



# 2016-01

Received the masks from toppan with new designs for BCB bridges

## Fabrication of new wafer BPC5

last wafer from batch 2602 [100] >10 kΩ.cm  
Al etch process  
started mardi 05/01  
See corresponding [BPC5](#) page for details

## Fabrication of development wafers BPCD8 & BPCD9 for multilayers

On standard Si/SiO<sub>2</sub> 500nm, we develop bi/trilayers to check :

- Tc
- Q factor of resonators
- contact resistance after 2nd litho
- ageing (at high temperature)

### • evap BPCD8 1st step (plassys550): 06/01/2016

pump 2h ->  $P_{ch} / P_{sas} = 9e-8 / 1.5e-7mb$   
pump Ti 20nm @ 0.2nm/s ->  $P_{ch} / P_{sas} = 3.8 / 7.5e-8mb$   
evap Al 102nm @ 1nm/s,  $P_{ch} / P_{sas} = 5 / 6e-7mb$  (hot) ->  $2.0 / 2.4e-7$  (c  
evap Pt 2.1nm @ 0.1nm/s,  $P_{ch} / P_{sas} = 4 / 4.7e-7mb$   
~5' wait time between end of Al and shutter open for Pt

### • evap BPCD9 1st step (plassys450): 06/01/2016

pump 2h ->  $P_{ch} = 3.9e-7mb$   
degas Au ->  $P_{ch} / sas = 1.5e-6 / 2e-6$   
degas Cu ->  $P_{ch} / sas = 1.1e-6 / 1.4e-6$   
pump Ti 20nm @ 0.2nm/s ->  $P_{ch} / sas = 1.8e-6 / 7e-7$  (hot)  
evap Al 101.8nm @ 1nm/s,  $P_{ch} / sas = 1.1e-6 / 4e-7$   
evap Cu 2.5nm (manual shunt) @ 0.5nm/s  
evap Au 2.5nm @ 0.5nm/s,  $P_{ch} / sas = 1.1e-6 / 4e-7$

jeudi 07/01

Cut part of the development wafers **BPCD8 et 9** to test ageing of the layers.

### First observation of BPCD8:

On the side of the layer, I could observe percolation of Al, because there was shadowing

by the clamp

In the bulk, I see grains of typical size 50-200nm (quite big!), and no apparent clusters of Pt.

-> The layer is wetting properly Al, or dissolving in it.

[BPCD8\\_ageing-v1.jpg](#)

**First observation of BPCD9:**

The average grain size is about the same, the percolation on the edge of the layer also. Remarkably, now we can see small clusters (<10nm) -tiny white spots- on top of the Al grains.

This might indicate the Cu/Au layer has not wetted the Al.

Note that Pt should have a high contrast with Al, similar to Au (they have about the same weight, neighbours in the table). On BPCD8 we see no white tiny spots.

Pictures: each column is taken at the same location on the sample, for different

magnifications (highest mag on top). The zoom position is shown by a rectangle.  
[BPCD9\\_ageing-v1.jpg](#)

**After 3h @ 220°C on hot plate**  
**left: BPCD8 (Pt)**

**right: BPCD9 (Cu/Au)**

BPCD8\_ageing-v2.JPG

BPCD9\_ageing-v2.JPG

Two different positions on each sample (in the bulk of the layer)

The Al/Pt layer was modified chemically, leading to the appearance of black spots, one can see on the optical microscope as well.

It seems (but hard to say) that the Cu/Au layer is a bit smoother (the tiny clusters may

have formed an alloy with Al). As Denis says, Cu and Au are mixable with Al.  
On the optical obs, it seems also that the Cu/Au turned a bit more pinky

left: BPCD8, right BPCD9; bottom: fresh, top: aged  
[BPCD8v1v2\\_x100.jpg](#)

[BPCD9v1v2\\_x100.jpg](#)

mardi 12/01

- Litho S1813 [mask 2013-09 BPCv1](#)

bake wafer 1' @ 110°C (solvent evaporation)  
spin S1813 60" @ 4krpm  
bake 2' @ 110°C  
Expo 150mJ/cm<sup>2</sup> @ 365nm, Vacuum contact -> 14.5" @ 11mW  
Dev MF319 50" @ 19.0°C, rinse ODI 60" beaker + 30" water tap

Done the same for both

On BPCD8, at 50", the Al layer is already etched, I see wavy strips (like level lines on a map) indicating so

I decide to finish etching in the developer: add something like 20" -> **1'10 to develop S1813 + etch 100nmAl/Pt**

**Note:** There are lots of tiny black peels on the developer and on the water beakers. I think this is the upper layer which has been "lifted". On many places of the wafer there are residues of this thin peel. This corroborates the hypothesis that the Pt top layer was protective against oxidation (-> continuous wetting layer) but not to wet etchant -> extremely reactive below the thin layer.

On BPCD9, everything goes fine, patterns are perfect. Cu/Au is probably not protective against oxidation (probably not continuous). (because it is certainly not protective against wet etch, as Ti 20 / Au 10 is not fully protective - see previous results on BPC2)

After etching is finished, I also see small peeling residues on the sides of the patterns (see pictures) indicating also that the top layer was more 'lifted' than etched away.

This is a problem for this kind of process, unless we first etch out this skin.

- Al-Etch (BPCD9 only). Peremption 2002 (!!)

With magnetic agitator in tall beaker (**no manual agitation**)  
etching starts from the edge and reaches slowly towards the center  
after 2' the edges seem to be etched  
after 2'30 the center seems also  
- leave 3', rinse ODI in beaker + 1' water tap

[BPCD9\\_S1813\\_160mJ\\_MF319-50"\\_Al-etch-3'\\_3um\\_x250.JPG](#)

- add 1', rinse ODI in beaker + 1' water tap

no change

- add 2', rinse ODI in beaker + 1' water tap

no change

- add 3', rinse ODI in beaker + 1' water tap

Can see clearly the overetch (resist on top:  $4\mu\text{m}$ , wire on the bottom:  $2\mu\text{m}$ )

[BPCD9\\_S1813\\_160mJ\\_MF319-50"\\_Al-etch-9'\\_3um\\_x250.JPG](#)

Below, after dicing and cleaning, BPCD8 (left), and BPCD9 (right)

BPCD8\_final\_3um\_x50.JPG

BPCD9\_final\_3um\_x50.JPG



BPCD8\_final\_res\_x5.JPG

BPCD9\_final\_res\_x5.JPG

- **Measurements: 23/02/2016**

[BPCD8\\_9\\_cooldown.jpg](#)

[BPCD8-9\\_Tc.jpg](#)

### **Fabrication of BPC6 (lift-off process)**

wafer from batch 3481  $\rho > 20 \text{ kohm.cm}$  SSP  
started vendredi 08/01  
Already measured good resonances after 1st step (Al layer only)  
See page [BPC6](#)

### **Test samples for Al resonator on Si**

- Layer of Al 20nm

On standard Si/SiO<sub>2</sub> 500nm, with 20nm Al evaporated on top

clean wafer in acetone + IPA for 10min  
heat wafer in hotplate at 120deg for 20min to eliminate solvents and water  
spin Ti primer 60sec at 4000rpm  
bake Ti primer at 120deg for 2min

spin S1813 for 60sec at 4000rpm  
bake S1813 for 2min at 110deg  
expose S1813 for 13.5sec (~148.5mJ) on vac contact  
develop 55sec in MF139 + clean in DI water

After 55sec of development there is still resine in between the lines of the bobine but in the corners the Al can be distinguished.(no picture taken, sorry)

develop 15 sec more in MF139 + clean in DI water

[Litho\\_S1813onAl\\_dose13p5sec\\_dev70sec\\_1.JPG](#)

[Litho\\_S1813onAl\\_dose13p5sec\\_dev70sec\\_2.JPG](#)

It thought at the beginning that there were still resine traces bat in fact, **it can be observed that Al has been etched in many places. !!!**  
**1 min or 1min 5sec is enough for developpement !!! (but it is not homogeneous across the wafer)**

develop 15 sec more in MF139 + clean in DI water -> 1'20

Litho\_S1813onAl\_dose13p5sec\_dev85sec\_1.JPG

The substrate can be observed in many places; We decide to cut the wafer in four pieces to test different Al etchings.

- **etching by developer (MF319) itself**

- add 90+45sec

- > the resist is gone and I etch the bobine;

- add only 105sec

- > apparently OK

**FIRST (135s) before**

TESTa-  
Litho\_S1813onAl\_dose13p5sec\_dev85sec\_1.JPG

TESTa-  
Litho\_S1813onAl\_dose13p5sec\_dev85sec\_2.JPG

FIRST(135s) after 90s

TESTa-  
Litho\_S1813onAl\_dose13p5sec\_dev85sec\_dev90sec\_1.JPG

TESTa-  
Litho\_S1813onAl\_dose13p5sec\_dev85sec\_dev90sec\_3.JPG

FIRST after 135s (in total is :  $55+15+15+135=220$ sec)

TESTa-  
Litho\_S1813onAl\_dose13p5sec\_dev85sec\_dev135sec\_1.JPG

TESTa-  
Litho\_S1813onAl\_dose13p5sec\_dev85sec\_dev135sec\_2.JPG

SECOND after 105 sec (in total is :  $55+15+15+105=190$ sec)

TESTb-  
Litho\_S1813onAl\_dose13p5sec\_dev85sec\_dev105sec\_1.JPG

TESTb-  
Litho\_S1813onAl\_dose13p5sec\_dev85sec\_dev105sec\_2.JPG



TESTb-  
Litho\_S1813onAl\_dose13p5sec\_dev85sec\_dev105sec\_5.JPG

TESTb-  
Litho\_S1813onAl\_dose13p5sec\_dev85sec\_dev105sec\_3.JPG

TESTb-  
Litho\_S1813onAl\_dose13p5sec\_dev85sec\_dev105sec\_4.JPG

lundi 11/01

- **etching by Al etch**

Where etching is done, patterns are OK, but many places where it does not etch. Means there is resist remaining.

5' -> etched patterns are OK

12' -> etched small patterns are gone. The ones with resist residues are still there

**The result of the "Al etch" is the following:**

-Resine S1813 is rather resistant to the etchant;

-After 1min 30sec, Al is gone from some places but not from everywhere; I wait 5 minutes but still the same. We constate on the microscope that the places where the Al is not gone are still cover by resin. We try a plasma oxygen for 1min to take out the remaining resist.

-After 20min there is not much change; The fine patterns (bobine par exemple) in the part of the sample where Al was already gone start to leave; That is reasonable since etching continues under the resist.

-> SHOULD IMPROVE RESIST DEVELOPMENT

- **etching by HF**

15" HF:ODI 1:20

Very inhomogeneous: either bobine pattern is gone, or there are still residues of Al

**The result of the "HF etch" is the following:**

-After 20 sec Al is gone; The result is not homogeneous. The bobine is gone in many places.

**Overall it seems that the development was not proper (very inhomogeneous), and that it makes it impossible to properly etch the layer with a solution that does not attack the resist**

The result is not homogeneous in the whole wafer.

The other two pieces of the wafer are etched in "Al etch" and in HF solution 1:20 (Helene verified that 100nm of Al are etched in 30sec).

## General questions and conclusions:

- Why is the litho so inhomogeneous? Is the spinning inhomogeneous? Is the resist old? Is a problem of the development?

-Which is the adequate dosis for S1813?

*Up to now, we have tried 16 sec (overdose) and 13.5 sec (slightly underdose) ; I would try 14 or 14.5 sec and 50-55 sec dev + plasma oxigen 1min*

-Which is the etching rate of Al in MF319 ?

*We have etched 20nm in 105 sec, in average.*

-HF seems to attack very fast also the resist

-MF319 leads to a good result. How long do we need for 100nm?

*It seems we would need 525sec (8 min 45 sec) but I dont think the resist is going to stand such a long time.*

-Al etch is very sensitive to residues of resist.

- **Layer of Al 100nm**

Si doped 100ohm.cm, unoxidized

Evap 100nm Al @ 0.1nm/s

heat wafer in hotplate at 120° for 15min to eliminate water

spin TI primer 60sec at 4000rpm

bake Ti primer at 120° for 2min

spin S1813 for 60sec at 4000rpm

bake S1813 for 2min at 110deg

**expose S1813 for 14.5sec on vac contact**

**develop 50sec in MF139 + clean in DI water + tap water**

TEST100-Litho\_S1813onAl\_dose14p5sec\_dev55sec\_1.JPG

TEST100-Litho\_S1813onAl\_dose14p5sec\_dev55sec\_3.JPG

TEST100-Litho\_S1813onAl\_dose14p5sec\_dev55sec\_4.JPG

TEST100-Litho\_S1813onAl\_dose14p5sec\_dev55sec\_5.JPG

This time resine has good aspect and litho seems homogeneous in the wafer. Dose is correct and development time is right.

Cut two pieces of the wafer to measure approximate etch time in MF319 and "Al etch" previous removal of resist in acetone heated at 60° for 10min.

**-MF319 --> 100nm gone in 6min10sec**

**-Al etch --> 100nm gone in 5min**

clean resis residues in 1min plasma Oxigen (0.2mbar, 100W, 41cc)  
etch Al in Al etch for 5min30sec+DI water + water tab

TEST100-Litho\_S1813onAl\_dose14p5sec\_dev55sec\_etch5min30secAetch\_1.JPG

TEST100-Litho\_S1813onAl\_dose14p5sec\_dev55sec\_etch5min30secAetch\_3.JPG

TEST100-Litho\_S1813onAl\_dose14p5sec\_dev55sec\_etch5min30secAetch\_4.JPG

TEST100-Litho\_S1813onAl\_dose14p5sec\_dev55sec\_etch5min30secAetch\_5.JPG

The result seems quite convincing ---> this wafer is called **BPCd10**

lundi 19/01

- **BCB step**

I put some BCB in the syringe as soon as possible to let all the possible bubbles go.

bake wafer 5' @ 110°C

spin AP3000 - 3" @ 1000rpm acc 300rpm/s; 60" @ 6000rpm ; bake 1' @

spin BCB4024-40 - 3" @ 1000rpm acc 300rpm/s; 60"@5000rpm ; bake 2'3

align layer 2 from mask with layer one

Expo 71mJ/cm<sup>2</sup> @ 365nm, Vacuum contact -> 6.5" @ 11mW

pre-bake wafer 30" @ 65°C before immediate development

Dev DS3000 at 31-32°C (very important) for 3min 30sec  
(previous check with test waffers), rinse ODI 60" beaker + 30" water

Result: oh fuck! The mask was inverted ! Alignment is right otherwise

[BCPtest\\_1.PNG](#)

[BCPtest\\_4.PNG](#)

**mardi 19/01**

- **removing BCB**

After the mistake with BPC5 (i tried to use AZ400 dev which etches Al and damages Si).

remove BCB in Remover 1165 @ 60°C for 20min + clean in DI H2O



I can observe the resist that leaves slowly (like in a lift off); I use short intervals of ultrasound to help this process.

- **BCB step (second time)**

bake wafer 5' @ 110°C

spin AP3000 - 10" @ 500rpm acc 250rpm/s; 60" @ 6000rpm ; bake 1' @ 110°C

spin BCB4024-40 - 10" @ 500rpm acc 250rpm/s; 60"@5000rpm ; bake 3'30 @90°

align layer 2 from mask with layer one (with the right orientation this t

Expo 71mJ/cm<sup>2</sup> @ 365nm, Vacuum contact -> 6.5" @ 11mW

pre-bake wafer 30" @ 65°C before immediate development

Dev DS3000 at 31-32°C (very important) for 4min

(previous check with test wafers), rinse ODI 60" beaker + 30" water tap

Result: NICE!

bake BPCD10 at 200°C for 1h.

### Test BCB

mardi 12/01

We prepare a little bottle with the new BCB4024-40. Since we take it out from the fridge, we have to wait for thermalization.

On standard Si/SiO<sub>2</sub> 500nm,

spin AP3000 - 3" @ 1000rpm acc 500rpm/s; 60" @ 6000rpm ; bake 1' @ 110°C

spin BCB4024-40 - 3" @ 1000rpm acc 500rpm/s; 60"@5000rpm ; bake 2'30 @90°

I guess that since there many bubbles in the bottle inside the fridge, they remain in the spinning. So **there are many bubbles after the spinning**;

I test the development time of the complete wafer. According to previous test fringes are gone at 1min30sec aprox. I first tried with the developer DS3000 at cleanroom temperature but the times are very long.

I re-start (take out bcb and re-spin) and try with the developer kept at 30°C (I had observed before that this was appropriate) and then **I obtain 1min25sec**. This would indicate that the development time is 3min;

expose BCB4024-40 with 50, 71 and 93mJ in vacuum contact using IEF mask;  
develop in DS3000 at 30°C for 3min (at this time I observe that the resist  
i.e. fringes disappear)

(50mJ dose - 3min dev)

[Litho\\_BCB\\_49mJ\\_3min\\_1.PNG](#)

A similar result can be observed in the optical microscope for the different doses.

**mercredi 13/01**

I cleave the wafer to observe part of it in the SEM.

[resume1.jpg](#)

In the SEM image it is clear that 3 min dev is fine to get well defined patterns but one more minute helps to get the right undercut. This undercut is more pronounced with less dose; After developing 6 min (3+3) the patterns are washed away.

jeudi 14/01

I bake the sample that was developed 3 min and the one that was developed 3+1

bake BCB4024-40 for 1h at 200°C

[resume2.jpg](#)

There are several combinations that are appropriate to do the third step after. **I think that 71mJ is the right dose and 3-4 min is OK.**

Once I have this process more or less established, I repeated the process in test wafer of standar Si ( Si/SiO<sub>2</sub> 500nm)

heat the wafer at 110°C for 5min to eliminate water and solvents  
spin AP3000 - 3" @ 1000rpm acc 300rpm/s; 60" @ 6000rpm ; bake 1' @ 110°C  
spin BCB4024-40 - 3" @ 1000rpm acc 300rpm/s; 60"@5000rpm ; bake 2'30 @90°C  
expose with doses 51mJ and 71mJ using IEF mask  
heat wafer at 65°C before develop  
develop 4 min in DS3000 @30°C

I cut the wafer in four (2 of 51mJ and 2 of 71mJ); I bake one of each 1h at 200°C. The observation in SEM is surprising: **The Result has nothing to do with the previous one**

[resume3.jpg](#)

What happened is that the **development time changed due to the aging of the resist.**

venerdi 15/01

The results are still not conclusive. I start with a new test wafer of standar Si ( Si/SiO<sub>2</sub> 500nm) and

heat the wafer at 110°C for 5min to eliminate water and solvents  
spin AP3000 - 3" @ 1000rpm acc 300rpm/s; 60" @ 6000rpm ; bake 1' @ 110°C  
spin BCB4024-40 - 5" @ 500rpm acc 250rpm/s; 60"@5000rpm ; bake 2'30 @90°C  
heat wafer at 65°C before develop  
develop unexposed wafer in DS3000 @30°C --> **fringes are gone after 3min5**

**OBS:** In agreement with Helenes recommendation, I should now take 6min10sec as the nominal dev time.

I spin resists again on the same wafer.

heat the wafer at 110°C for 5min  
spin AP3000 - 3" @ 1000rpm acc 300rpm/s; 60" @ 6000rpm ; bake 1' @ 110°C  
spin BCB4024-40 - 5" @ 500rpm acc 250rpm/s; 60"@5000rpm ; bake 2'30 @90°C  
expose four patterns: 50-71-93-115 mJ  
develop in DS3000 @30°C for 6min. I observe that the fringes disappear at  
clean water DI

I cut the wafer, bake the half of each chip and look at SEM

[resume4.jpg](#)

[resume5.jpg](#)

These results are more consistent with the previous findings. The conclusion is that much care has to be taken in how long has been the resist out of the fridge. It is convenient to perform a test of an unexposed wafer (from which and approximative dev time can be obtained) and a litho test from which this time can be verified. Taking 2 times the time at which the interference fringes disappear from the unexposed wafer seems to work quite fine.

### **Test spinning on BCB for third etape**

**lundi 18/01**

I add more BCB from the fridge to the little bottle and let it thermalize.

On two standard Si/SiO<sub>2</sub> 500nm BCB-TEST1 and BCB-TEST2,

heat wafer at 110°C for 10 min

spin AP3000 - 3" @ 1000rpm acc 300rpm/s; 60" @ 6000rpm ; bake 1' @ 110°C

spin BCB4024-40 - 3" @ 1000rpm acc 300rpm/s; 60"@5000rpm ; bake 2'30 @90°

Not so many bubbles.

I test the development time of the complete wafer.

develop BCB-TEST1 in DS3000 at 30°C --> approx 1min 40sec

As the resist has been just taken out from the fridge, the time is like at the beginning of the tests (mardi 12/01);

expose BCB-TEST2 dose 71mJ (6.5 sec at 11mW) in vac contact  
heat wafer at 65°C for 30sec previous to dev  
develop in DS3000 @30°C for 3min30sec. I observe that the fringes dissape  
clean water DI;

## IMAGES

bake BCB-TEST2 for 1h at 200°C

### **Test LOR+AZ5214 for third step**

jeudi 21/01

I want to test the litho process using LORXB +AZ5214E . We want to use it for the third step on our fab. We have some requirements: 1- it has to go up the BCB (max 4 microns) ; 2- we need good undercut since it is a lift-off process; 3- we need a good contact with the mask to define the motifs; 4- we need a development that does not damage the Al.

[spinLOR.jpg](#)

So, LOR5B is 500nm at 3000rpm and LOR20B is 2000nm; both are conformal which means that they follow the shape of what is below. This would mean that in one case we would have 500nm or 2000nm of LOR on top of the 4microns of BCB, respectively. AZ5214 is just 1.4 microns; I think it is better to test with LOR5B.

[bakeLOR.jpg](#)

With respect to the time and baking temperature(150-200°C), I think 170°C is reasonable (to get the largest undercut in the shortest time) during 5min;

- On a standard Si/SiO<sub>2</sub> 500nm, which I call **LORAZ-T1**

bake wafer 5' @ 110°C (solvent evaporation)  
spin LOR5B (per 12/01/2014) 60" @ 3krpm  
bake LOR5B 5min at 170°C  
spin AZ5214E (per 16/12/2014) 60" @ 4krpm  
bake 1'30 @ 110°C  
Expo 31mJ/cm<sup>2</sup> @ 365nm (2.8" @ 11mW), Vacuum contact  
bake 3' @ 120°C (=setpoint). (Increase time because resist perempted)  
Flood expo 27"  
Dev MIF726 55" @ 19.0°C, rinse ODI 60" beaker + 30" water tap

[resume\\_litho1\\_55.jpg](#)

It can be observed that some parts of the litho are gone due to the strong undercut. According to the measurements, the undercut was about 5 microns. I observed that the resist was gone after 30sec so in 25sec the undercut etch was then 5000nm/25s=200 nm/s (for a bake at 170deg C). I do 15 sec more to get a relative measurement

Dev MIF726 15" @ 19.0°C, rinse ODI 60" beaker + 30" water tap

[resume\\_litho1\\_55plus15.jpg](#)

After 15 sec the undercut has changed from 5.5 to 8.9 microns thus 230nm/s which is consistent with the previous result.

Remov Remover PG @ 60°C for 30min, rinse ODI 60" beaker + 30" water tap

Restart --> **change bake Temp for LOR5B** (this is suppose to reduce the rate for the undercut)

bake wafer 5' @ 110°C (solvent evaporation)

spin LOR5B (per 12/01/2014) 60" @ 3krpm

bake LOR5B **5min at 180°C**

spin AZ5214E (per 16/12/2014) 60" @ 4krpm

bake 1'30 @ 110°C

Expo 31mJ/cm<sup>2</sup> @ 365nm (2.8" @ 11mW), Vacuum contact

bake 3' @ 120°C (=setpoint). (Increase time because resist perempted)

Flood expo 27"

Dev MIF726 45" @ 19.0°C, rinse ODI 60" beaker + 30" water tap

[resume\\_litho2\\_45.jpg](#)

Result: I observe that resist is gone after 30 sec of development; The undercut is between 1.3 and 2 microns. the rate is then approx 100nm/s. The line of the TL is still there. I do 10sec more to compare with before;



Dev MIF726 10" @ 19.0°C, rinse ODI 60" beaker + 30" water tap

[resume\\_litho2\\_45plus10.jpg](#)

[resume\\_litho2\\_45plus10b.jpg](#)

Result: The rate is much slower;

- On a standard Si wafer, which I call **LORAZ-T2**

bake wafer 5' @ 110°C (solvent evaporation)

spin LOR5B (per 12/01/2014) 60" @ 3krpm

bake LOR5B 5min at 180°C

spin AZ5214E (per 16/12/2014) 60" @ 4krpm

bake 1'30 @ 110°C

Expo 31mJ/cm<sup>2</sup> @ 365nm (2.8" @ 11mW), Vacuum contact

bake 3' @ 120°C (=setpoint). (Increase time because resist perempted)

Flood expo 27"

Dev MIF726 40" @ 19.0°C, rinse ODI 60" beaker + 30" water tap

[resume\\_litho3\\_40.jpg](#)

Here the undercut is very small, I could not measure it; It is maybe just the limit time to develop the AZ

[resume\\_litho3\\_40b.jpg](#)

venerdi 22/01

I reprocess both test wafers

- On **LORAZ-T1**

remove resist in Remover PG @ 60°C for 30min

- On a standard Si wafer, which I call **LORAZ-T2**

remove resist in Remover PG @ 60°C for 30min

- **Etching rate of Al in MIF-726**

Hélène did an ion millig test on a wafer with 100nm of Al. After the etching, Al is gone in the center of the wafer but remains on the edges. I cut some pieces of this wafer to check the etching rate of Al in the developer we are using for AZ5214 which is MIF726.

I obtain the series of times: 1'40" , 1'10" , 2'05" , 1'20" , 2'40" , 2'30" depending on the width of the Al in the chips; if the longest time has 100nm, the rate is 0.6nm if not less, **rate <= 0.6nm/s**.

I want to do a test now of the lithography on Al and on BCB. I will prepare four wafers simultaneously: LORAZ-T1 and LORAZ-T2, a clean wafer with 100nm Al and BCB-TEST2.

- On **LORAZ-T1**

bake wafer 5' @ 110°C (solvent evaporation)  
spin LOR5B (per 12/01/2014) 60" @ 3krpm  
bake LOR5B 5min **at 170°C**  
spin AZ5214E (per 16/12/2014) 60" @ 4krpm  
bake 1'30 @ 110°C  
Expo 31mJ/cm2 @ 365nm (**2.8" @ 11mW**), Vacuum contact  
bake 3' @ 120°C (=setpoint). (Increase time because resist perempted)  
Flood expo 27"  
Dev MIF726 **35"** @ 19.0°C, rinse ODI 60" beaker + 30" water tap

[resume-litho4b.jpg](#)

Result: Litho is perfect, maybe to much undercut. In the other LORAZ-T2 I try just 30sec.

- On a standard Si wafer, which I call **LORAZ-T2**

bake wafer 5' @ 110°C (solvent evaporation)  
spin LOR5B (per 12/01/2014) 60" @ 3krpm  
bake LOR5B **5min at 170°C**  
spin AZ5214E (per 16/12/2014) 60" @ 4krpm  
bake 1'30 @ 110°C  
Expo 31mJ/cm2 @ 365nm (**2.8" @ 11mW**), Vacuum contact  
bake 3' @ 120°C (=setpoint). (Increase time because resist perempted)

Flood expo 27"

Dev MIF726 **30"** @ 19.0°C, rinse ODI 60" beaker + 30" water tap

[resume-litho5.jpg](#)

Result: This is the right time;

- On wafer with 100nm Al which I call **LORAZ-AL**

bake wafer 5' @ 110°C (solvent evaporation)

spin LOR5B (per 12/01/2014) 60" @ 3krpm

bake LOR5B **5min at 170°C**

spin AZ5214E (per 16/12/2014) 60" @ 4krpm

bake 1'30 @ 110°C

Expo 31mJ/cm<sup>2</sup> @ 365nm (**2.8" @ 11mW**), Vacuum contact

bake 3' @ 120°C (=setpoint). (Increase time because resist perempted)

Flood expo 27"

Dev MIF726 **30"** @ 19.0°C, rinse ODI 60" beaker + 30" water tap

[resum-lor-al-a.jpg](#)

Result: The undercut is much smaller;

I decide to add 10" more to the development

Dev MIF726 10" @ 19.0°C, rinse ODI 60" beaker + 30" water tap

[resum-lor-al-b.jpg](#)

Some motifs are much better resolved. Why we need more dev?

- On **BCB-TEST2**

bake wafer 5' @ 110°C (solvent evaporation)

spin LOR5B (per 12/01/2014) 60" @ 3krpm

bake LOR5B **5min at 170°C**

spin AZ5214E (per 16/12/2014) 60" @ 4krpm

bake 1'30 @ 110°C

Expo 31mJ/cm<sup>2</sup> @ 365nm (**2.8" @ 11mW**), Vacuum contact

bake 3' @ 120°C (=setpoint). (Increase time because resist perempted)

Flood expo 27"

Dev MIF726 **30"** @ 19.0°C, rinse ODI 60" beaker + 30" water tap

[resum-lorazBCB.jpg](#)

Result: It is hard to say whether AZ is on top of BCB; it seems that the resin on top of BCB is more "weak" and leaves easily with the developer. That would indicate that the dose on top of BCB is not enough for a good reticulation and crosslinking. This has to be tested.

We decide to add 10" more to the development

Dev MIF726 10" @ 19.0°C, rinse ODI 60" beaker + 30" water tap

The undercut in the Si part has increased a lot but we don't observe this undercut on top but on the contrary, just the fact that the pattern on top is larger than nominal.

We try another dose of flood exposure and 15 sec of dev

Flood expo 27"

Dev MIF726 **15"** @ 19.0°C, rinse ODI 60" beaker + 30" water tap

[resum-lorazBCB2.jpg](#)

Result...

We observe then at the profilometer and observe two things: 1-LOR+AZ gives almost 2microns width which is as expected. 2- The profil of BCB is very strange, like it had really spilled a lot. Which is not what I would expect according to the parameters.

remove resist in Remover PG @ 60°C for 30min

BCB does not seem to be damaged.

I check the profile in the stepmeter and got what was expected for the BCB. When we had done the same measurement but with the resin on top, the mountain was very extended and very tall, only 1.4 microns

lundi 25/01

I prepare three waffers with BCB to test the litho parameters in parallel with BCB-TEST2

- On **LORAZ-BCB1**

heat wafer at 110°C for 10 min

spin AP3000 - 3" @ 1000rpm acc 300rpm/s; 60" @ 6000rpm ; bake 1' @ 110°C

spin BCB4024-40 - 3" @ 1000rpm acc 300rpm/s; 60"@5000rpm ; bake 2'30 @90°C

Flood expose dose 71mJ (6.5 sec at 11mW) in vac contact

bake for 1h at 200°C

spin LOR5B (per 12/01/2014) 60" @ 3krpm

bake LOR5B 5min at 170°C

spin AZ5214E (per 16/12/2014) 60" @ 4krpm

bake 1'30 @ 110°C

Expo 4 times IEF mask: **31mJ/cm<sup>2</sup> (2.8" @ 11mW) - 46mJ/cm<sup>2</sup> (4.2" @ 11mW) - 62mJ/cm<sup>2</sup> (5.6" @ 11mW) - 77mJ/cm<sup>2</sup> (7.0" @ 11mW)** , Vacuum contact

bake 3' @ 120°C (=setpoint). (Increase time because resist preempts)

**Flood expo 45"**

Dev MIF726 **30"** @ 19.0°C, rinse ODI 60" beaker + 30" water tap

The pictures are organized from lower dose on top to higher dose in the bottom;

Left is 30sec develop and right is 30+10sec

resume-bcb1aa.jpg

resume-bcb1ba.jpg

resume-bcb1ab.jpg

resume-bcb1bb.jpg

This result indicates that a higher dose is necessary when there is BCB in the bottom; 5.6 s and 7.0 s give almost the same result. In addition, 30 sec seems reasonable.

- On **LORAZ-BCB2**



heat wafer at 110°C for 10 min  
spin AP3000 - 3" @ 1000rpm acc 300rpm/s; 60" @ 6000rpm ; bake 1' @ 110°C  
spin BCB4024-40 - 3" @ 1000rpm acc 300rpm/s; 60"@5000rpm ; bake 2'30 @90°  
Flood expose dose 71mJ (6.5 sec at 11mW) in vac contact  
bake for 1h at 200°C

spin LOR5B (per 12/01/2014) 60" @ 3krpm  
bake LOR5B 5min at 170°C  
spin AZ5214E (per 16/12/2014) 60" @ 4krpm  
bake 1'30 @ 110°C

- On LORAZ-BCB3

heat wafer at 110°C for 10 min  
spin AP3000 - 3" @ 1000rpm acc 300rpm/s; 60" @ 6000rpm ; bake 1' @ 110°C  
spin BCB4024-40 - 3" @ 1000rpm acc 300rpm/s; 60"@5000rpm ; bake 2'30 @90°  
Flood expose dose 71mJ (6.5 sec at 11mW) in vac contact  
heat wafer at 65°C for 30" previous dev  
**Dev DS3000 @31°C for 5min20"**  
bake for 1h at 200°C

spin LOR5B (per 12/01/2014) 60" @ 3krpm  
bake LOR5B 5min at 170°C  
spin AZ5214E (per 16/12/2014) 60" @ 4krpm  
bake 1'30 @ 110°C

Expo 4 times IEF mask: **31mJ/cm2 (2.8" @ 11mW) - 46mJ/cm2 (4.2" @ 11mW)**  
**- 62mJ/cm2 (5.6" @ 11mW) - 77mJ/cm2 (7.0" @ 11mW)** , Vacuum contact  
bake 3' @ 120°C (=setpoint). (Increase time because resist perempted)  
**Flood expo 45"**  
Dev MIF726 **30"** @ 19.0°C, rinse ODI 60" beaker + 30" water tap

The pictures are organized from lower dose on top to higher dose in the bottom;

Left is 30sec develop and right is 30+10sec

resume-lorazBCB3aa.jpg

resume-lorazBCB3ba.jpg

resume-lorazBCB3ab.jpg

resume-lorazBCB3bb.jpg

When the BCB has been developed and then baked, the previous result holds. A higher dose is still necessary and the same develop time leads to a similar result. I decided to use 7sec for the exposure and 30sec for the development.

- On **BCB-TEST2**

heat wafer at 110°C for 10 min  
spin LOR5B (per 12/01/2014) 60" @ 3krpm  
bake LOR5B 5min at 170°C  
spin AZ5214E (per 16/12/2014) 60" @ 4krpm  
bake 1'30 @ 110°C

align and expo with dose 77mJ/cm2 (7.0" @ 11mW), Vacuum contact  
bake 3' @ 120°C (=setpoint). (Increase time because resist perempted)  
Flood expo 45"  
Dev MIF726 30" @ 19.0°C, rinse ODI 60" beaker + 30" water tap

Result is not as I expected...

[resum-lorazBCB-litho2a.jpg](#)

[resum-lorazBCB-litho2b.jpg](#)

[resum-lorazBCB-litho2c.jpg](#)

mardi 26/01

I prepare a test with LOR30B instead;

- On **LORAZ-30B**

heat wafer in hotplate at 120°C for 20min to eliminate solvents and water  
spin TI primer 60sec at 4000rpm  
bake Ti primer at 120°C for 2min

spin LOR30B (per //) 60" @ 3krpm  
bake LOR30B 5min at 170°C  
spin AZ5214E (per 16/12/2014) 60" @ 4krpm  
bake 1'30 @ 110°C

Expo **62mJ/cm2 (5.6" @ 11mW)** , Vacuum contact  
bake 3' @ 120°C (=setpoint). (Increase time because resist perempted)  
**Flood expo 45"**  
Dev MIF726 **30"+10"+45"** @ 19.0°C, rinse ODI 60" beaker + 30" water tap

Development takes longer; LOR30B from Camille bottle maybe has some crystalized resin!!

[resume-LOR30B.jpg](#)

Remove remover PG @60°C for 30min

- On **BCB-TEST2**

Remove remover PG @60°C for 30min  
heat wafer in hotplate at 120°C for 10min  
spin TI primer 60sec at 4000rpm  
bake Ti primer at 120°C for 2min

spin LOR30B (per //) 60" @ 3krpm  
bake LOR30B 5min at 170°C  
spin AZ5214E (per 16/12/2014) 60" @ 4krpm  
bake 1'30 @ 110°C

Expo 62mJ/cm2 (5.6" @ 11mW) , Vacuum contact  
bake 3' @ 120°C (=setpoint). (Increase time because resist perempted)  
Flood expo 45"  
Dev MIF726 **1min** @ 19.0°C, rinse ODI 60" beaker + 30" water tap

[litho3\\_60sec\\_1.PNG](#)

I was not well aligned... this will reflect after the lift

Evapotate 300nm Al @ 1nm/s in old evap

We cut a piece of the wafer to observe the undercut in SEM and we remove the resine for the rest

loraz\_1.JPG

loraz\_2.JPG

loraz\_4.JPG

loraz\_6.JPG

[loraz\\_7.JPG](#)

[loraz\\_8.JPG](#)

Lift-off resist in Remover PG @60°C for 1h+ultrasound (not a good idea)

mercredi 27/01

- On **PMGIAZ-T1 (standard non oxidized Si wafer)**

I prepare a test with PMGI -SF8 ; this resist is 1 micron width at 1000rpm ( I will spin 3 times )

heat wafer in hotplate at 120°C for 20min to eliminate solvents and water  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 215°C (in oven)  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 217°C  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"



bake 5' @ 217°C  
---After Check of profile in **ALTEST**  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 210°C  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 212°C  
---After Check of profile in ALTEST  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 218°C  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 214°C  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 218°C

----  
spin AZ5214E (per 16/12/2014) 60" @ 4krpm  
bake 1'30 @ 110°C

- **On ALTEST- BPCd10**

heat wafer in hotplate at 120°C for 20min to eliminate solvents and water  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 215°C  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 217°C  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 217°C

---Check profile in profilometer; There is still 1micron of height difference

spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 210°C  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 212°C

---check profile, now there is 0.5micron difference  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 218°C  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 218°C  
spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 1000rpm 60"  
bake 5' @ 218°C

---check profile, **it seems very flat**

spin AZ5214E (per 16/12/2014) 60" @ 4krpm  
bake 1'30 @ 110°C

The good news is that it is possible to planarize. The bad news is that it is very annoying to spin and bake 8 times. We have to get PMGI-SF11 or SF15. The other negative problem is that I had some cristalized ugly residues in the border of the wafer. I don't know the origin but I think it has to due with the oven.

Due to this border it was impossible to try the lithography so I decided to remove the resist in Remover PG

- On **PMGIAZ-T1**

Remove resist Remover PG @60°C + clean water

- On **ALTEST- BPCd10**

Remove resist Remover PG @60°C + clean water

When I was taking out the wafer from the water I accidentally cleave it. So ..... I'm the king of fucking it up.-

### **Fabrication of wafer for junction ox and bilayer contact resistance tests**

mardi 12/01

New wafer Si-P batch rho=1-10ohm.cm, 100nm oxide

expo with [2015-01\\_JJox + cap](#) mask

- Negative resist :

bake wafer 1' @ 110°C (solvent evaporation)

spin AZ5214E (per 16/12/2014) 60" @ 4krpm

bake 1'30 @ 110°C

Expo 31mJ/cm<sup>2</sup> @ 365nm (2.8" @ 11mW), Vacuum contact

bake 3' @ 120°C (=setpoint). (Increase time because resist perempted)

Flood expo 27"

Dev MIF726 50" @ 19.0°C, rinse ODI 60" beaker + 30" water tap

- Residue removal

1' ozone plasma @ 100W, 200µb

mercredi 13/01

- Evaporation Ti/Au

old plassys

5nm Ti @ 0.1 nm/s

50nm Au @ 1nm/s

- Lift warm acetone, 20min

perfect

- PMGI/PMMA Bilayer spin

bake 120°C 10'

spin TI prime @ 4000rpm 60"

bake 120°C 1'

spin PMGI SF8 (batch 12060407, exp. 7/1/2013) @ 3000rpm 60"

bake hot plate setpoint 180°C, 5', under beaker (measured: 159.6°C, same

spin PMMA A6 (batch 14020130, exp. 3/1/2015) @ 6000rpm 60"

bake hot plate setpoint 180°C, 15', under beaker

spin UV III (viven bottle) @ 4000rpm 60"

bake hot plate 140°C, 90"

- Dice on scriber

OK, only a few samples lost

### Tests for making contact on oxidized Al layer

I take the same drawing that I will use for JJox tests. In the center I also put some dose tests for the junctions

[JJox\\_mask-dose.jpg](#)

WJOX1\_1

vendredi 15/01

exp 30keV, 300 $\mu$ C/cm<sup>2</sup>

!/\ during expo, the SEM diffusion pump was making noise (shot noise)

[dev standard for PMGI/PMMA bilayer](#) (1'30 MIBK, 1' MIF 726)

[WJOX1\\_1\\_dev-std.jpg](#)

(NB: for this first chip, I forgot to put correct contact pads on the bottom JJ)

lundi 18/01

- evap 1st layer

Al 30nm @ 1nm/s, 0°

- oxidation

2h on hot plate 110°C under beaker

mardi 19/01

- etch + evap 2nd layer

etch 500V, 65mA, 2'

Al 50nm @ 1nm/s, 35°

- lift

30' in remover PG @ 60°C

3" US at the end, when the layer is fully lifted

[WJOX1\\_1\\_sem.jpg](#)

WJOX1\_2

lundi 18/01

exp 30keV, 300μC/cm2

!/ I waited for a quiet moment of the SEM diffusion pump to launch exposition

dev standard

[WJOX1\\_2\\_dev-std.JPG](#)

- evap 1st layer (old plassys)

Al 30nm @ 1nm/s, 0°

- oxidation

one night: from 16h on monday to 11h on tuesday

- etch + evap 2nd layer

no etch

Al 50nm @ 1nm/s, 35°

- lift

20' in remover PG @ 60°C

3" US at the end, when the layer is fully lifted

[WJOX1\\_2\\_sem.jpg](#)

WJOX1\_3

lundi 18/01

exp 30keV, 300 $\mu$ C/cm<sup>2</sup>

/!\ I waited for a quiet moment of the SEM diffusion pump to launch exposition, but it turned back on during expo, close to the end; probably junctions were finished but not center patterns.

dev standard

[WJOX1\\_3\\_dev-std.JPG](#)

mardi 19/01

- clean resist residues

30" ozone plasma, 200 $\mu$ b (40cc O<sub>2</sub>), 100W

- evap two layers (new plassys)

Al 30nm @ 1nm/s, 0°

Al 50nm @ 1nm/s, 35°

- lift

20' in remover PG @ 60°C

3" US at the end, when the layer is fully lifted

[WJOX1\\_3\\_sem.jpg](#)

### **CONCLUSION OF WJOX1\_1-2-3:**

it seems that the etching damages a lot the mask, even the simple ozone plasma

Or maybe the litho is not reproducible

The noise on the pump might be affecting a bit the deposited dose (see comparison of pattern center 1 for WJOX1\_2: dose OK and \_3: underdosed)

WJOX1\_4

mercredi 20/01

Design was slightly modified for the doses of the connecting pads, and the width of the island

Also, I added a new design to test junctions in line without having an overlap of the island



JJox\_mask\_dose\_2016-01-20.jpg

JJox\_mask\_dose\_zoom\_2016-01-20.jpg

exp 30keV, 300 $\mu$ C/cm<sup>2</sup>

/!\ I waited for a quiet moment of the SEM diffusion pump to launch exposition  
dev 30" in MIF726 instead of 1' -> undercut is  $\sim$ 1 $\mu$ m instead of 2 $\mu$ m

WJOX1\_4\_dev-30".JPG

**NO CLEANING**

- evap 1st layer (new plassys)

Al 30nm @ 1nm/s, 0°

jeudi 19/01

Oxidation 1 night (from 18h mercredi to 10h jeudi)

- etch + evap two layers (old plassys)

etch Ar: Vbeam= 532V, Vacc=32V, Ibeam = 5mA, P=3e-4mb, **50"**, 0°

Al 50nm @ 1nm/s, 35°

- lift

3h in remover PG @ 60°C

3" US at the end, when the layer is fully lifted

WJOX1\_4\_sem.jpg

WJOX1\_5

mercredi 20/01

exp 30keV, 300 $\mu$ C/cm<sup>2</sup>

/!\ I waited for a quiet moment of the SEM diffusion pump to launch exposition, but some people were there and talked in the SEM room

dev 30" in MIF726 instead of 1' -> undercut is still  $\sim 1\mu\text{m}$  instead of  $2\mu\text{m}$   
[WJOX1\\_5\\_dev-30".JPG](#)

jeudi 19/01

- evap 1st layer (new plassys)

Al 30nm @ 1nm/s,  $0^\circ$

Oxidation from jeudi 11h to 17h

- etch + evap 2nd layers (old plassys)

etch Ar: Vbeam= 532V, Vacc=32V, Ibeam = 5mA, P=3e-4mb, **2'**,  $0^\circ$

Al 50nm @ 1nm/s,  **$37^\circ$**  (should give  $\sim 25\text{nm}$  more displacement)

- lift

20' in remover PG @  $60^\circ\text{C}$

3" US at the end, when the layer is fully lifted

WJOX1\_5\_sem.JPG

**WJOX1\_6**

Put the face to face junctions closer to each other

Modify design on B to make it less sensitive to ion etching -be sure that the connection

wire is still conducting  
[JJox\\_mask\\_dose\\_2016-01-26.jpg](#)

NB: For \_6 and \_7, I made a mistake, the spacing was smaller (was 280nm on the left, and 330 on the right, I think)

mardi 26/01

- exp 30keV, 300 $\mu$ C/cm<sup>2</sup>

/!\ I waited for a quiet moment of the SEM diffusion pump to launch exposition,  
The last 15 patterns are exposed with noise  
dev 30" in MIF726

mercredi 27/01

- evap 1st layer (old plassys)

Al 30nm @ 1nm/s, 0°

- Oxidation from mercredi 12h to jeudi 16h

jeudi 28/01

- etch+ evap 2nd layer (old plassys)

20160128\_wjox1\_6.jpg

50nmAl\_Al2O3\_18'.JPG

etch: 532 Vbeam -32V acc, 5mA, 5'30  
Al 50nm @ 1nm/s, +35°

- lift

1h in remover PG @ 60°C  
3" US at the end, when the layer is fully lift

WJOX1\_6\_sem.jpg

The mask was badly injured by ion milling, but there remains on most patterns a gap, so the contact resistance can be measured. It seems that the Al layer was not etched away, even at tiny places (it could be that etching is more uniform than I feared)  
Contact resistance is perfect. We could recover the same values as unoxidized chip (WJOX1\_3)

WJOX1\_7

mardi 26/01

- exp 30keV, 300 $\mu$ C/cm<sup>2</sup>

! I waited for a quiet moment of the SEM diffusion pump to launch exposition,  
All patterns are exposed during quiet time  
dev 30" in MIF726

mercredi 27/01

- evap 1st layer (old plassys)

Al 30nm @ 1nm/s, 0°

- Oxidation from mercredi 12h to mardi 02/02 12h30
- etch + evap 2nd layer (new plassys)

etch 500V 130mA 1'20 (no spinning)

Al 50nm @ 1nm/s, 35°



Note that after etch, I notice the Al layer is transparent (either gone or oxidized)

- lift

15' in remover PG @ 60°C

5" US at the end -> small residues. Layer was not fully lifted, but I was

[WJOX1\\_7\\_sem.JPG](#)

The mask was badly injured by ion milling, but there remains a gap.  
Contact resistance is >1GOhm on all!  
Not enough milling, or too much???

WJOX1\_8

mardi 20/01

- exp 30keV, 300μC/cm<sup>2</sup>

!/ I waited for a quiet moment of the SEM diffusion pump to launch exposition,  
Almost all patterns are exposed with noise  
dev 30" in MIF726

mercredi 27/01

- evap 1st layer (old plassys)

Al 30nm @ 1nm/s, 0°

- Oxidation from mercredi 12h to mardi 02/02 16h30

- etch + evap 2nd layer (new pclassys)

etch 500V 130mA 1'40 (no spinning)  
Al 50nm @ 1nm/s, 35°

Note that after etch, I notice the Al layer is transparent (either gone or oxidized)  
[wjox1\\_8\\_etched.jpg](#)

- lift

1h in remover PG @ 60°C  
3" US at the end when layer is fully lifted

[WJOX1\\_8\\_sem.JPG](#)

probe station: Could get a perfect contact back!

WJOX1\_9

**This one is meant for determining if the etch is homogeneous, and if 1'40 is sufficient to take contact on the edge of the wafer**

09/02

exp 30keV, 300 $\mu$ C/cm<sup>2</sup>

/!\ I waited for a quiet moment of the SEM diffusion pump to launch exposition,  
But all patterns were exposed with noise

dev 30" in MIF726

[WJOX1\\_9\\_dev-30".JPG](#)

- evap 1st layer (old plassys)

Al 30nm @ 1nm/s, 0°

- Oxidation from mardi 09/02 15h to mercredi 24/02 15h
- etch + evap 2nd layer (new plassys)

[wjox1\\_9\\_init.jpg](#)

etch 500V 130mA 1'40 (spinning 16°/s)  
Al 60nm @ 1nm/s, 35°

- lift (25/02)

20min in remover PG @ 60°C  
3" US at the end when layer is fully lifted

[WJOX1\\_9\\_sem.jpg](#)

probe station: Could get a perfect contact back, even though the mask was not deformed!!

#### Summary

**evap 1st layer:** 30nm @ 0°

**evap 2nd layer:** 50nm @ 35°

**etch process:** 500V, 5mA in old evap -- 520V, 130mA, 16°/s planetary rotation in new evap

**measurement setup:** my own polar box, with different bias resistor to choose to limit current, and a convenient potentiometer to progressively apply bias.

sample	oxy (h)	etch time (s)	evap	RA (Ω)	SA $10^{-3}\mu\text{m}^2$	nb sq A	RB (Ω)	SB $10^{-3}\mu\text{m}^2$	nb sq B	RC (Ω)	SC $10^{-3}\mu\text{m}^2$	nb sq C	RD (Ω)	SD $10^{-3}\mu\text{m}^2$	nb sq D	average R <sub>contact</sub> (Ω* $\mu\text{m}^2$ )	comment
WJOX1_2	15	0	old	**	19.6		**	20		**	141.4		**	55.4		**	
WJOX1_3	no	0	old	24.8	23.9		23.6	22		19.9	174.3		23.7	62.3		1.5 +/- 1.37	
WJOX1_4	15	50	old	**	7		**	8.2		**	126.4		**	41.2		**	side of ion beam
WJOX1_5	6	120	old	**	6.7		**	7.5		**	105.5		**	39.2		**	side of ion beam
WJOX1_6	28	330	old	67.8	18		16.5	10		43.7	73.5		3.3*	*		1.5 +/- 1.5	max of ion beam

sample	oxy (h)	etch time (s)	evap	R <sub>A</sub> (Ω)	S <sub>A</sub> 10 <sup>-3</sup> μm <sup>2</sup>	nb sq A	R <sub>B</sub> (Ω)	S <sub>B</sub> 10 <sup>-3</sup> μm <sup>2</sup>	nb sq B	R <sub>C</sub> (Ω)	S <sub>C</sub> 10 <sup>-3</sup> μm <sup>2</sup>	nb sq C	R <sub>D</sub> (Ω)	S <sub>D</sub> 10 <sup>-3</sup> μm <sup>2</sup>	nb sq D	average R <sub>contact</sub> (Ω*μm <sup>2</sup> )	comment
WJOX1_7	144	80	new	**	10.1		**	14.8		**	100.9		**	56.7		**	max of ion beam (center of plate)
WJOX1_8	148	100	new	5*	*		12.6	18.8		24	97.6		27.8	59.9		1.4 +/- 1.07	max of ion beam
WJOX1_9	360	100	new	90	1	21	56.7	1	17	59.3	111.1	18	74.2	40	18		edge of wafer

\* Shorted on SEM

\*\* OL on the 4W fluke (limited to 1GΩ). To be measured again, with high enough voltmeter internal impedance, and with higher bias resistance to make a proper division bridge.

\*\*\* Open on SEM

NB: for all before WJOX1\_9 should revise the analysis accounting for number of squares and square resistance in series with the contact (if contact is perfect, what we measure should be the sheet resistance of the Al wire \* number of squares).

#### discussion avec Marco sur les interfaces Al - métal noble:

facteur de stoner Pd : pair breaking important. bande d, paramagnons, susceptibilité magnétique non nulle. Etats dans le gap.

idem Pt.

Raisonnable d'estimer T<sub>c</sub> avec toute la DOS de la bande d.

Al-Pd nanotubes. Avait l'air de garder Al supra. 5nm OK.

As: chauffe à 200-250°C dans evap, et s'en va. Utilisé pour protéger.