DTU Danchip

Supporting investigations of surface defects in MR-I 7000E imprint resist on ON209 virgin wafers

To NILT, DTU Nanotech, DTU Danchip

Re Defects in MR-i7030 imprint photoresist coatings on wafers

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NCR 0001 - supporting notes
AJOE

Root cause conclusion

The defects seen in MRi-7030E resist on wafers were due to the condition of the photoresist itself. The problem could be solved by degassing and filtering the resist.
The investigations leading to this conclusion are described in the remainder of this note.

Saeed Abadei from DTU Nanotech assisted with experiments as well as parts of this note, and his assistance is acknowledged.

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Resist information

Name: MR-i7030E
Type: Thermal plastic resist
Solvent: PGMEA
Recommended pretreatment: Bake out of wafers for 120 sec at 140ºC

From the Microresist website it is stated that: "**mr I 7000E, mr I 8000E:** No adhesion promoter necessary on silicon, SiO_2, aluminium, or chromium surfaces" (http://www.microresist.de/download_en/faq/faq_nil_materialis_en.htm)

Following enquiry, the supplier of the resist (Microresist) has told that they have changed the method by which they filter the resist. This change in procedure took place at a non-specific date in the first half of 2009, and apparently happened without informing the customers.

Defect description

There are basically two types of defects:

1. Round objects in the resist with what appears as a ‚volcano' shape around it (Figure 1)
2. Elongated areas of what appears to be thicker resist (Figure 2 and Figure 3)

The lines extending from the structure are consistent with the spin pattern.

Figure 1. Structure on a new ON209 wafer straight from the box.

Figure 2. Structure on a new ON209 wafers straight from the box.

Figure 3. Structure on a new ON209 wafers straight from the box.

The experiment leading to this investigation consisted of spin-coating numerous wafers as summarized in the nearby table (Table 1).

Table 1: Defect count as well as maximum spin-speed for the experiment leading to the extended evaluation.

Wafer number	Spin speed	No of defects	Notes
1	No spin		
2	No spin		
3	No spin		
4	3500	5	Last sample
5	3500	3	
6	3500		Dead
7	3500	5	
8	3500	7	Resist refilling
9	4500	5	
10	4500	5	
11	4500	5	
12	4500	3	
13	4500	4	
14	5000	4	
15	5000	8	
16	5000	3	
17	5000	5	
18	5000	10	Resist refilling
19	4000	2	
20	4000	6	
21	4000	3	
22	4000	2	
23	4000	5	
24	4000	4	
25	4000	6	First sample

Ishikawa diagram

The most obvious and quite a few unlikely explanations to the defects have been collected in the fishbone diagram (Figure 4).

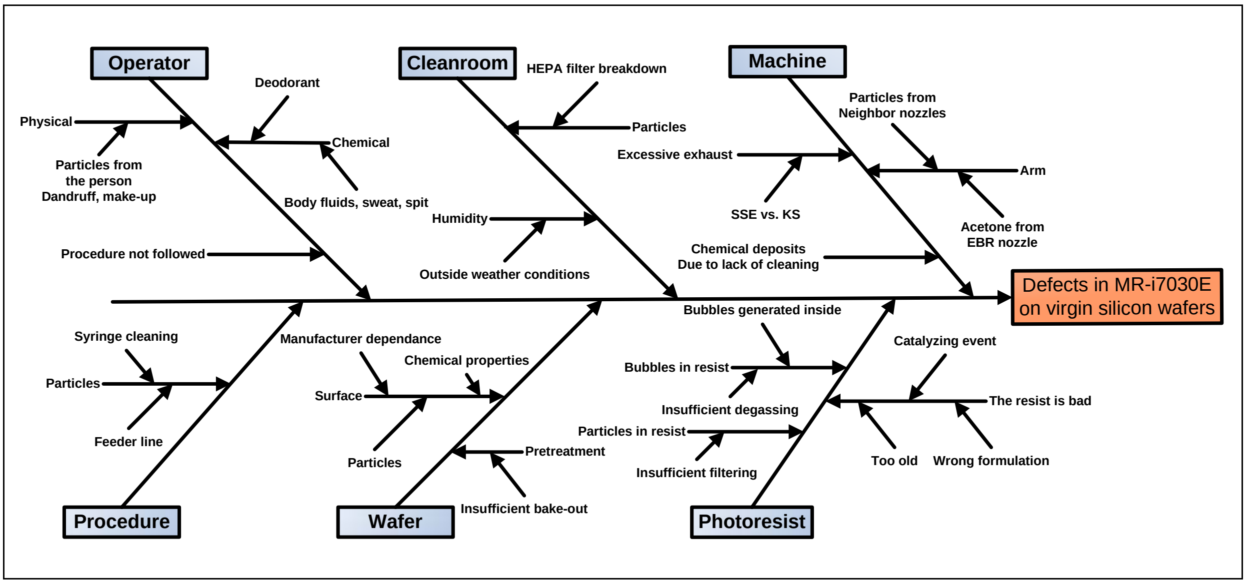

Figure 4. Ishikawa (fishbone) diagram for the defects on wafers.

Investigations

Plotting the run order and spin speed as a function of defect count (Figure 5 and Figure 6) does not reveal anything conclusive. There is a hint of higher defect count with higher speeds, but the data does not give enough statistical basis to draw solid conclusions.

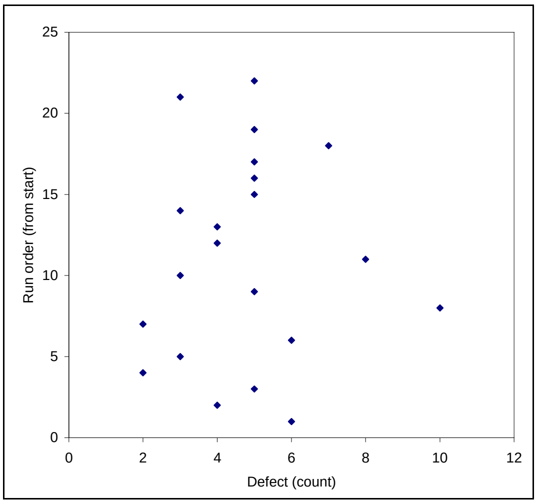

Figure 5. Plot of run order vs. defect count.

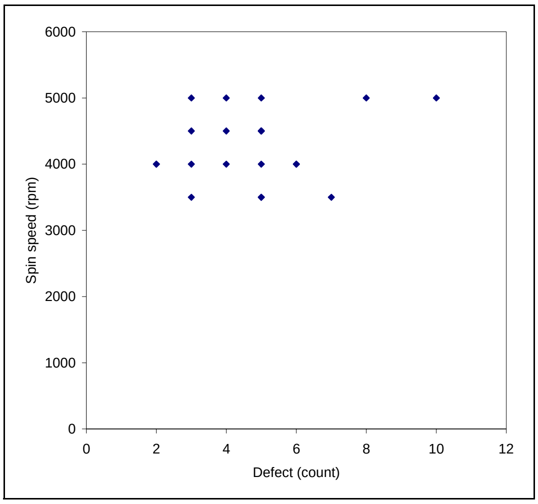

Figure 6. Plot of spin speed vs. defect count

Key investigations of cause categories

In order to get to the root cause of the defects a number of investigations were carried out. The table below summarizes the key investigation for each category of causes (Table 2).

Table 2: Summary of category investigations for the root cause analysis. The photoresist itself is found to be the root cause.

Category	Key investigation	Root cause?
Operator	Two different operators made coatings. Neither could make a blemish-free coating	No
Cleanroom	Coatings done both under in-control conditions and too high humidity conditions had defects	No
Machine	The MR-i7030e was applied on both the SSE Maximus spinner, SSE Manual spinner and the Karl Süss RC8 spinner. Defects could be seen on coatings from all machines	No
Procedure	The same procedure using ZEP resists produced defect-free coatings	No
Wafer	Different wafer types as well as lift-off resist (LOR) coated wafers all showed similar defects	No
Photoresist	Degassing and filtering the resist lead to defect-free coatings	Yes

Cause category investigations

A number of investigations of the various causes were carried out in order to investigate the root cause (Table 3). The various experiments are discussed in the later sections.

Table 3: The various investigations carried out in order to analyze the individual possible causes of the defects.

Category	Cause	Investigation	Outcome
Operator	Procedure not followed	Two persons present for deposition	When stringent procedure was observed there was still defects (Experiment 1)
	Chemical pollution from operator	Two different operators perform the deposition	No significant difference between results from the different operators (Experiment 1)
	Physical pollution from operator	Two different operators perform the deposition	No significant difference between results from the different operators (Experiment 1)
Cleanroom	Particles	Test the deposition on the manual spinner located in another airflow section	No significant difference between Maximus and Manual spinner (Experiment 2)
	Humidity	Analyse test results obtained on a day when the humidty level was out of control in the cleanroom with the manual spinner	No significant difference between Maximus and Manual spinner regardless of the humidity level. (Experiment 3)

Category	Cause	Investigation	Outcome
Machine	Chemical deposits due to lack of cleaning	Test the deposition on the manual spinner	No significant difference between Maximus and Manual spinner (Experiment 2)
	Acetone from EBR nozzle	Disconnect acetone or try manual spinner	There is no acetone connected to the nozzle
	Particles from neighbor nozzles	Test the deposition by direct deposition of resist	No significant difference between using the deposition arm on the Maximus and direct deposition (Experiment 4)
	Excesssive exhaust	Run same deposition on Karl Süss RC8	No significant difference between Maximus and Karl Süss RC8 (Experiment 5)
Procedure	Particles	Examine if the same procedure works in any situation	The procedure works with ZEP520A photoresist. It is possible to create defect-free coatings (Experiment 6)
Wafer	Surface	Coat p-type wafers instead	Still coating defects (Experiment 7)
	Surface	Coat on top of a coating of lift-off resist	Still coating defects, although not quite as severe (Experiment 8)
	Lack of bakeout	Bakeout wafers at 250ºC for 10 min, 30 min, 24 hours	No significant difference seen on wafers with different bakeout durations (Experiment 7)
Photoresist	Too old	Try a new bottle of MR-i7030	No significant difference between the new bottle and the old one (Experiment 9)
	Wrongfully formulated resist	Ask the supplier for a batch check	Microresist reports that they are capable of making 'perfect' coatings based on a sample from either batch of bottles (Experiment 10)
	Catalyzing event	Not investigated	
	Dissolved gas in the resist	Degas the resist	Degassing the resist at 0.2 – 0.3 Bar (absolute) for 30 min significantly reduced the defect count (Experiment 11)
	Particles in the resist	Filter the degased resist	Filtering the resist through a 0.1 µm PTFE filter reduced the defect count (Experiment 12)

Combining degassing and filtering led to perfect coatings on both the Manual and the Maximus spinner.

Experiment 1 – Two operators

(Investigations performed by Saeed and Elena)

Two different operators performed the spinning on Maximus spinner with the same parameters on the identical substrates with the same resist. Each operator spun 5 wafers, which are investigated with the optical microscope. The wafers displayed similar number of defects.

Experiment 2 - Differences between spinners

(Investigations performed by Saeed and Elena)

Wafers were spun on the Maximus (located in the new yellow room) and the manual spinner (located in the old yellow room). The wafers displayed similar defects regardless of which spinner was used. The Karl Süss RC8 has a different machine layout and different exhaust and could be further investigated.

Experiment 3 – Humidity differences

(Investigations performed by Elena)

The spinning performed in two different cleanrooms: cleanroom 3 and cleanroom 14, with the different humidity conditions. The test was carrying out in order to investigate the resist adhesion which can cause some of the defects (e. g. small air bubbles trapped between the substrate's surface and resist). The spinning with the same parameters was done in two different CR in two different spinners. The same defects were observed on the wafers after the spinning.

Experiment 4 – Direct deposition on Maximus SSE

(Investigations performed by Saeed)

To exclude the possibility of the external particles contaminations from the equipment itself (the movable media arm, the movable coater cover) the manual resist validation was performed in Maximus. The standard recipe was changed in order to do prevent all unnecessarily movements during spinning: the manual validation was chosen in stead of automatic validation with the media arm; the movement of the coater cover was tuned off. The tests showed no improvements regarding to number of defects on the wafers.

Experiment 5 – Different local exhausts RC8 and Maximus SSE

(Investigations performed by Elena)

Since all previously test were run on the same type of spinner (Maximus or OptiCoat Spinners are both from ATMSSE http://www.sse-semi.com/docs/index.aspx?id=32381&domid=1068&sp=E&m1=31886&m2=32381) another spinner was chosen for test- RC8 KS Spinner. The main different between those two types is the local exhausts on the chuck during spinning step in RC8 can not be turned off as in Maximus. Therefore we can expect that the different conditions during the spinning can be affecting the spinning results, especially number of particles on the surface. The tests on the RC8 spinner were performed with the same parameters as the previously tests, but did not show the significant difference between the equipments types.

Experiment 6 - Defects on wafers processed 2 weeks earlier

(Wafers processed and inspected by Elena)

A set of wafers covered with ZEP520A resist were inspected for defects (Table 4). These wafers had been processed on the Maximus spinner two weeks prior to the problematic runs with MRi-7030E.

Table 4: Visual inspection of 24 test wafers with ZEP520A resist

Wafer number	Number of defects	Comments
1	0	
2	0	
3	0	
4	1	
5	0	syringe refill
6	0	
7	0	
8	1	
9	0	
10	2	syringe refill
11	2	
12	1	
13	1	
14	0	
15	1	
16	8	last wafer before refill
17	0	syringe refill
18	0	
19	0	
20	0	
21	0	
22		incomplete coverage due to not enough resist in the syringe
23	0	syringe refill
24	0	

The next serie of the experiment will describe the test with the variable pretreatment step of the test wafers. We desided to implement those experiments because some of defects could be addressed to bad adheasion between the surface and the resist.

Experiment 7 - Difference in surface treatment

(Investigations performed by Saeed and Elena)

In this experiment 3 different surfaces pretreatment were investigated and compared:

1. BHF dip for 30sec.: removing the native oxide from the virgin wafers.
2. HMDS treatment in HMDS oven : surface adhesion promoter
3. 250deg. C bakeout in 250deg oven: demoisterizing of wafers

The results after pre-treatment were compared with the control group of the non-pre-treated wafers. No different appeared between the groups: the same number and the same kind of defect are detected after the spinning.

Some examples of the defects are illustrated (Figure 7 and Figure 8). All defects looked like coming from small particles or bubbles.

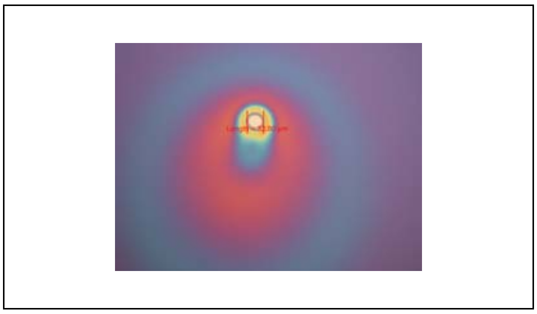

Figure 7. Structure on a ON209 wafer subjected to BHF dip prior to spinning.

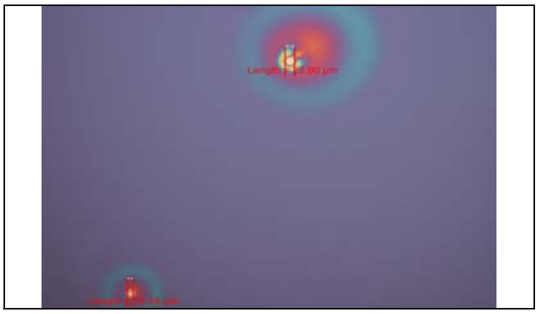

Figure 8. Structures on a ON209 wafer subjected to BHF dip prior to spinning.

Experiment 8 – Coating on lift-off resist (LOR)

(Investigations performed by Saeed)
The wafers displayed similar defects, no improvement.

Experiment 9– Different bottles of MRi-7030

(Investigations performed by Saeed and Elena)
To investigate and compare the resist quality 2 different batch of the same resist MRi-7030 were used for spinning: the old bottle, which was opened and used for all previously experiments, and a new bottle, which was ordered for the new tests. All spinning were done in Maximus with the same parameters and conditions. No improvements were seen after the spinning. It was decided to consult the MicroResist for processing advice and to get more information about MRi-7030 resist, practically for the batches we used in out tests.

Experiment 10 – Microresist’s batch control inquiry

(Investigations performed by Saeed)
MicroResist did a control spinning with the same resist batch on the tests wafers. 10 test wafers were send to MicroResist. Wafers were pre-treated by bakeout at 250ºC before coating.
The results of the tests are shown (Figure 9 and Figure 10). Generally the coatings looked better but did have some defects at the edge of wafers similar to previously seen.

Figure 9. Structure on a pre-treated wafer, spin-coated By Microresist. Defects at the edge of sample.

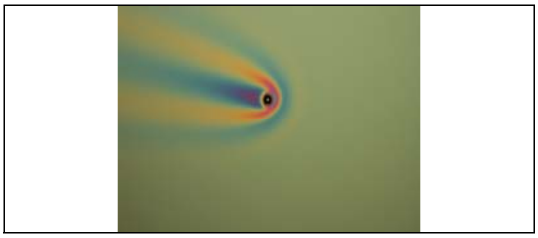

Figure 10. Structure on a pre-treated wafer, spin-coated By Microresist. Defects at the edge of sample.

Experiment 11 – Degassing of the MRi-7030

(Investigations performed by Elena)

To avoid the micro bubbles, which were observed on the surface after the spinning, we decided to degas the resist prior the spinning. Degassing was performed in a dessicator chamber outside the CR at 0,2- 0,3 bar for 30 min. No visual changes of the resist quality or significant change of the resist amount were observed under the degassing. The degassed resist was spun in the manual spinner. The result showed fewer defects on the surface, where especially the defects caused the micro bobbles in the resist were gone. To continue with the progress we made with resist degassing we decided to filter the degassed resist prior the spinning.

Experiment 12 – Degassing and filtering the MRi-7030 resist

(Investigations performed by Elena and Saeed)

0.1 um PTFE filters were used for filtering of the degassed resist. The filter was mounted on the syringe and the resist was squeezed through the filter into a new clean bottle. The spinning test was performed first in the manual spinner. No defects were observed after the spinning. The experiment was repeated in the Maximus: no defects were observed there either.