8. Appendix

8.1. Run sheets

During this thesis were performed six runs. Unfortunately, two of them couldn't be carried out until the end. In run #1, after performing the first etch the 6-inch wafer showed signs of corrosion, and the film seemed to start pilling off, that's why that run was terminated. This was an important drawback; since junctions take time to be deposited, plus the machines not always are available to deposit junctions since other works are been carried out at same time. In the run #5 the junction's magnetic properties were not right. There was problem with one of the targets during the deposition. The results obtained are mostly from the run #2 and #4. The 6-inch junction TJ1268 was processed in the run #2 and the samples N2TJ68 were processed in the run #4.

Run Sheet: # 1 **Responsible:** André Augusto

Structure	**Sample: GTJ301 (on 6-inch Si wafer)**
Junction Structure	Ta (30Å) / Ru (150Å) / CoFeFe (150Å) / MnIr (150Å) / CoFeFe^7 (40Å) / (Mg (5Å) w/o Oxidation) x 1 + (Mg+ox (5Å)) x 5 / CoFeFe^7 (50Å) / Ta (100Å)
Barrier Oxidation Conditions	5x8.0 sccm(O2); 2.2x10^-4 Torr (Cryo Hv Open); 500"/step ; 200" Mg preclean/step with wafer in the loadlock
Deposition Conditions	Mg deposited using +7mA; +746V; CoFe1 :+25mA; +1280V; CoFe2,3 :+15mA; +998V Remaining layers: +22mA; +1200V

Deposition Conditions ($TiWN_2$)			**Deposition Time:** 30s
N7000	**Power (W)**	**Gas Flow (sccm)**	**Pressure (mTorr)**
Setpoint	70 40	50	3
Read	69 39	50.2	3.1

Deposition Conditions (Al_2O_3)			**Deposition Time:** 32 min
UHV2	**Power (W)**	**Gas Flow (sccm)**	**Pressure (Torr)**
Setpoint	200	45	-
Read	200-199	44.9	4.0×10^{-3}

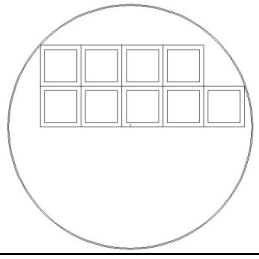
STEP 0 **Junction Deposition (A) + Passivation Layer (B) + Oxide layer (C)**

(A) (B) Responsible: Susana Freitas **(A) Machine:** Nordiko 3000 **(B) Machine:** Nordiko 7000

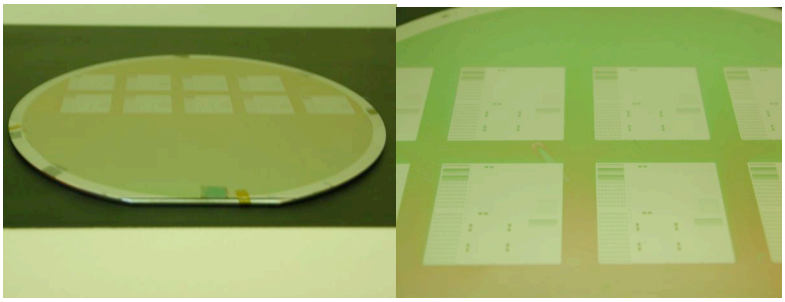
(**C) Responsible:** Fernando Silva **Machine:** UHV2

STEP 1.1. **1st Exposure – Bottom electrode definition** Date: 24 /04 / 07

Coating PR: 1.5 μm PR (Recipe 6/2) **Developer :** Recipe 6/2

Machine: DWL **Mask Name: etchL1**	**X=-56000 , Y=0.0**
- Cross Center: X=168 , Y=54 - Die: 20000 x 20000 μm - Die Frame: 25400 x 25400 μm - Vapor Prime: 5' recipe-0 - Photo Resist: 1.5 μm - WAFER6.map/dwl/wa/fa	

Optical Inspection:



STEP 1.2.	**1st Ion Milling –** Total Structure Etch	Date: 25/04 /2007

Machine: N3600 **Batch:** Junction_etch

Wafer recipe: etch_function_stack / (etch_pan 60 deg (300s) and cool-down 200s)$_{x4}$

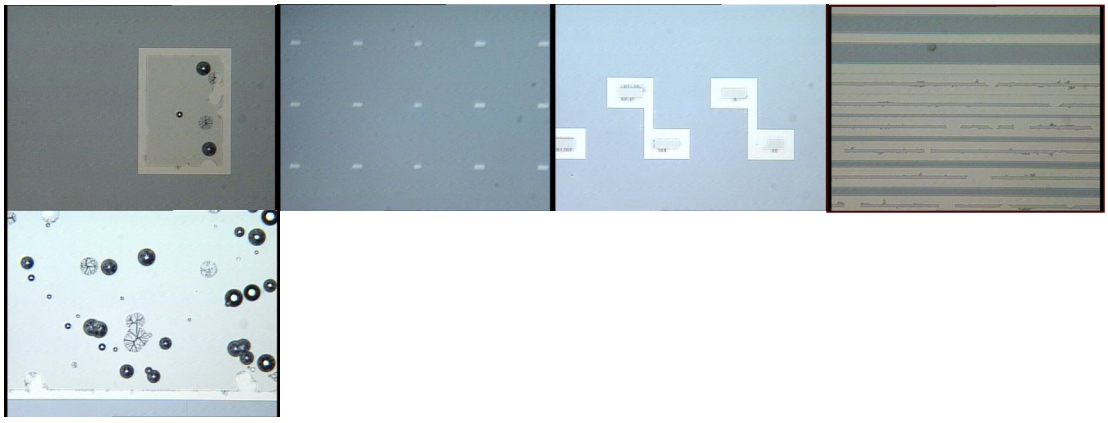
Etching Conditions:

Assist Gun: 170 W +725 V/-345 V 10 sccm Ar
Assist Neut: 364 V 3 sccm Ar
Sub Rotn: 30 rpm
Sub Pan: 60 deg

STEP 1.3	**Resist Strip**	Date: 26 / 04/ 2007

Hot Micro-Strip + Ultrasonic
Rinse with IPA + DI water
Acetona bath

Optical Inspection:



Comments:

The sample seems to show signs of corrosion and the film is pilling off. The process has to be stopped.

Run Sheet: # 2 **Responsible:** André Augusto

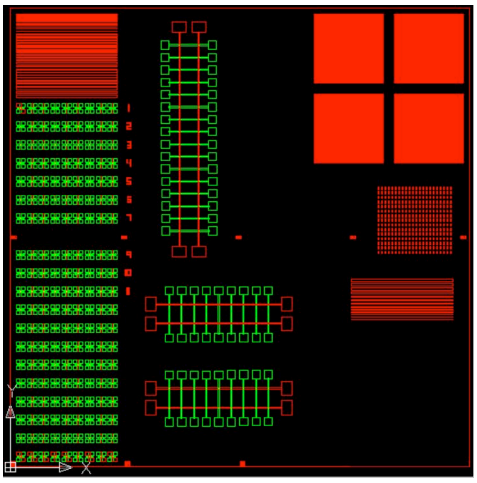
Structure	**Junction TJ1268 (6-inch size)**
200'' Etch 30º//Ta (30Å) / Ru (150Å)/ CoFeFe[a] (150Å)/ MnIr (150Å)/ CoFeFe[b] (40Å) / Ru (8Å) / CoFeB20 (50Å) / (Al (5Å) + ox rem. plasma) /CoFeB20 (50Å) / Ru (100Å) / TiWN (150Å)	
Substrate = Si 6-inch/Al_2O_3 1000 Å /Al 600 Å heated *a deposited with 25 mA* b deposited with 15 mA CoFeFe = $Co_{56}Fe_4$	
Oxidation Conditions Spot_a/Spot_b/Spot_c/Spot_1 (**5"**)	110W; +0V/-0V; 5x8.0 sccm(O2); 2.2x10^-4 Torr (Cryo Hv Open)

STEP 0.A **Junction Deposition** Date: 3 / 12 / 2006

Responsible: Susana Freitas

Machine: Nordiko 3000

STEP 0.B **CAD design and mask conversion**



Micron-size (lithography to be done by DWL)

- Minimum feature size: 1 μm
- Line/rectangle width from 1 to 100 μm

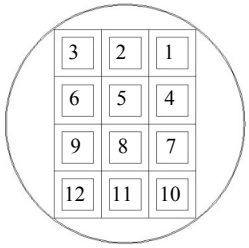
To measure the magnetic properties by VSM: Matrix of small rectangles: 3μm x 6μm

Conversion step

L1 – Bottom Electrode - Inverted
L2 – Junction Pillar - Inverted
L3 – Top Electrode - Not Inverted

STEP 1.1. **1st Exposure – Bottom electrode definition**

Coating PR: 1.5 μm PR (Recipe 6/2) **Developer :** Recipe 6/2

Machine: DWL **Mask Name:** aaEtchL1	**X=-45000 , Y=0.0**
- Cross Center: X=168 , Y=54 - Die: 20000 x 20000 μm - Die Frame: 30000 x 30000 μm - Vapor Prime 5 min. (Recipe - 0) - Energy: 35 % - Focus: 35 - Photo Resist: 1.5 μm - WAFER6PA.map/dwl/wa/fa - Development time : 60 sec	

Comments: All structures are OK

STEP 1.2. **1st Ion Milling –** Total Structure Etch

Machine: N3000 **Batch:**
Junction_etch

Wafer Recipe: etch-gun-stab / junction_etch / end etch

Read	
Assist Gun	60W / +500 V / -200 V / 8 sccm Ar
Assist Neutralizer	Not need
Sub Rotn	40% (30 rpm)
Sub Pan	70 degs

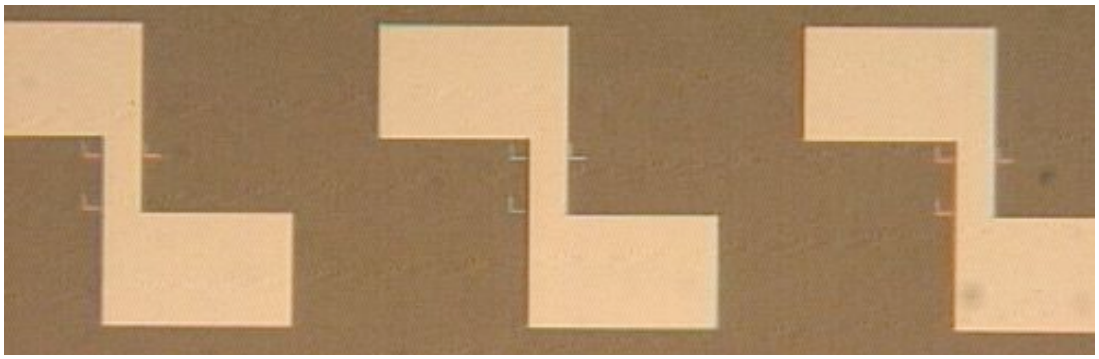
Time: 1600s

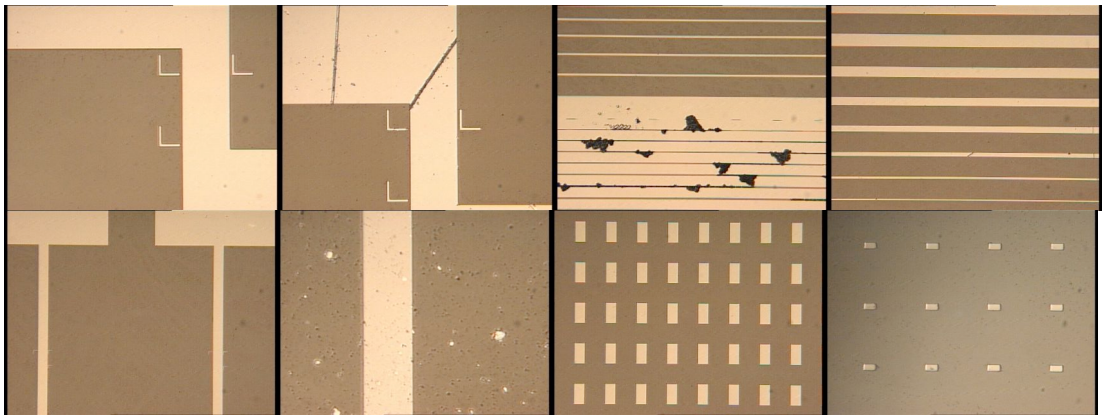
STEP 1.3 **Resist Strip**

Hot Micro-Strip + Ultrasonic
Rinse with IPA + DI water

Total Time in Hot Micro-Strip : some hours Ultrassonic Time : few

Optical Inspection:



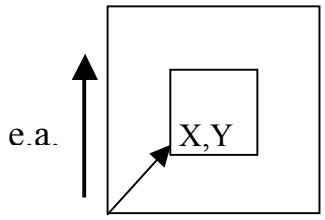


Comments: All dies are OK. Inside the dies the majority of the features are also OK

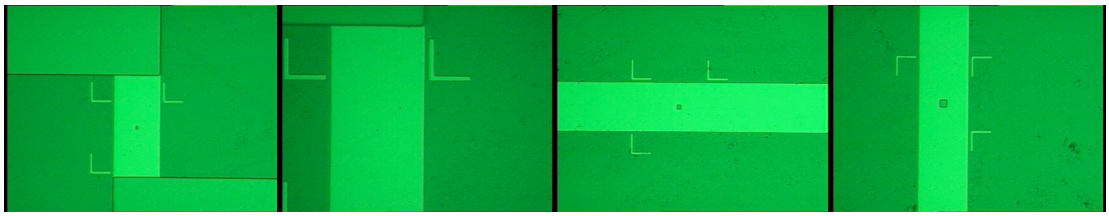
Aim
Study the impact of oxidation on redeposition

STEP 2.1 2nd Exposure – Junction area definition: Samples 4,5,6,7

Coating PR: 1.5 μm PR (Recipe 6/2) **Developer :** Recipe 6/2

Machine: DWL	**Mask Name: aaEtchL2**
- Cross Center: X=168 , Y=54 - Vapor Prime 5 min. (Recipe - 0) - Energy: 30 % - Focus: 35 - Photo Resist: 1.5 μm - Development time: 55” - WAFER6PA.map/dwl/wa/fa	

Optical Inspection:



The 1 μm size features seem smaller and in some samples they simple don’t exist. Maybe do the exposure energy or development time.

STEP 2.2 2nd Ion Milling – Junction area definition Date: 16 / 01 /07

Machine: N3600

Batch: Junction_etch

Wafer recipe: etch_junction_top_electrode + O2*

Process Step: Load wafer at 60º / etch pan 60 deg(**300 s**) / etch pan 30 deg (**100s**) / **oxidation beam (x s)*** / end_junction_etch

Samples	Oxidation Beam time (x /s)
6*	0
4	20
5	50
7	300

* The wafer recipe and process step don’t contain the red part. The sample #6 is etched with the standard conditions.

P=1,8E-4 Torr **T**=21ºC **Sub Rotn**: 30 rpm

Sample	Step	Read
6	60 deg	**Assist Gun**: 135W(250W) /1W/ 724V/ 35,9mA (48,7mA)/ 345V/ 0mA/10,2sccm **Assist Neut**: 0mA/ 398,4V/ 3,0 sccm
	30 deg	**Assist Gun**: 142W/2W/ 724,3V/ 104,8mA / 345V/ 2mA/10,1sccm **Assist Neut**: 183mA/ 345,6V/ 3,0 sccm
4	60 deg	**Assist Gun**: 199W/2W/ 725V/ 104,5mA/ 344,8V/ 0mA/10,2sccm **Assist Neut**: 178mA/ 344V/ 3,0 sccm
	30 deg	**Assist Gun**: 134W/2W/ 724,3V/ 104,6mA/ 345V/ 2,3mA/10,2sccm **Assist Neut**: 47mA/ 318,3V/ 3,0 sccm
	O2 beam	**Assist Gun**: 145W/1W/101V/ 45mA / 350V/ 2sccm Ar/ 20 sccm O2 **Sub Pan:** 0 deg **Shutter Open:** 20"
5	60 deg	**Assist Gun**: 187W/2W/ 724,3V/ 104,3mA/ 344,8V/ 0mA/10,2sccm **Assist Neut**: 176mA/ 344V/ 3,0 sccm
	30 deg	**Assist Gun**: 125W/1W/ 725V/ 104,2mA/ 344,8V/ 2,2mA/10,2sccm **Assist Neut**: 116,5mA/ 319,5V/ 3,0 sccm
	O2 beam	**Assist Gun**: 147W/1W/101V/ 45mA / 345V/ 1,1mA/2,1sccm Ar/ 20 sccm O2 **Sub Pan:** 0 deg **Shutter Open:** 50"
7	60 deg	**Assist Gun**: 239W/2W/ 724V/ 107,3mA/ 344,8V/ 0,2mA/10,2sccm **Assist Neut**: 177mA/ 342V/ 3,0 sccm
	30 deg	**Assist Gun**: 162W/1W/ 724,3V/ 105mA/ 344,8V/ 2,7mA/10,2sccm **Assist Neut**: 117mA/ 318,3V/ 3,0 sccm
	O2 beam	**Assist Gun**: 145W/1W/101V/ 45mA / 345V/1.3mA/ 2,2sccm Ar/ 20,1 sccm O2 **Sub Pan:** 0 deg **Shutter Open:** 300"

STEP 3.1. Insulating Layer Deposition- 500Å of Al_2O_3

Machine: UHV2	Power /W	Gas Flow /sccm	P /Torr
Setpoint	200	45	3,3E-3
Read	200	45	
Time	27 min		

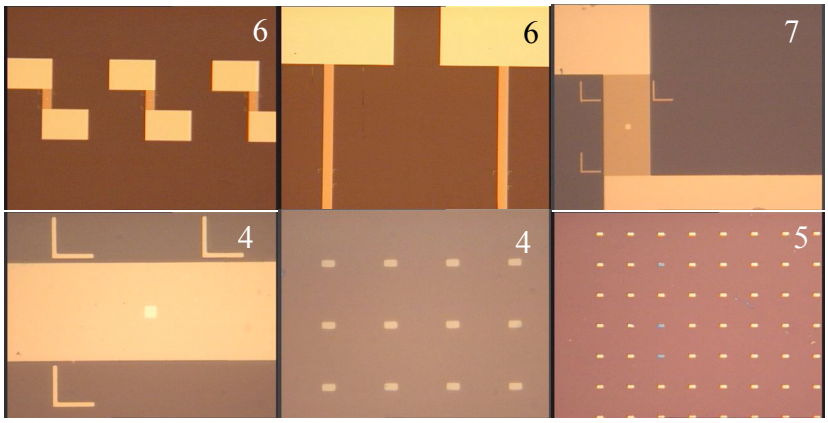
Thickness verification

Ellipsometer: n=1,64 t = 491 A
Perfilometer: t = 500 A

STEP 3.2. Oxide Lift-Off

Hot u-strip + ultrasonic
Rinse with IPA + DI water **Time:** 24h

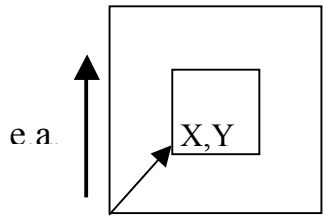
Optical inspection:



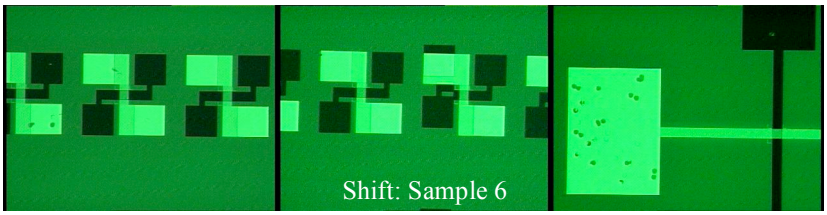
The features bigger than 2x1 or 3x1 seem OK. However, the ones that are smaller than the previous dimensions are close or are not seen on microscope (see visual inspection paper on run sheet). After etching and liftoff the samples present several kind of bubbles on the pads (except sample 6).

STEP 4.1 3rt Exposure – Top electrode metallization

Coating PR: coat 1.5 μm PR (Recipe 6/2) **Developer :** Recipe 6/2

Machine: DWL	**Mask Name: aaEtchL3**
- Cross Center: X=168 , Y=54 - Vapor Prime 5 min. (Recipe - 0) - Energy: 35 % - Focus: 35 - Photo Resist: 1.5 μm - Recipe: 6/2 - WAFER6PA.map/dwl/wa/fa	

Optical inspection:



Comments: The majority is OK. Few are bad.

STEP 4.2 Contact Leads Deposition (AlSiCu) Date: 25 / 01 /07

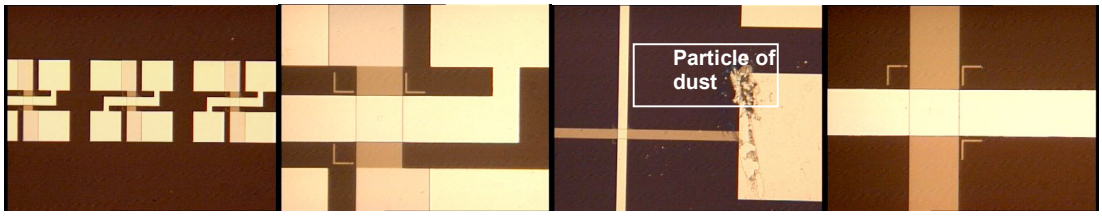
Machine: **Nordiko 7000**

Sequence 48

Module 2:	F9, 60” F69, 30W, ROB99, 50.2sccm, 3mT
Module 4:	F1, 120”, 3000 Å, AlSiCu 2.0kW, 410V, 4.9A, 50.4sccm, 3.0mT
Module 3:	F19, 27”, 150Â, $TiWN_2$ 0.5 kW, 431V, 1.2A, 50.65sccm, 3.1mT, 10scc

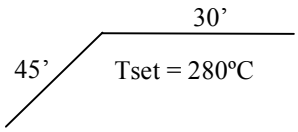
STEP 4.3 AlSiCu Liftoff

Hot μ-strip + ultrasonic
Rinse with IPA + DI water



STEP 5. **Annealing** Date 28 /02 / 07

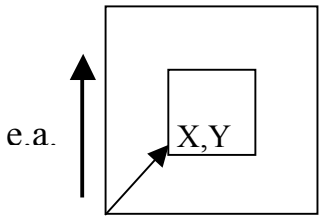
Machine: big annealing setup **Annealing conditions**: 30 min@280ºC



Aim
Study the impact of the etching angle variation

STEP 2.1 **2nd Exposure – Junction area definition: Samples 3,8,9,11**

Coating PR: 1.5 μm PR (Recipe 6/2) **Developer :** Recipe 6/2

Machine: DWL	**Mask Name: aaEtchL2**
- Cross Center: X=168 , Y=54 - Vapor Prime 5 min. (Recipe - 0) - Energy: 30 % - Focus: 35 - Photo Resist: 1.5 μm - Development time: 55" - WAFER6PA.map/dwl/wa/fa	

Observations:

#8	all junctions are there, the 1x1 seem little bit small
#9	all junctions are there, the 1x1 seem better than #8
#11	same as #9
#3	all junctions are there, the 1x1 seem a little bit big

STEP 2.2 **2nd Ion Milling –** Junction area definition Date: 27 / 01 /07

Machine: N3600 **Batch:** Junction_etch

Wafer recipe: etch_junction_top_electrode

P=1,8E-4 Torr **T**=21ºC **Sub Rotn**: 30 rpm

Sample #11	**#9**	**#8**	**#3**
Sub pan 30º (400 s)	**Sub pan 60º (300 s)** **Sub pan 40º (100 s)**	**Sub pan 60º (300 s)** **Sub pan 50º (100 s)**	**Sub pan 60º (300 s)** **Sub pan 40º (100 s)** **Oxidation beam 20sccm (50 s)**

STEP 3.1. Insulating Layer Deposition- 500Å of Al_2O_3

Responsible: Filipe Cardoso

Machine: UHV2	Power /W	Gas Flow /sccm	P /Torr
Setpoint	200	45	3,3E-3
Read	200	45	
Time	27 min		

STEP 3.2. Oxide Lift-Off Date: 06/02/07

Hot u-strip + ultrasonic
Rinse with IPA + DI water **Time:** 24h

Optical inspection:

Comments: The sample #3 is the one less define in shape and size

Details: #3 Areas bigger than expect: the junctions are 1μm side bigger
Round corners (worst one)
Not very well define
Some of the big areas didn't open

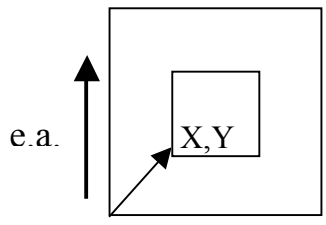
#8 Below 1μm side the junctions are about 20% smaller, some are not there
Above 2μm side the junctions are well define

#9 Junctions sizes are very close to the expected (betters)
(1x1→1.1x1.2 or 1x5→1.1x5.2)
The smaller and thinners junctions, seem little bit unfocus

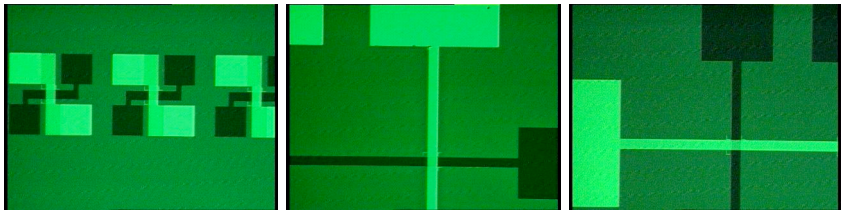
#11 Areas bigger than expect: the junctions are 1μm side bigger
(1x1→1.5x1.5 or 1x2→1.5x2.5 and then 1x5→2x6…)
Junctions are well define, corners ± round (better than #3)

STEP 4.1 3rt Exposure – Top electrode metallization

Coating PR: coat 1.5 μm PR (Recipe 6/2) **Developer :** Recipe 6/2

Machine: DWL	**Mask Name: aaEtchL3**
- Cross Center: X=168 , Y=54 - Vapor Prime 5 min. (Recipe - 0) - Energy: 35 % - Focus: 35 - Photo Resist: 1.5 μm - Recipe: 6/2 - WAFER6PA.map/dwl/wa/fa	

Optical inspection:



Comments: They seem OK. The left squares matrix only appears after the liftoff.

STEP 4.2	**Contact Leads Deposition (AlSiCu)**	Date: 07 / 02 /07

Machine: **Nordiko 7000**

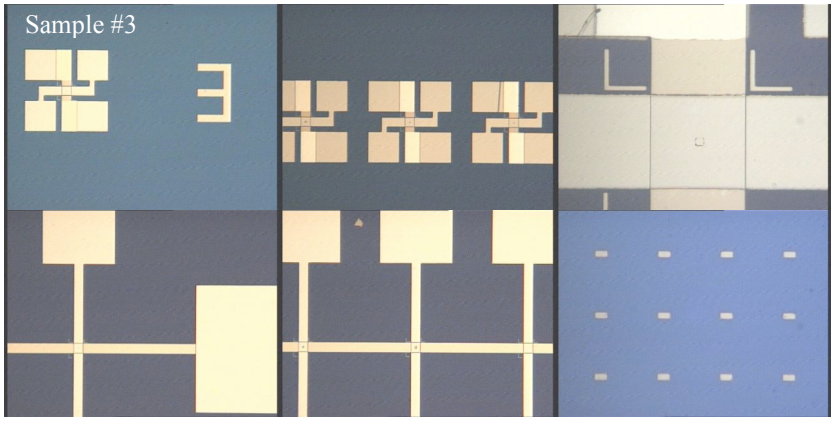
Sequence 48

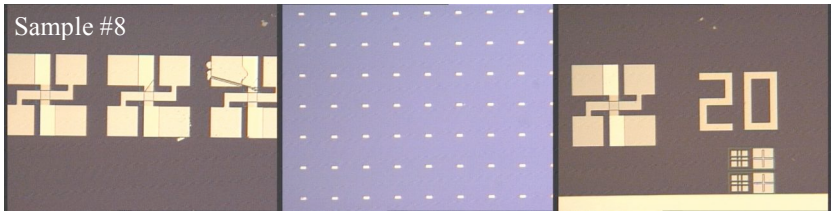
Module 2:	F9, 60” F69, 30W, ROB99, 50.2sccm, 3mT
Module 4:	F1, 120”, 3000 Â 2.0kW, 410V, 4.9A, 50.4sccm, 3.0mT
Module 3:	F19, 27”/ 54”*, 150Â/ 300Â* 0.5 kW, 431V, 1.2A, 50.65sccm, 3.1mT, 10sccm

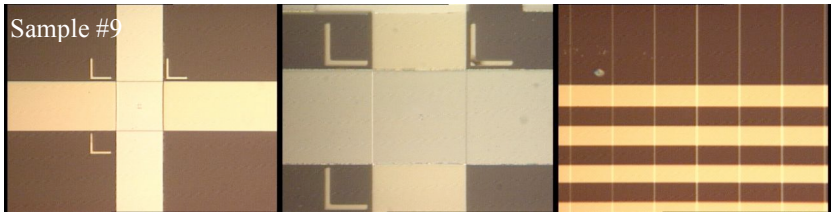
* First time was tried to deposit 300 Å of $TiWN_2$, however there was a pressure problem, and system had to be reboot, so on second time was deposited 150Å of $TiWN_2$, instead 300Â.

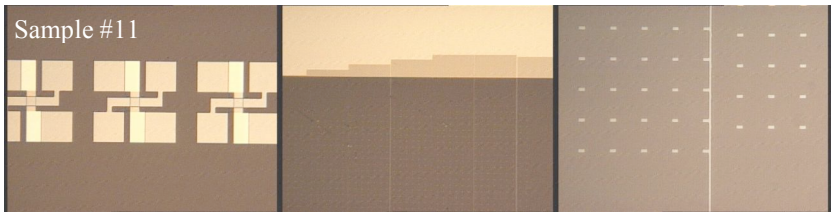
STEP 4.3	**AlSiCu Liftoff**

Hot µ-strip + ultrasonic
Rinse with IPA + DI water



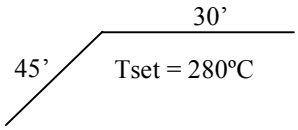




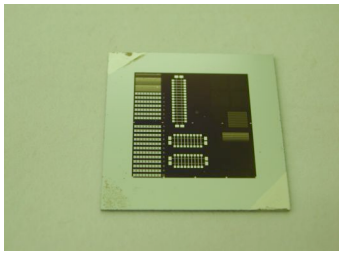


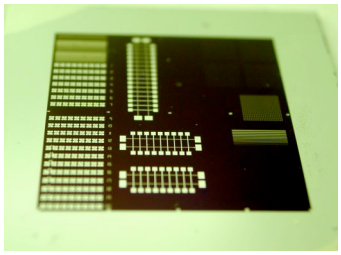
STEP 5. **Annealing**	Date 28 /02 / 07

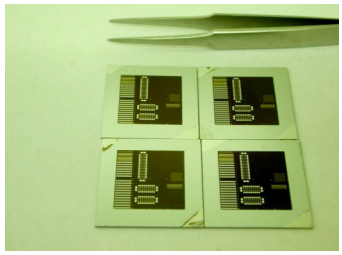
Machine: big annealing setup **Annealing conditions**: 30 min@280ºC



Final outlook







Run Sheet: # 4 **Responsible:** André Augusto

Structure			**Substrate:** Glass
Junction	**Bottom Electrode**	**Barrier**	**Top Electrode**
N2 TJ68 4 samples (A,B,C,D)	Ta50/Ru180/Ta30/MnPt200/CoFe20/Ru9/ CoFeB30	MgO 8 A,	CoFeB 30/Ru50/Ta50/ $TiWN_2$ 150A

STEP 0 **Junction Deposition (A) and passivation Layer(B)**

Responsible: Piotr/Zhao

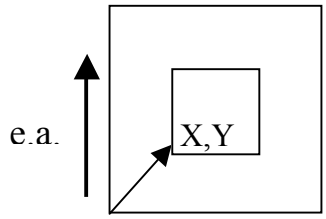
(A) Machine: **Nordiko 2000**

(B) Machine: **Nordiko 7000**

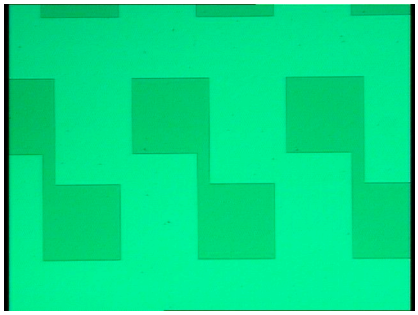
Seq.17 –Mod 2 funct. 9 (contetch) (**60")** P=70W/40W, p=3mTorr, 50 sccm Ar
Mod.3 funct.19 (svpassiv) (**150A $TiWN_2$**, 27") 0.5 kW, 3mTorr, 50 sccm Ar + 10sccm N_2

STEP 1.1. **1st Exposure – Bottom electrode definition** Date: 24 /04 / 07

Coating PR: 1.5 μm PR (Recipe 6/2) **Developer :** Recipe 6/2

Machine: DWL	**Mask Name: amsmtj1**
- Cross Center: X=168 , Y=54 - Vapor Prime 5 min. (Recipe - 0) - Energy: 47.5 % - Focus: 25 - Photo Resist: 1.5 μm - Development time: 60" - map:AMSION	

Optical Inspection:



Comments:

The structures from A, B, C and D samples are well defined. They are quite clean, but there are some particles that can have influence on photoresist uniformity.

STEP 1.2.	**1st Ion Milling –** Total Structure Etch	Date: 25/04 /2007

Machine: N3600

Standard Etching Recipe (Junction Etch) : **Etch pan 60°**

Total thickness to etch: **867 A**

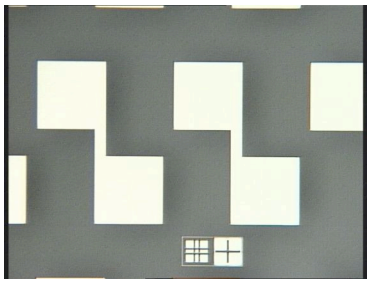
Samples	Conditions
A,B, C	3x(300" @ 60deg + 120 " cool down)
D	4x(270" @ 60deg + 120" cool down)

STEP 1.3	**Resist Strip**	Date: 26 / 04/ 2007

Hot Micro-Strip + Ultrasonic
Rinse with IPA + DI water

Total Time in Hot Micro-Strip: some hours Ultrasounds Time : few

Optical Inspection:

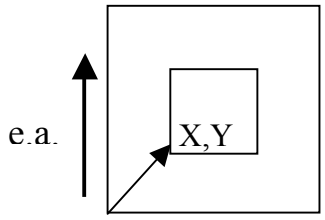


Comments:

All samples look OK.

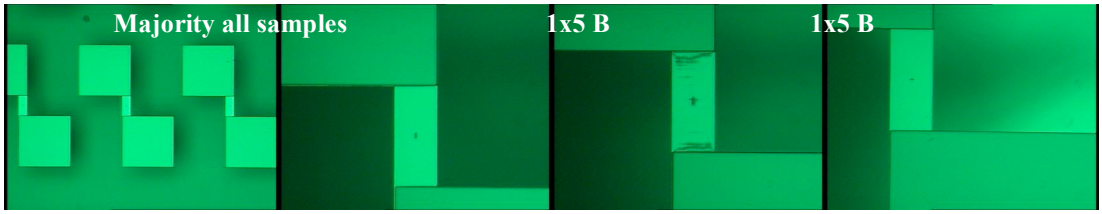
STEP 2.1	**2nd Exposure – Junction area definition**	Date: 07 /05 /2007

Coating PR: 1.5 μm PR (Recipe 6/2) **Developer :** Recipe 6/2

Machine: DWL	**Mask Name: aaAMSTJL2**
- Cross Center: X=168 , Y=54 - Vapor Prime 5 min. (Recipe - 0) - Energy: 45 % - Focus: 25 - Photo Resist: 1.5 μm - map:AMSION	

Sample	Development time
A, B, C	35"
D	30"+5"

Optical Inspection:



Comments: All samples can have devices just a little bit bigger that the ones that were designed: more critical for 1µm side devices.

Sample B The developing was not so good like others, but there is no critical issue. 1x3 um^2 size devices can be 1x2 μm^2.

STEP 2.2 **2nd Ion Milling –** Junction area definition

Machine: N3600

Read Pressure (Torr) : 10^{-4}

Wafer Recipe **: #10: etch junction top electrode + O2**

Process Step **: Load Wafer/etch pan 60 deg (270")/etch pan 30 deg (60")/oxidation beam (Xs)/ end function**

Total thickness with overetch (rate 1A/s): 330 A

Calibration Sample	Structure
Piotr	$TiWN_2$150/CoFeB 30/Ru50/Ta50/MgO/CoFeB30

Sample	Oxidation Beam X (s)
A	No O2
B	100
C	20
D	300

Assist Gun / Neutralizer	Power (W)	V+ (V)	I+ (mA)	V- (V)	I- (mA)	Gas Flow (sccm)	I neutr (mA)	V neutr (V)	Sub Rotn (rpm)	Sub Pan (deg)
Read Values Etch steps	158	724	105	345	2.3	10.2 He 0.1 O2	116,5	330V	30	60/30
Read Values O2 step	135	101	45.2	345	1.2	2.2 He 20.1 O2	-	-	30	0

Comments: The read values above written are similar for all samples.
-There was a problem with sample D, the arm stuck and the sample didn't come out. Maybe the etching wasn't done (the wafer didn't enter in the main chamber)!!!!!!!!!!!!!

STEP 3.1.	**Insulating Layer Deposition- 500Å of Al_2O_3**	Date: 11 /05 / 2007

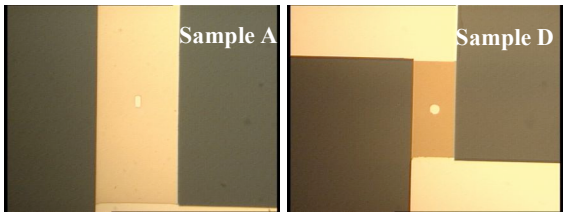
Machine: **UHV2**

Deposition Time	AlO_2 thickness	Gas Flow	Base Pressure	Power Source
27 min	500 A	45 sccm	3 mT	200 W

STEP 3.2.	**Oxide Lift-Off**	Date: 11 / 05 / 2007

Hot u-strip + ultrasonic
Rinse with IPA + DI water

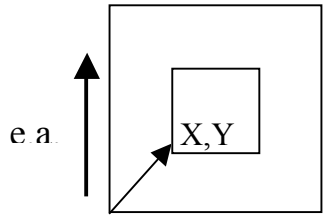
Optical inspection:



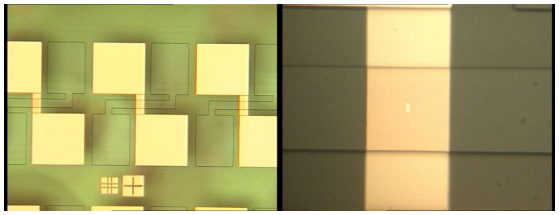
Comments: The samples A, B and C seem all ok. But the sample D faced several problems in the liftoff.

STEP 4.1	**3rt Exposure – Top electrode metallization**

Coating PR: 1.5 μm PR (Recipe 6/2) **Developer :** Recipe 6/2

Machine: DWL	**Mask Name: amsmtj3**
- Cross Center: X=168 , Y=54 - Vapor Prime 5 min. (Recipe - 0) - Energy: 47.5 % - Focus: 25 - Photo Resist: 1.5 μm - Development time: 60” - map:AMSION	

Optical inspection:



Comments: All samples are OK.

STEP 4.2. **Contact Leads Deposition (AlSiCu)**

Machine: **Nordiko 7000**

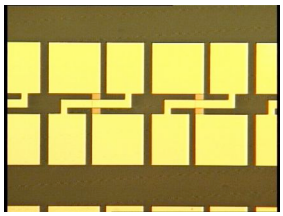
Sequence 48

Module 2:	F9, 60” F69, 30W, ROB99, 50.2sccm, 3mT
Module 4:	F1, 120”, 3000 Å, AlSiCu 2.0kW, 410V, 4.9A, 50.4sccm, 3.0mT
Module 3:	F19, 27”, 150Â, $TiWN_2$ 0.5 kW, 431V, 1.2A, 50.65sccm, 3.1mT, 10scc

STEP 4.3 **AlSiCu Lift-Off**

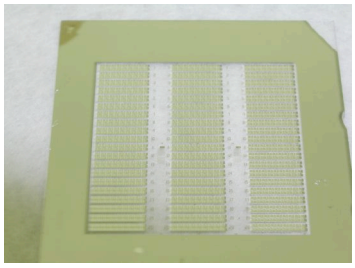
Hot µ-strip + ultrasonic
Rinse with IPA + DI water

Optical inspection:



Comments: All samples OK.

Final outlook:



STEP 4 **Annealing** Date 07 /06 / 07

First the samples were annealed in the bigger setup at 280ºC for 1h and the TMR signal was lower than expected. Then the samples were sliced and annealed in smaller setup at 320ºC for 1h (field 4kOe), and the TMR improves a lot.