli-nanolab-delft/equipment/process-recipes/resist-recipes/hsq-xr-1541-fox-12-recipe/)

HSQ recipe for covering gate electrodes

1. Pure HSQ, 8000 rpm, bake for 10 min in 90C oven
2. Exposure: 1000uC/cm2
3. Develop: MF322 for 1min + MF322:H2O (1:9) for 15sec+ H2O for 15sec

HSQ recipe for high-resolution dry-etching

1. Bottom layer: AR-P 6200.09, 4000 rpm, bake 3 min 150C
2. Top layer: HSQ:MIBK (1:1), 10000rpm, bake 2 min 150C **and** 2 min 220C
3. Exposure: 1000uC/cm2
4. Develop: MF322 for 1min + MF322:H2O (1:9) for 15sec+ H2O for 15sec
5. Etch bottom layer in F1/F2: 20 sccm O2, 60W, 50 ubar for 1min

HSQ recipe for hBN/Gr shaping (outdated)

1. Bottom layer: 495 A4 @ 4000rpm, bake at 175C for 10 min (180 nm)
2. Top layer: HSQ (1:1) @ 10,000rpm, bake at 150C for 2 min and 220C for 2 min (first spinner recipe 20; "try to avoid 8000rpm") (Felix: 8000 rpm should give around 50 nm)
3. Exposure: 950uC/cm2
4. Develop: MF322 for 1min + MF322:H2O (1:9) for 15sec+ H2O for 15sec
5. Etch bottom PMMA in F1/F2: 20sccm O2, 60W, 50ubar for 1min

- Stripping HSQ requires a short dip in HF (1:200 diluted works), but with the additional PMMA bottom layer the mask should come off when left in warm acetone for 30min and cold acetone overnight (maybe even better when doing lift-off?)
- Since HSQ seems to have rather low contrast, proximity correction might be considered important. (Felix)

SAL

A super-fast resist that has good dry etch resistance

Electra 92 (replacement for aquasave)

- Coating for Ebeam resist to reduce charging effect. Used when patterning directly on insulating substrate e.g. sapphire.
- Kept in fridge inside CR. Remember to let it warm up prior to spincoating.
- Dedicated spinners?

1. Prepare Ebeam resist as usual.
2. Spin Electra at 2000 rpm; 80C for 90 sec.
3. Ebeam exposure as usual.
4. Rinse in DI water and blow-dry.
5. Develop as usual.

- Note: Is not supposed to be spun onto samples at the usual spinners, use the spinner behind the BN module instead!
