CEITEC NANO

e-beam photoresist PMMA, CSAR

Standard operating procedure

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0.1	1.4.2020	Hrdy
0.2	30.11.2020	Zita

Location: Building C, room C1.31

Cleanroom class: 100 (1000 in corridor)

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Training requirements: Safety training 100

FUMEHOOD SC1 certificate

RCD certificate

DIENER certificate

Requirements for work: Wafer PEEK tweezers or any other PEEK tweezers

Vacuum tweezers

Completely new or pre-cleaned wafers

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Introduction to PMMA and CSAR resist

Polymethylmethacrylate (PMMA) Resist is the industry standard electron beam resist used across academia and industry for high resolution features and lift-off applications. It can also be used in nanoimprint applications as well as other fab and R&D processes such as graphene flake transfer.
CSAR is a high-tech resist for electron beam lithography which allows the implementation of high-end applications in microelectronics, e.g. for the aerospace industry or high-performance computers. Even incredibly small structures of < 10 nm for the highly integrated circuits of a microchip can be realized with this resist.
According to the applications in our laboratory, electron-beam sensitive resists are used. The example of electron-beam resist is polymethylmethacrylate PMMA diluted in chlorobenzene, anisole or ethylactate solvent. The differences between both solvents are in possibilities of diluting to lower viscosity of resist. The ethylactate is generally used for thinner version of resist.
In the process of exposure, polymer chains are broken down in the case of positive tone, thus resulting in empty areas on the substrate after development. On the contrary, irradiated area in negative resist is cross-linked/hardened, so developer won't attack it – it will dissolve only unexposed resist.

Before you start working, READ the datasheet of e-beam resist producer!

PMMA AR-P 672 series

PMMA AR-P 679 series

CSAR AR-P 6200 series

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SOP for PMMA and CSAR

The SOP is optimized for syringe spin coating on RCD8.

Resist preparation (Optional)

If you need to prepare a lower concentration other than is disposable.

Strongly recommendation for this step to ask for help to the chemical laboratory supervisor or experienced user

1. Resist diluting

a) Wash and clean all bottles, syringes, pistons and tips, beware of acetone.
b) Finally clean them in proper solvent – anisole, ethlylactate – read on the original bottle of resist
c) Dry them by nitrogen
d) Temper the resist and thinner to the room temperature
e) Mix the resist and thinner according to the formula, where c_{resist} is concentration of resist labeled on original bottle:

$$m_{thiner} = m_{reisit} \frac{(c_{resist} - c_{desired})}{c_{desired}} = 100g \frac{(0.5 - 0.2)}{0.2} = 150g\ of\ thiner$$

f) Prepare the beaker (250 ml) on laboratory scales and TARE the beaker weight to ZERO.
g) Fill the beaker by resist max ¼ of volume, CALCULATE THE FINAL VOLUME. *The syringe has 60 ml volume!*
h) Use the weight of resist to the formula, calculate the weight of thinner.
i) TARE the laboratory scale to ZERO. The beaker with resist must stay on scale.
j) Fill in the beaker with thinner to the calculated weight.
k) Use magnetic stirrer and slowly mix the new resist min 1 hour.
l) Store the resist in the fridge.

2. Syringe filling

a) Close the syringe by tip
b) Fill the syringe by wall side to prevent bubbles presence, leave the space for piston
c) Prewet the rubber ring of piston by resist and gently press it to syringe
d) Fix the syringe to RCD8 motor syringe holder, hold it with tip to up and open the tip.
e) Release the remained air bubbles by manual operation mode of RDC8 – check RCD8 manual
f) Release syringe and switch RCD8 to automatic mode
g) Wait minimally 6 hours (12 hours is optimal) before using or store the syringe in the fridge.

Resist coating

1. Clean wafers

a) Remove photoresist if wafers are being reworked using proper ethyllactate, anisole, **DO NOT USE ACETONE or IPA**
b) Optional - RCA cleaning of wafers - for more details go to- SOP - RCA cleaning of wafers
c) Dehydration baking at Hot plate: 5 min @ 150 °C minimum

2. Promotor (Optional)

Use the promotor for adhesion improvement if it is necessary

d) If the promotor is needed go to Chapter 4 - SOP - SUSS-VP8 Vapor primer
e) Use the O2 plasma (DIENER receipt) for activation of surface, dehydration. For clean Si/SiO_2 wafers only. Do not use plasma with surface oxidizing metals presence.

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3. Spin-coat wafers

a) Fix the syringe to RCD8 motor syringe holder and open the tip.
b) Release the remained air bubbles by manual operation mode of RDC8 – check RCD8 manual
c) Switch RCD8 to automatic mode and fix the syringe to arm holder for syringe
d) The syringe MUST be tempered to the room temperature.
e) Select the proper program. For example, *@ CSAR62 SID4* or *@ PMMA SID4*
f) START the program
g) Prewet the wafer by proper thinner.

Application note: *We strongly recommend testing the procedure on the test wafer and measure the thickness. Especially, if you are planning to use diluted resist with non-standard concentration.*
The program usually consists of 3 parts:
First one is prewet/cooling phase – The wafer is rotate on ~3000 rpm to be cooled from previous fabrication step and wetting by solvent. The RCD8 use PGMEA as standard solvent. However, you need the anisole or ethylactate to be used. The program must not have any Solvent, EBR or BSR functions. The prewet phase must be done manually with syringe. The prewet phase also has an important cleaning function.
The second phase is the coating of resist - check if the resist is completely covering the wafer, especially in the center. Check the dose of resist. Optimal dose is 4,5 – 5.5 ml according to viscosity. If it is necessary, correct the parameters of arm, speed of arm, dose.
The third main part is spin – The final thickness of the resist layer strongly dependents on the rotation speed and concentration. Set the right rotation speed. The usual time of rotation is 60 sec. If you are using high viscosity resist, such as PMMA A11 or more. You must prolong the time of rotation for 180 sec and use slow ramp of rotation for example 250 rpm/sec instead 1000 rpm as usual. Otherwise, your resist layer will be not homogenous on the edge. For more details, look at the summarized table.

h) After finishing the above process go to the Soft bake

Resist	Thickness	Speed rpm /ramp rpm.sec	SoftBake°C	SB Time
CSAR 6200.02	50	1500/800	150	1min
CSAR 6200.02	46	2000/800	150	1min
CSAR 6200.02	41	5500/800	150	1min
CSAR 6200.03	66	8000/800	150	1min
CSAR 6200.03	69	5500/800	150	1min
CSAR 6200.04	125	1100/800	150	1min
AR-P 679.01	26	1500/800	150	1min
AR-P 679.01	32	1000/800	150	1min
AR-P 679.015	50	3500/800	150	1min
AR-P 679.015	55	3000/800	150	3min
AR-P 679.02	125	1250/800	150	3min
AR-P 672.07	1100	2000/300	150	3min
AR-P 672.09	1120	5500/250* 3min	150	10min**
AR-P 672.09	1300	3100/250* 3min	150	10min**
AR-P 672.09	1590	2000/250* 3min	150	10min**

* be careful on acceleration

**** slowly increase the temperature and slow cooldown to avoid stresses in the layer.**

For more details about automatic deposition Spin coating in Chapter 5 - SOP - Resist deposition - RCD8

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4. Soft bake

(a) Hot plate for thin resist (<100nm): 150°C, 90 sec, use recipe **@PMMA/CSAR softbake**
(b) Hot plate for thicker resist: 150°C, 300 sec minimally, compare your resist thickness with datasheet instructions. Foe more detail look at Table 1.
(c) Proximity mode – if necessary – Use the same temperature as previous and prolong the time for a double.

For more details about automatic soft bake after spin-coating go Section 5.3 - Baking of the freshly spin-coated wafer (Soft-bake) SOP - Resist deposition - RCD8

5. Thickness check – reflectometry (Optional)

a) Use halogen lamp for thicknesses in range of tens of nm, for hundreds of nm thickness it is not necessary
b) Use the proper Cauchy parameters for fitting or use the PMMA 950 or CSAR models in library
c) Use the proper substrate to background detection
d) Check the thickness in the center and edge of your sample to confirm right spin coating

For more details about automatic reflectometry check – Quick guide for reflectometry.

E-beam lithography details in E-beam lithography SOP

EBL is quite commonly used technique in academia and industry, offering high resolution patterning by high energy electrons (2 to 100 KeV). Beam can be focused into a narrow beam area in gaussian scanning systems or shaped into various geometric structures in shape-beam systems. This technique offers precise control of energy and dose delivered to the electron beam sensitive resist coated wafers. Quite commonly, the masks for photolithography are manufactured by the utilization of this technique. Typically, the writing strategy does not require a hard mask. Instead the beam position is precisely controlled by computer-controlled pattern generator according to the design file.

Development

a) Select the proper receipt.

PMMA and copolymer resists can be developed using immersion, spray puddle and spray process techniques.

For more details about automatic development on RCD8 check SOP - Resist development - RCD8

PostBake (Optional)

(a) Hot plate: 100°C, 90 sec, use recipe **@PMMA/CSAR PoExpBake**
Application note: Be careful the reflow temperature is 125°C.
(b) Proximity mode or thicker resist (>100 nm) – if necessary – Use the same temperature as previous and prolong the time to the double.

Descum and optical check

a) Use a DIENER O_2 plasma descum procedure 10 sec – 1 min – if your critical detail is more than 1 µm
b) For saving of small critical details we recommend using fluorine RIE.
c) Check the final structure.

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Removal techniques

a) Wet: e-beam resist remover, Acryl strip or standard laboratory solvents. Do not use acetone. Dioxolane is required for CSAR. The PMMA could be also striped by ethanol or methanol. Rinse the wafer for 60 sec in DI and blow dry by dry N_2.
b) Bath: time as required at ambient temperature
c) Dry: plasma O2

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