EE412 2011 Fall Quarter Final Report

Characterization of Fiji ALD Film Quality and Conformality in High Aspect Ratio/Deep Etched Structures

Insun Park, Jooyong Sim, Young Ik Sohn

Mentor: J Provine

Introduction

Complex surface topographies consisting of high aspect ratios, sharp asperities, and nano-roughness significantly challenge the ability to conformally coat complex nanostructures using traditional physical vapor deposition (PVD) and chemical vapor deposition (CVD) techniques. However, atomic layer deposition (ALD) offers high-quality films with atomic level thickness control and unprecedented uniformity and conformality in the most demanding 3D nanostructures. Now the SNF possesses new Fiji systems which are capable of depositing various films with thermal and plasma ALD. Thermal ALD has better coverage over structures while plasma ALD has less dependence on substrate type. This work addresses how the Fiji ALD covers on high aspect ratio/deep etched structures and compares the performance of plasma/thermal methods and savannah/fiji machines.

Materials and Methods

We fabricated reentrant structures (i.e., deep trench with a spherical undercut at bottom) with high aspect ratio on <100> L prime Si wafers. The fabrication process is shown in Fig. 1. First, we thermally oxidized the wafers to thickness of 1.3um. Then we photolithographically patterned the holes with the diameter of 1um, 3um and 5um using ASML i-line stepper, and transferred the pattern through the SiO_2 layer using AMT etcher (Fig.1(a)). Next, we DRIEd the wafers on the order of 50um using the STS etcher 2 and performed 1um-thick sidewall oxidation to protect the etched trenches from the subsequent isotropic Si etch. The bottom oxides were removed by the AMT etcher (Fig.1(b)), and the sphere-like undercut structures were made using the Si isotropic etch recipe in the STS etch 2. We gave variation to samples by stripping remaining SiO_2 layer only for the half of the samples in 6:1 BOE solution. The sample variation is summarized in the Table 1. As a final step, various cycles of thermal/plasma ALD films such as Al_2O_3, TiN, and Pt were deposited on the structures (Fig.1(c)).

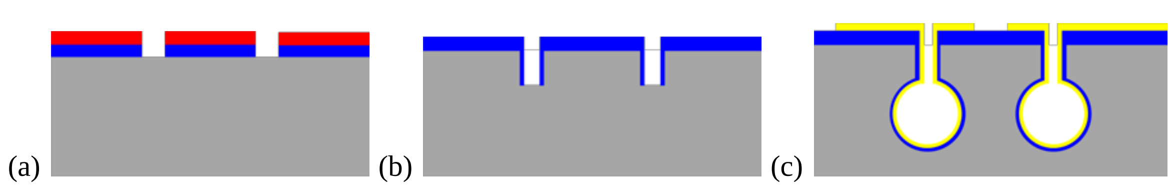


Figure 1: Test structure fabrication (a) thermal oxidation and pattern transer to the SiO_2 hard mask using photolithography (b) 1st DRIE on the order of 50um followed by sidewall thermal oxidation and bottom oxide removal (c) Isotropic etch to create compliant termination and ALD coating (SiO2 was removed from half of the samples before the ALD step)

Hole Diameter (D)	1 um	3 um	5 um
Depth(um)	32um	44um	50um
Aspect Ratio	1:32	1:14	1:10

Table 1: Sample Variation

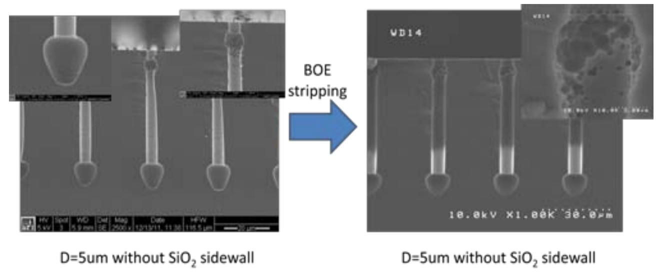


Figure 2: D=5um samples before ALD coating

The ALD films are usually too thin for us to observe its uniformity and conformality on the structures in SEM. Therefore, we used pinhole etch test where the ALD-coated structures went through XeF_2 etch for 10 cycles (30 seconds/cycle) (Fig.2). This method can indirectly check the coverage of the film on the structures because XeF_2 can leak into the Si substrate and isotropically etch them in the presence of pinholes in the film. The degree of the structural damages indicates how bad the uniformity of the film is. The crossections of the cleaved samples were inspected using SEM. The samples without the sidewall oxidation can show how well the ALD covers the whole structures while the ones with the sidewall oxidation exclusively demonstrate the undercut coverage of ALD films because the sidewall protection from XeF_2 comes from both the existing SiO_2 layer and the ALD films.

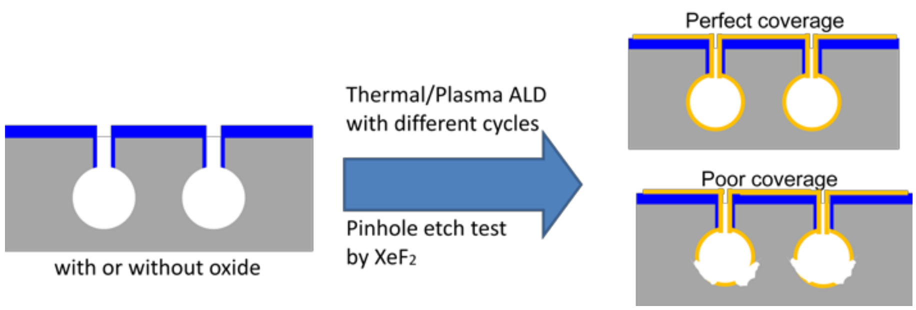


Figure 2: Pinhole etch test as a verification method for the ALD films

When we designed the photomask, we made hole arrays with each row offsetted in such a way that a straight cleaved line pass through at least one hole at its center. However, when we cleaved some small sample pieces, the cleave planes did not go straight, resulting in partial structure exposure in crossection images. In this case, we combined different parts from different crossection locations to evaluate the film coverage.

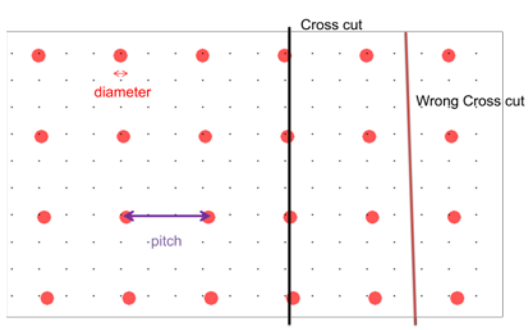


Figure 3: An example of a hole array and cleave planes

Results

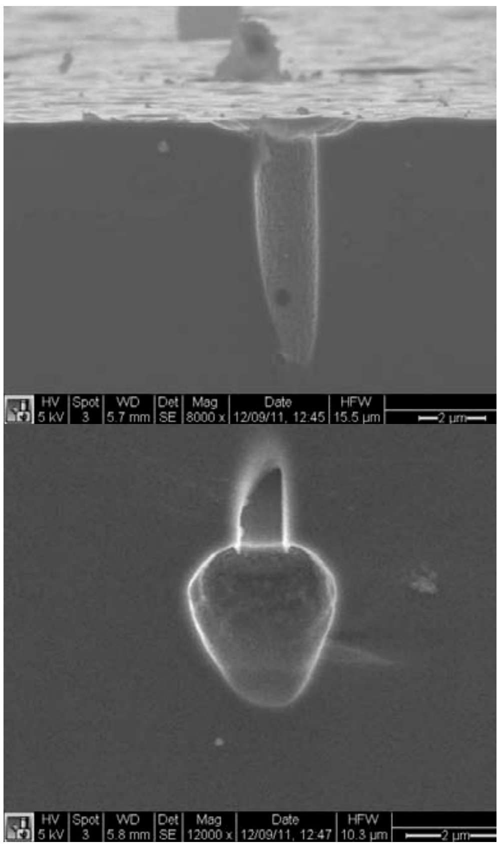
We did pinhole etching test to analyze the coverage of ALD Al_2O_3, TiN and Pt films. <u>All the test results shown below went through 10cycles XeF_2 etch (30seconds/cycle) in Xactix after ALD deposition.</u>

1 Reference Structure

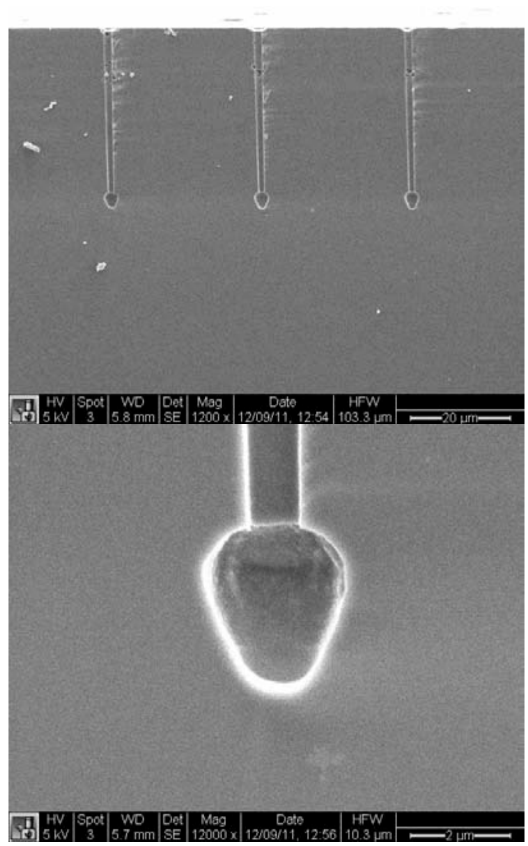
1.1 D = 1um

Test structures before XeF_2 etch

With SiO_2 on the sidewall



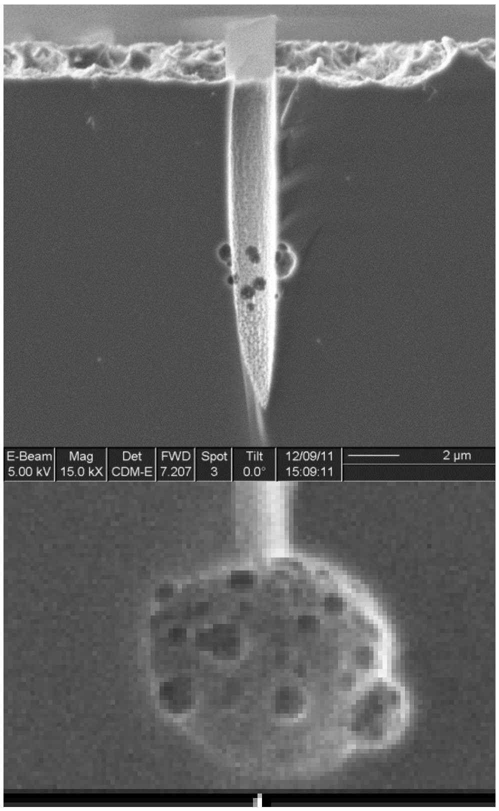
Without SiO_2



Sphere-like undercut structures were made as intended.

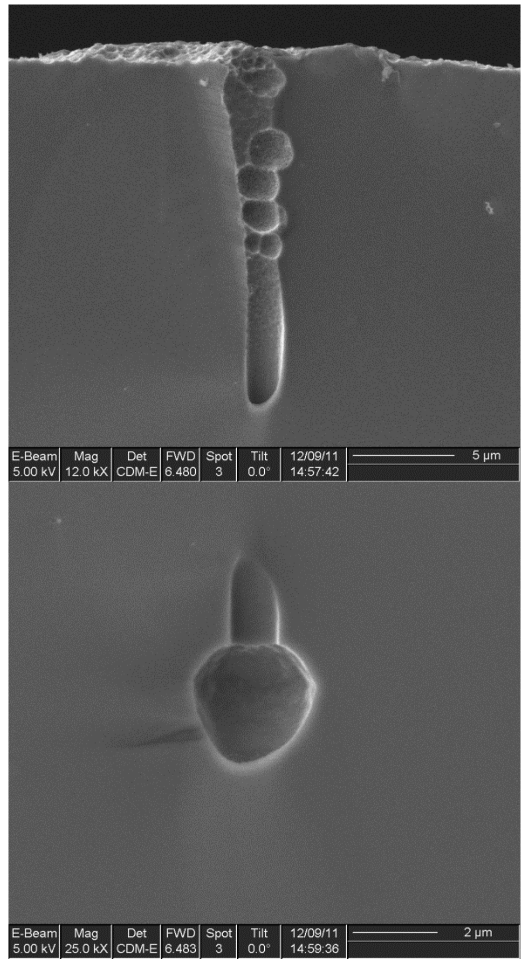
Test structures after XeF_2 etch

With SiO_2 on the sidewall



Sidewall remains the same as before XeF_2 etch, because XeF_2 barely attacks the SiO_2. Some traces of attack were caused by SF_6, not by XeF_2. On the surface of undercut structures, XeF_2 attacks and many pinholes were made.

Without SiO_2

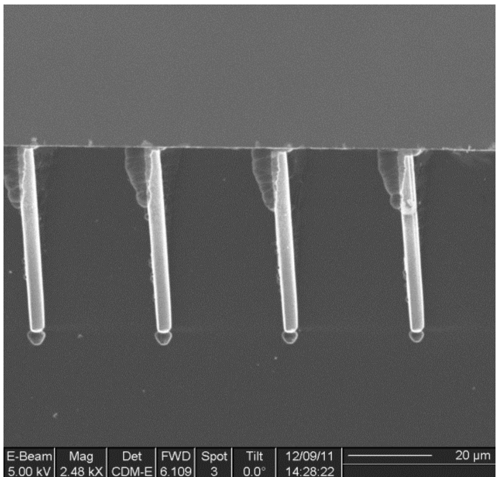


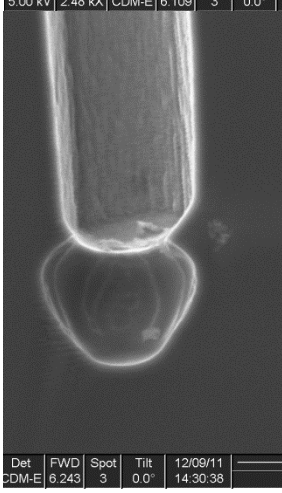
Without SiO_2 on the sidewall, XeF_2 attacks sidewall heavily. Sidewall was largely deformed by the attack. Undercut structure remains the same, because most of XeF_2 gas reacts and consumed with the sidewall. Therefore, this picture cannot be used as a valid reference.

1.2 D = 3um

Test structures before XeF_2 etch

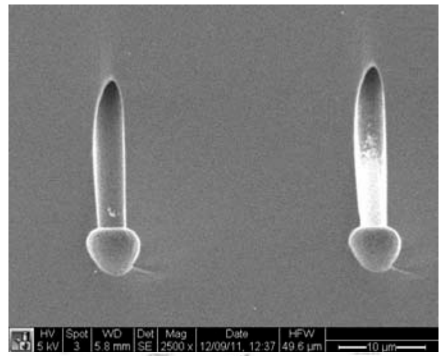
With SiO_2 on the sidewall

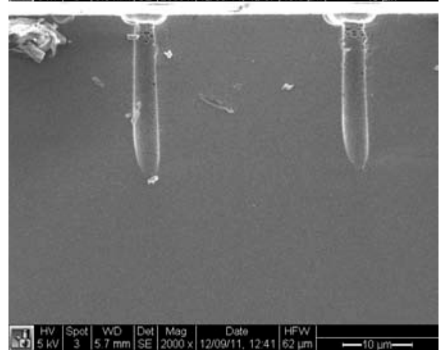




Holes were tilted from the normal direction by the malfunction of stsetch2. However, it works fine as a reference.

Without SiO_2



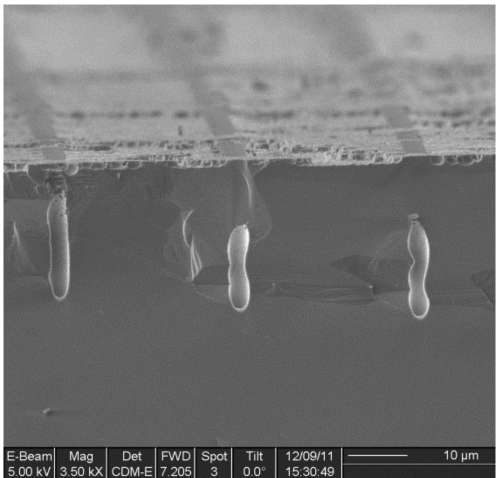


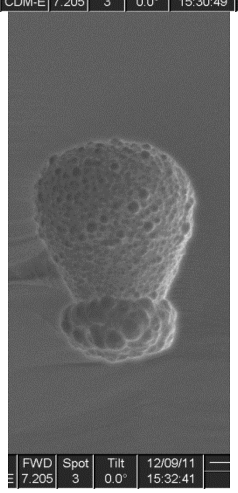
Near the bottom, thermal SiO_2 was not removed completely. BOE could not reach the bottom of the structure for some holes.

For 3um holes, Sphere-like undercut structures were made well.

Test structures after XeF_2 etch

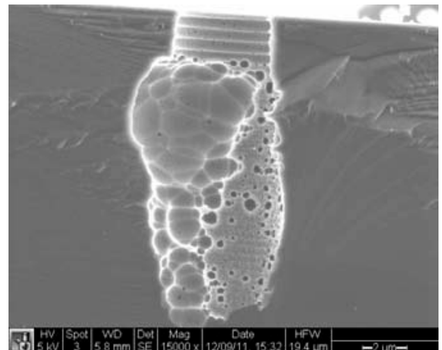
With SiO_2 on the sidewall

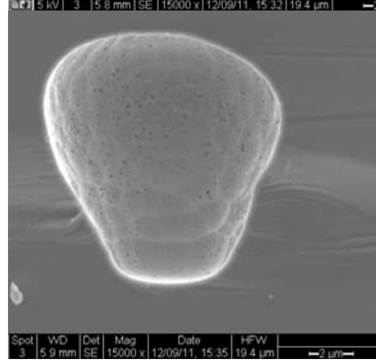




From pictures, it can be found that sidewall remains the same but undercut structures were etched much by XeF_2.Overall shape changed a lot and many pinholes were formed.

Without SiO_2



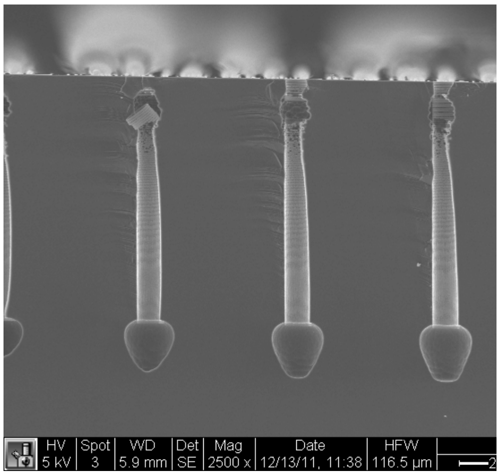


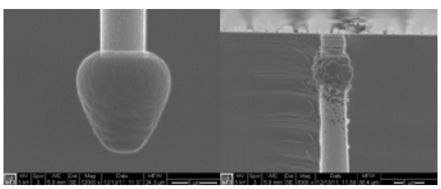
Without SiO_2 on the sidewall, XeF_2 attacks sidewall heavily. Sidewall was largely deformed by the attack. Although there are some tiny pinholes spread on the surface, the overall shape of undercut structure remains the same, because most of XeF_2 gas reacts and consumed with the sidewall. Therefore, this picture cannot be used as a valid reference.

1.3 D = 5um

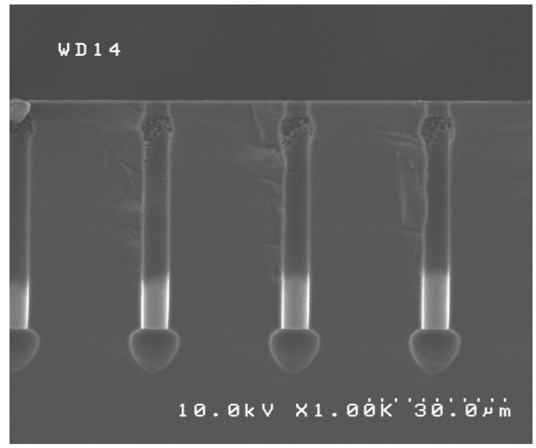
Test structures before XeF_2 etch

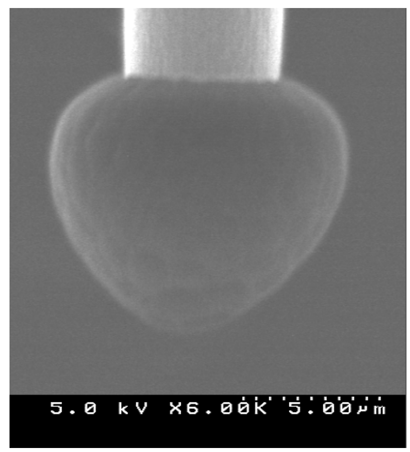
With SiO_2 on the sidewall





Without SiO_2



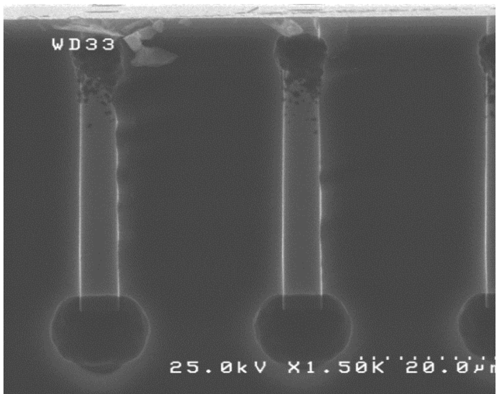


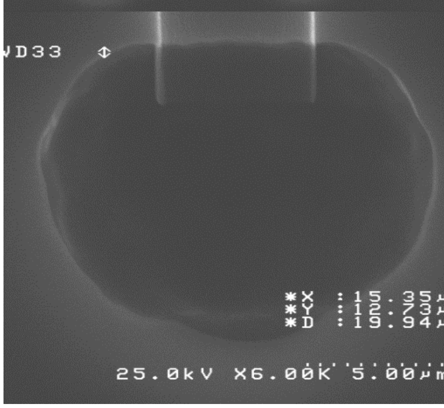
Near the bottom, thermal SiO_2 was not removed completely. BOE could not reach the bottom of the structure for some holes.

For 5um holes, Sphere-like undercut structures were made well. Near the surface, sidewalls were heavily attacked by SF_6 gas during the isotropic etch step.

Test structures after XeF_2 etch

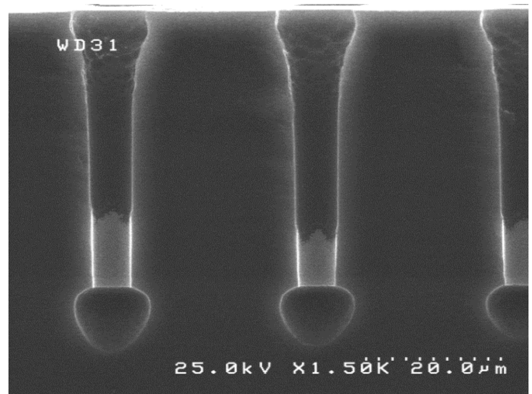
With SiO_2 on the sidewall

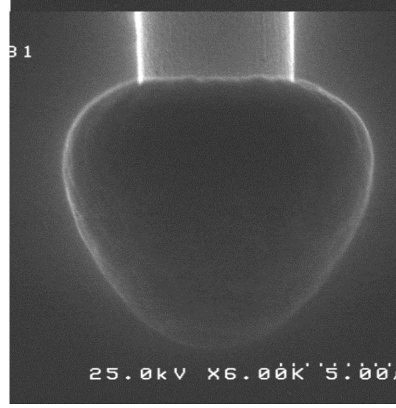




From pictures, it can be found that sidewall remains the same but undercut structures were etched much by XeF_2.Overall shape changed a lot and many pinholes were formed.

Without SiO_2





Without SiO_2 on the sidewall, XeF_2 attacks sidewall heavily. Sidewall was widened from the top and was tapered along the vertical direction. The undercut structure remains the same, because most of XeF_2 gas reacts and consumed with the sidewall. Therefore, this picture cannot be used as a valid reference.

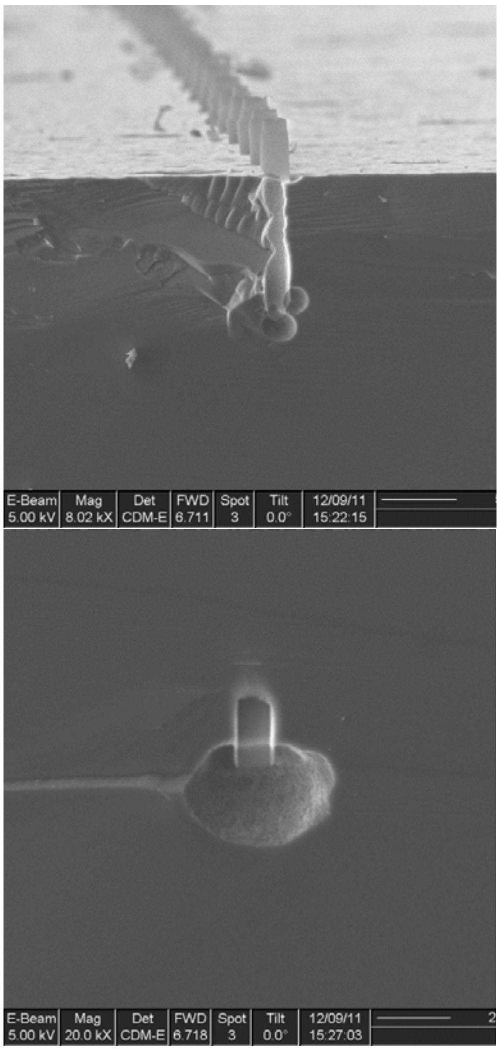
2 Al_2O_3

2.1 D = 1um

2.1.1 20cycles

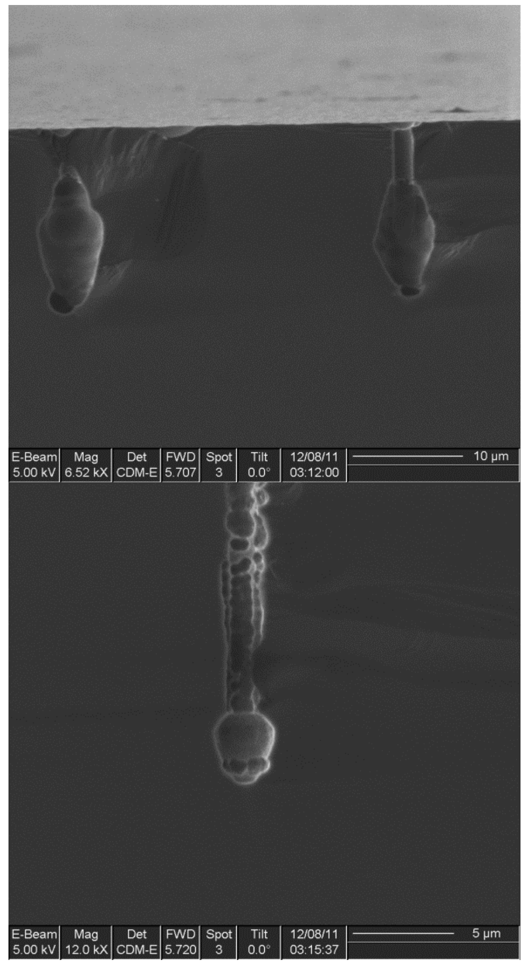
Plasma ALD

With SiO_2 on the sidewall



Since sidewall was protected by the combination of SiO_2 and Al_2O_3 film, sidewall protection by ALD cannot be judged. Undercut structure became much larger compared to the XeF_2 etched reference.

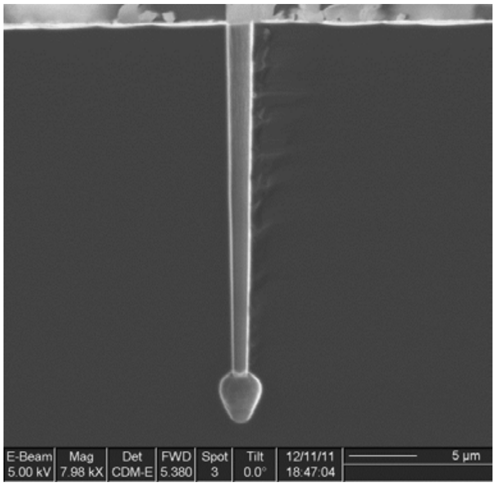
Without SiO_2



Sidewalls were attacked much by XeF2 and became much bigger. Undercut structure was attacked as well, and deformation was observed.

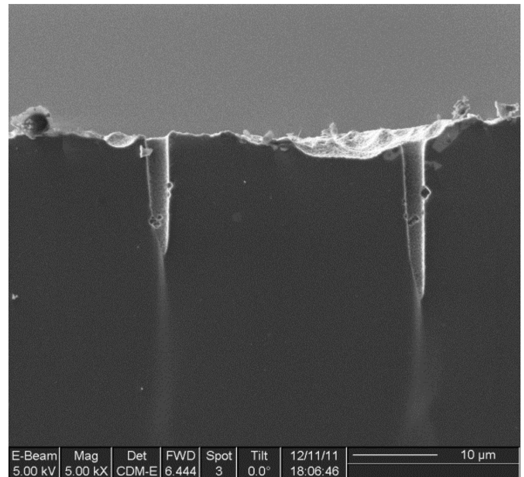
Thermal ALD

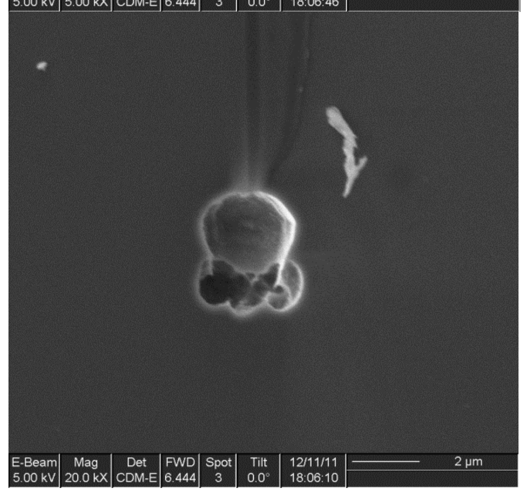
With SiO_2 on the sidewall



Undercut structure was completely protected from XeF_2 etch. Al_2O_3 film conformally coated the undercut structure nicely.

Without SiO_2



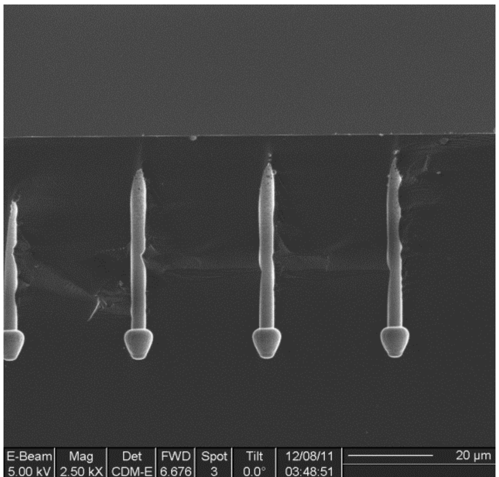


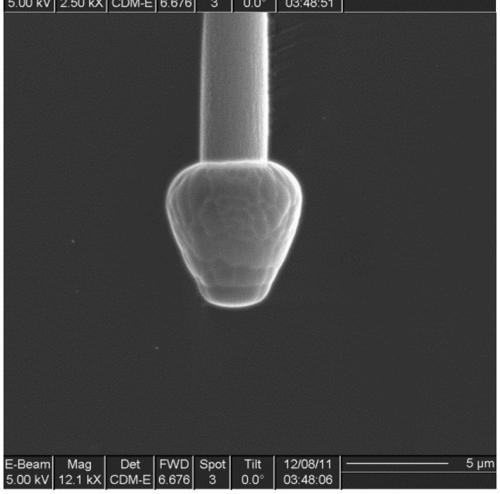
Sidewalls were protected fairly well with ALD, but the undercut structure was etched much by XeF_2.

2.1.2 100cycles

Plasma ALD

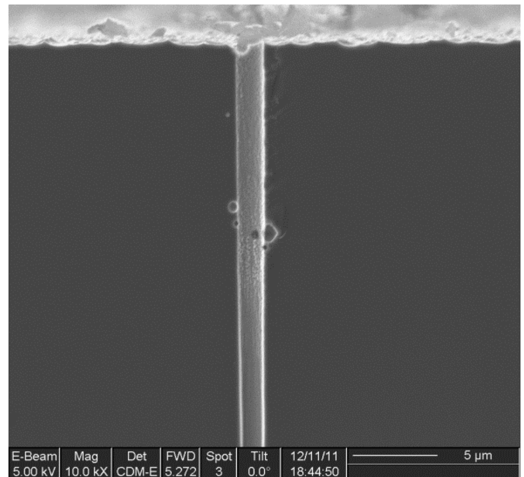
With SiO_2 on the sidewall

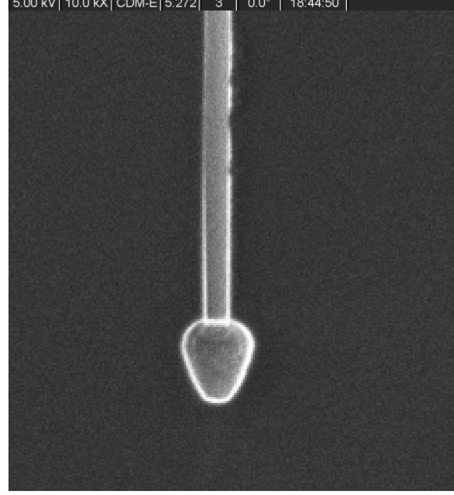




Since sidewall was protected by the combination of SiO_2 and Al_2O_3 film, sidewall protection by ALD cannot be judged. Undercut structure was protected completely by ALD coating. There is no pinhole at all for the entire structure.

Without SiO_2



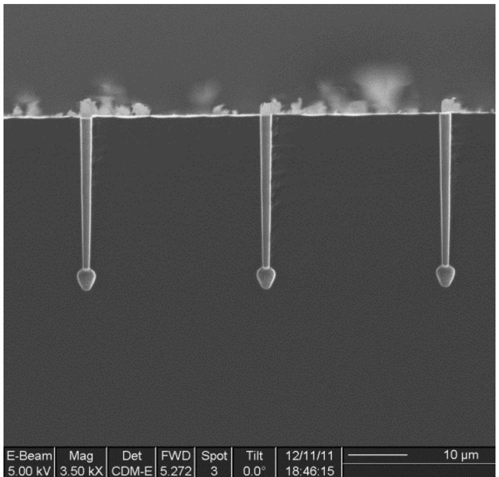


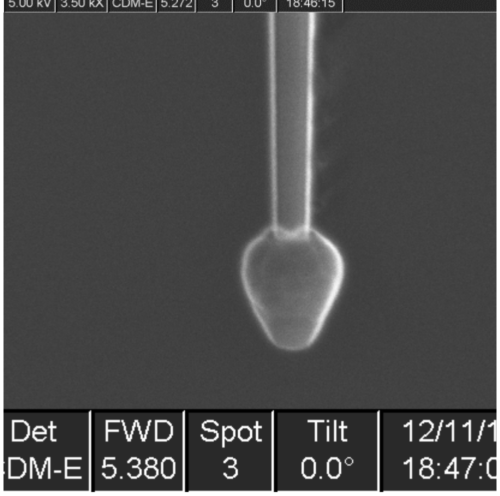
Undercut structure was protected well and there is no sign of attack.

For both test samples, Al_2O_3 ALD film coated the undercut successfully and prevented it from being etched by XeF_2.

Thermal ALD

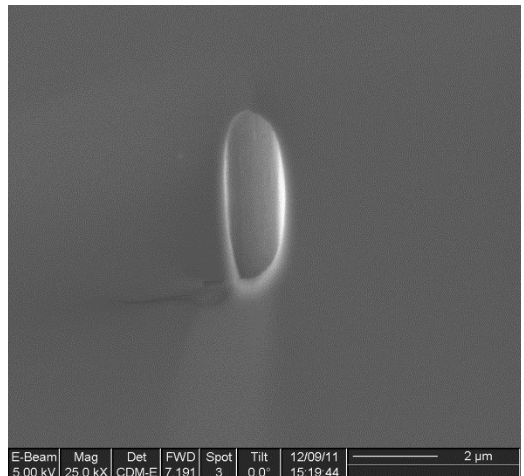
With SiO_2 on the sidewall





Undercut structure was protected nicely. Al_2O_3 film successfully coated the undercut.

Without SiO_2



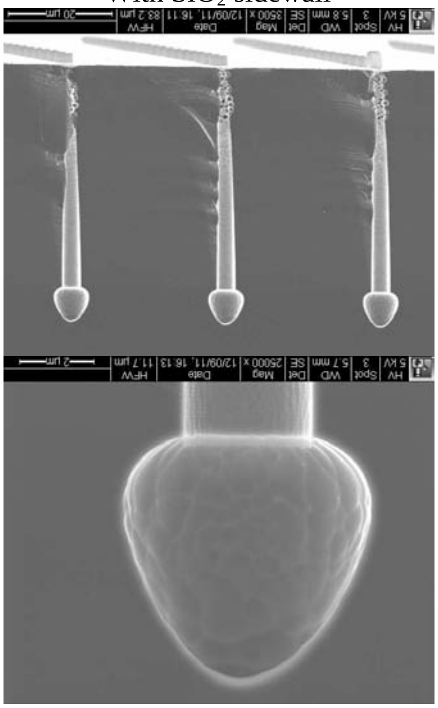
Sidewalls were protected very well with Al_2O_3 film, and the undercut structure was coated well too.

2.2 D = 3um

2.2.1 20 cycles

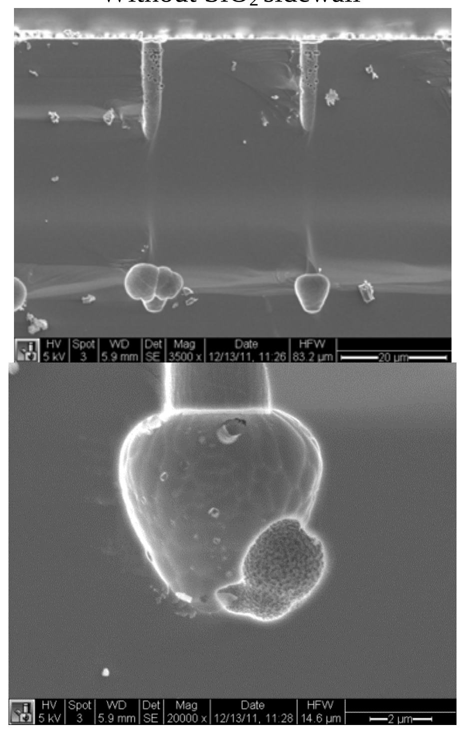
Plasma ALD

With SiO_2 sidewall



Undercut structure remained same as the non-etched reference

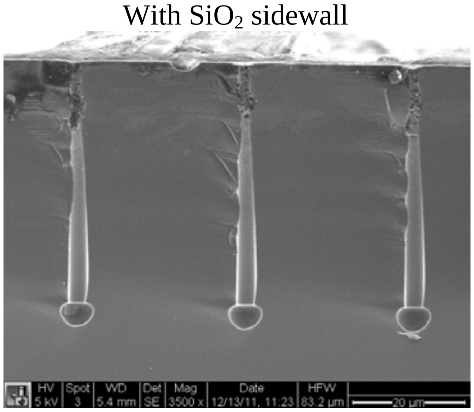
Without SiO_2 sidewall



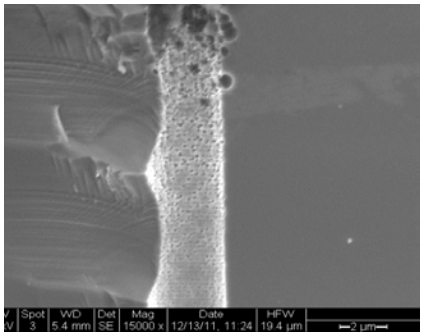
Although sidewalls were well protected, the undercut structure were damaged by XeF_2

Thermal ALD

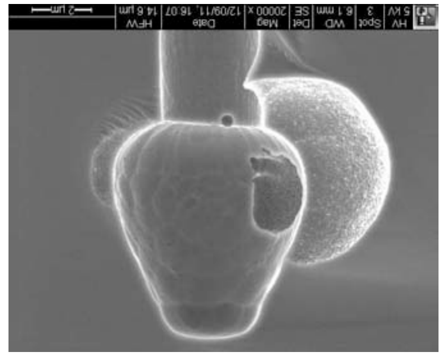
With SiO_2 sidewall



Without SiO_2 sidewall



Undercut structure remained same and protected well

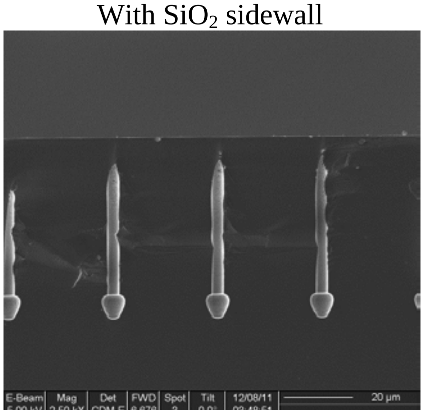


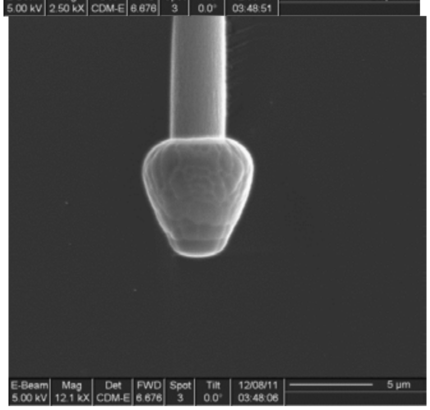
Although sidewalls were well protected, the undercut structure were damaged by XeF_2

2.2.2 100 cycles

Plasma ALD

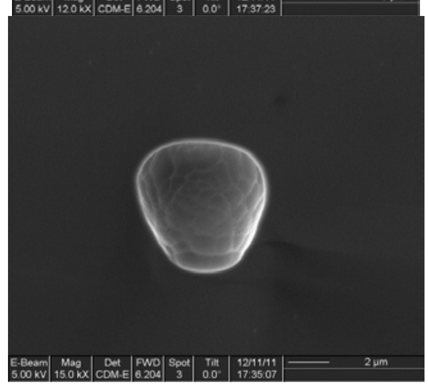
With SiO_2 sidewall





Undercut structures remained intact

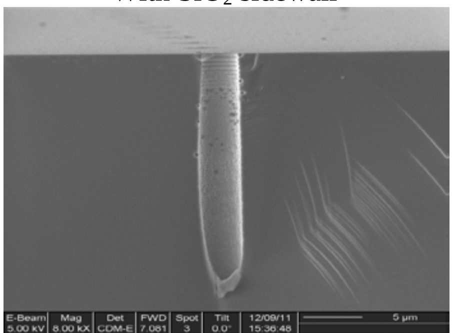
Without SiO_2 sidewall



Both sidewalls and undercuts showed no sign of XeF_2 attack

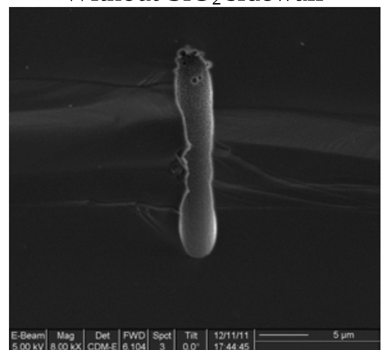
Thermal ALD

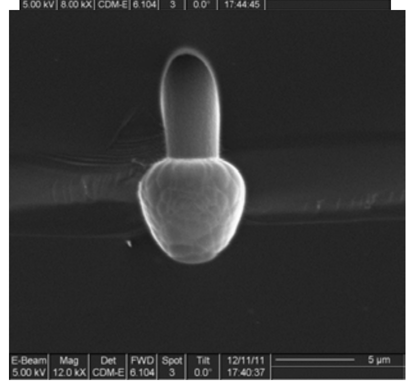
With SiO_2 sidewall



Undercut structures remained intact

Without SiO_2 sidewall





Both sidewalls and undercuts were coated well

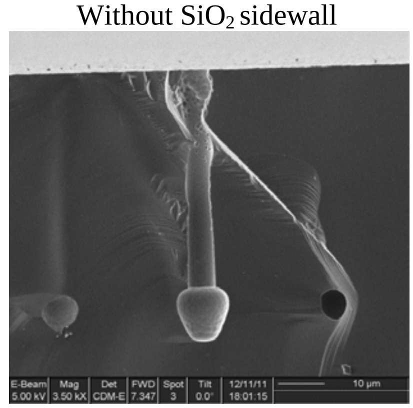
2.3 D = 5um

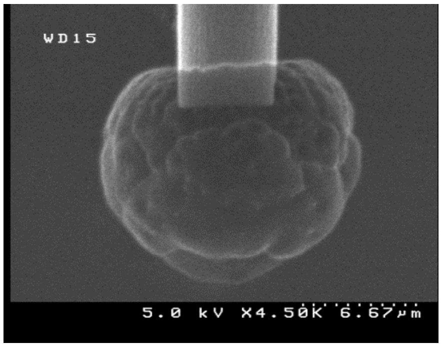
2.3.1 20 cycles

Plasma ALD

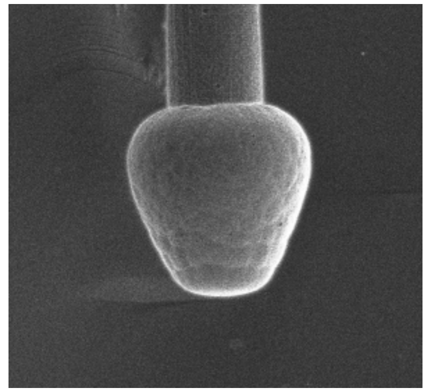
With SiO_2 sidewall

Without SiO_2 sidewall





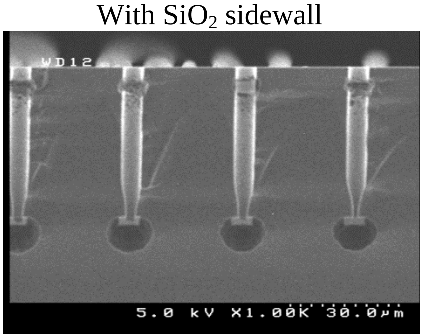
Undercut structure became much larger compared to the XeF_2 etched reference. This sample is suspicious and will be discussed later

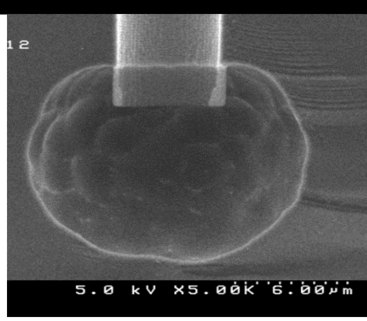


Whole structures are well protected

Thermal ALD

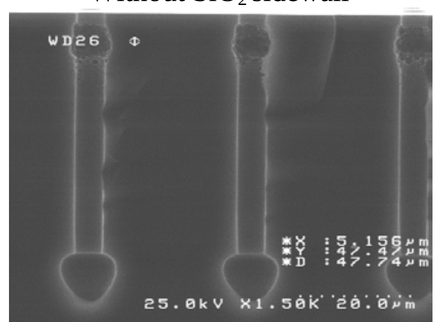
With SiO_2 sidewall

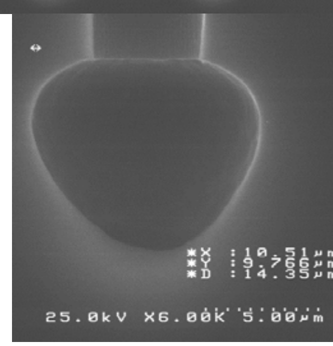




Undercut structure became much larger compared to the XeF_2 etched reference. This sample is also suspicious and will be discussed later.

Without SiO_2 sidewall



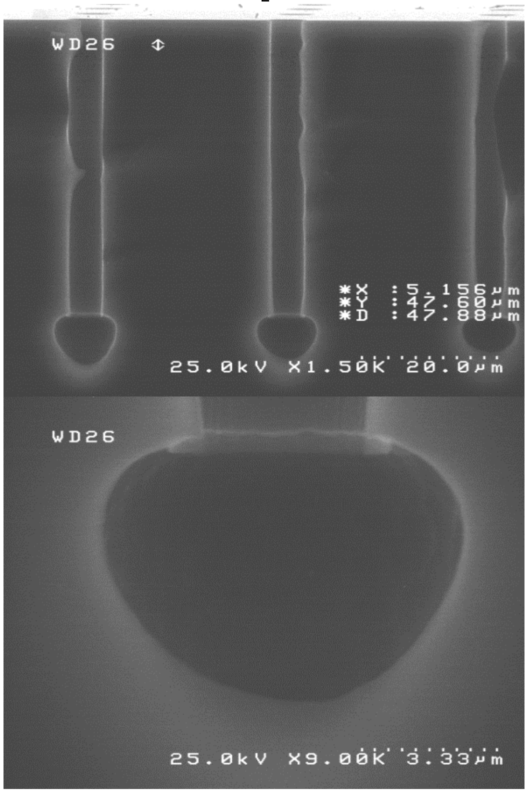


Whole structures are well protected

2.3.2 100 cycles

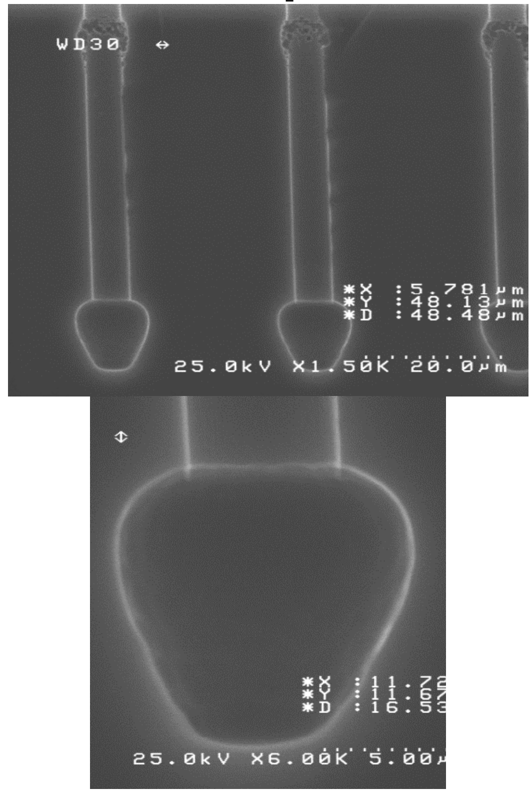
Plasma ALD

With SiO_2 sidewall



Undercut structure is preserved without any sign of XeF_2 attack

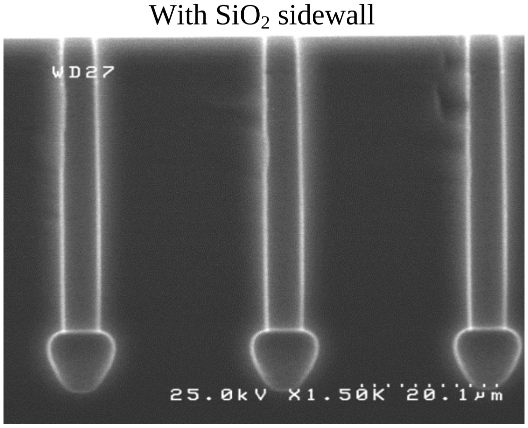
Without SiO_2 sidewall



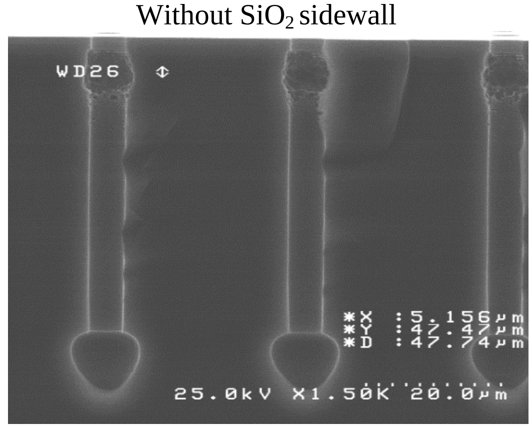
Whole structures are perfectly covered with Al_2O_3

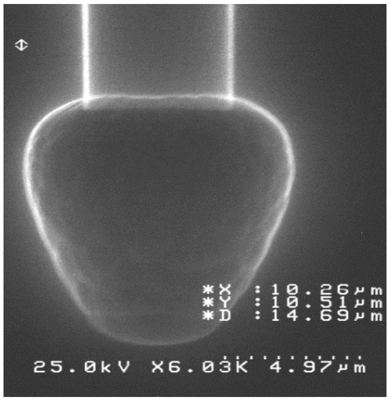
Thermal ALD

With SiO_2 sidewall

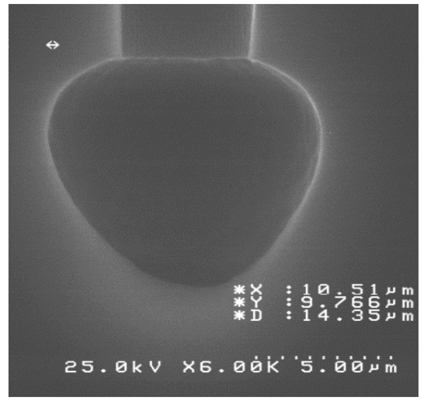


Without SiO_2 sidewall





Undercut structure is preserved well.



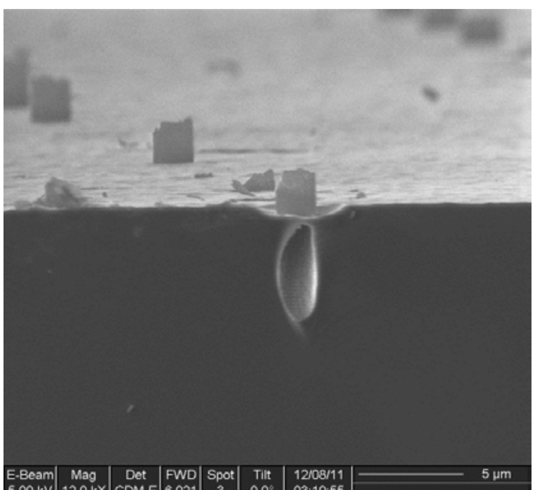
Whole structures are perfectly covered with Al_2O_3

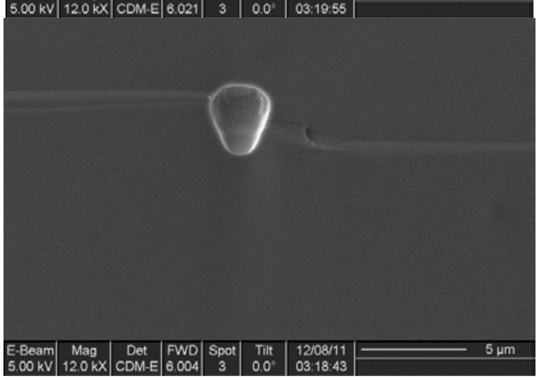
3 Savannah Al_2O_3

3.1 D = 1um

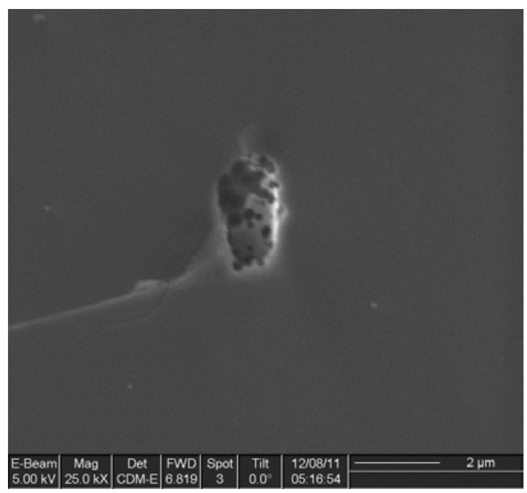
3.1.1 20 cycles

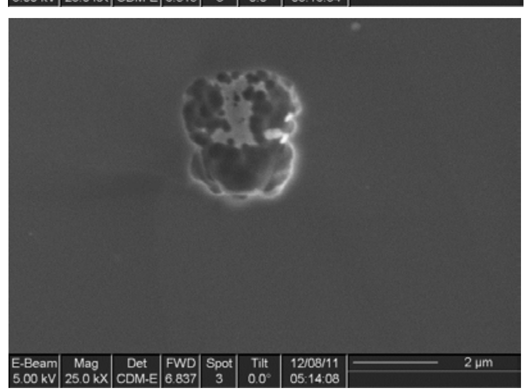
With SiO_2 sidewall





Without SiO_2 sidewall



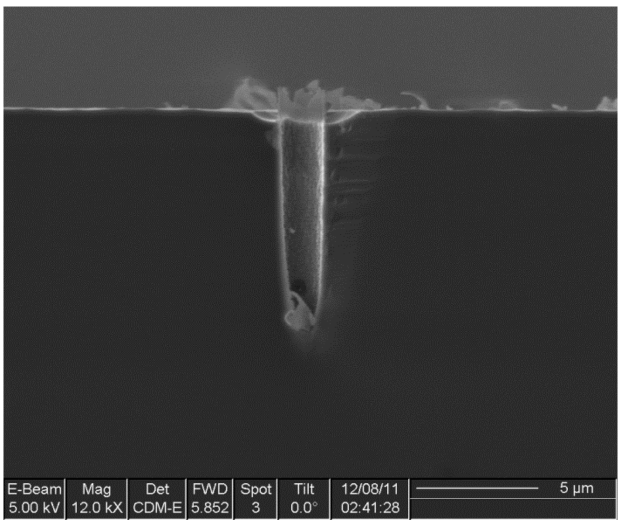


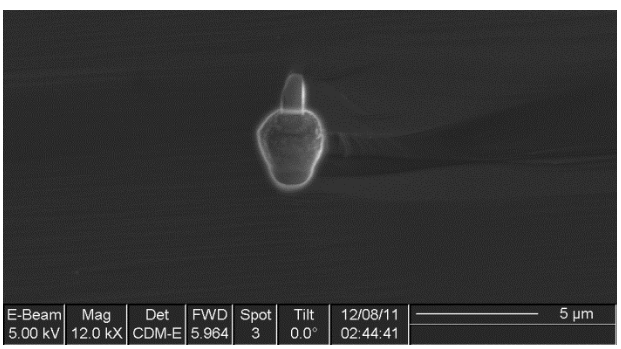
Since sidewall was protected by the combination of SiO_2 and Al_2O_3 film, sidewall protection by ALD cannot be judged. Undercut structure was protected completely by ALD coating. There is no pinhole at all for the entire structure.

Without SiO_2 on the sidewall, XeF_2 attacks sidewall heavily. Sidewall was deformed by the attack. Undercut structure was attacked by XeF_2 and deformed heavily.

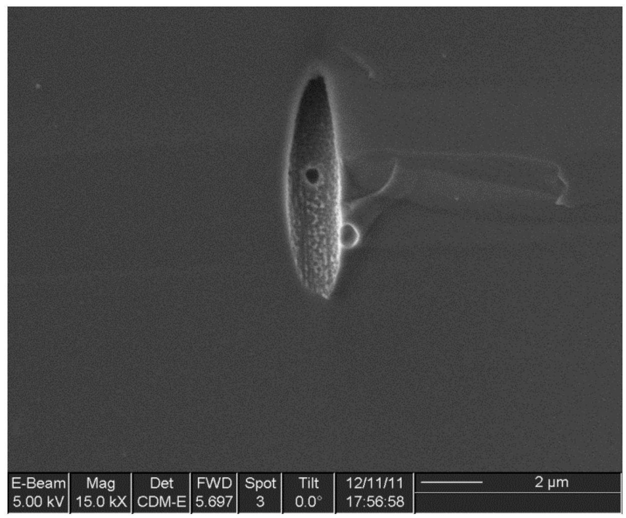
3.1.2 100 cycles

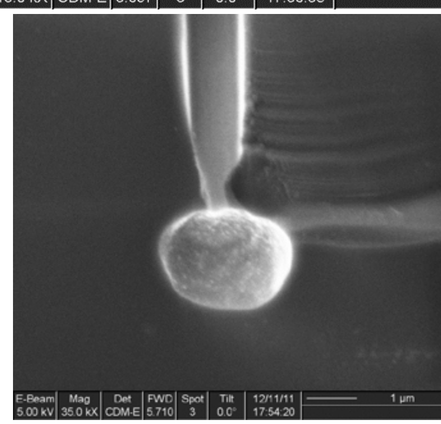
With SiO_2 sidewall





Without SiO_2 sidewall





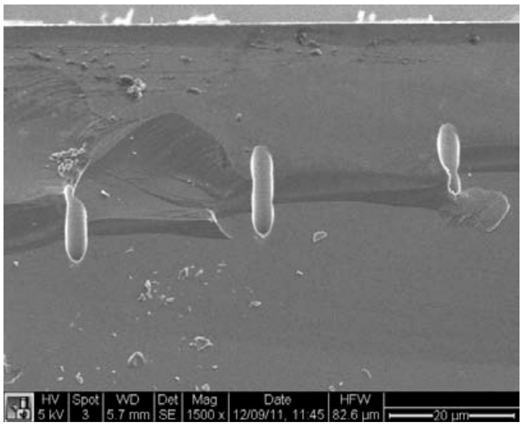
Since sidewall was protected by the combination of SiO_2 and Al_2O_3 film, sidewall protection by ALD cannot be judged. Undercut structure was protected completely by ALD coating. There is no pinhole at all for the entire structure.

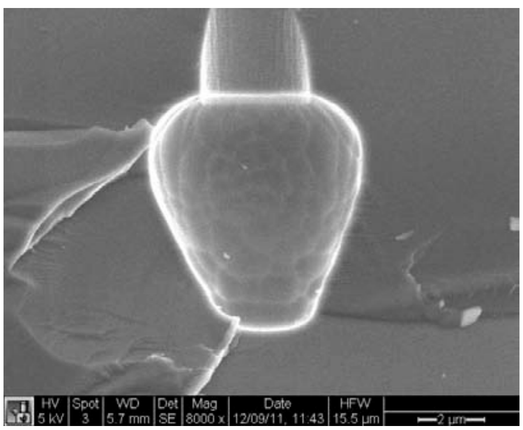
Without SiO_2 on the sidewall, XeF_2 attacks sidewall and creates cavity as an evidence of pinhole. Undercut structure remained same and protected by Al_2O_3 film.

3.2 D = 3um

3.2.1 20 cycles

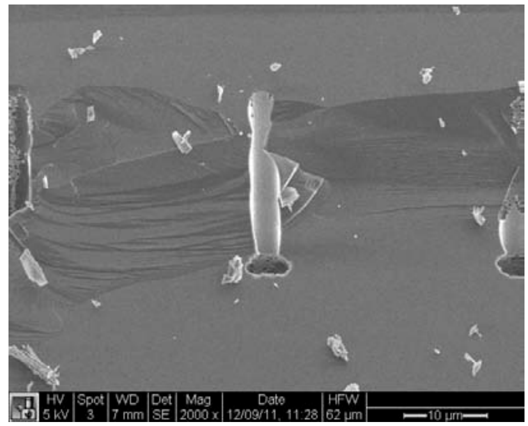
With SiO_2 sidewall

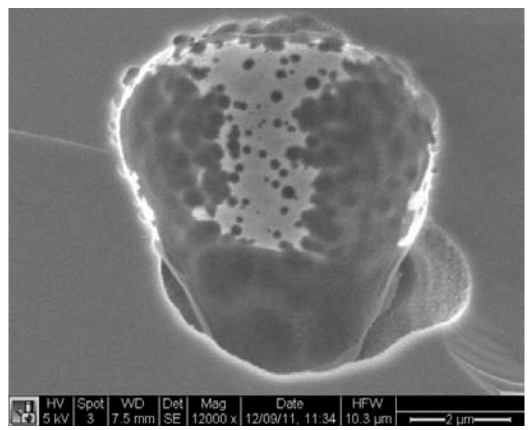




Since sidewall was protected by the combination of SiO_2 and Al_2O_3 film, sidewall protection by ALD cannot be judged. Undercut structure was protected completely by ALD coating. There is no pinhole at all for the entire structure.

Without SiO_2 sidewall

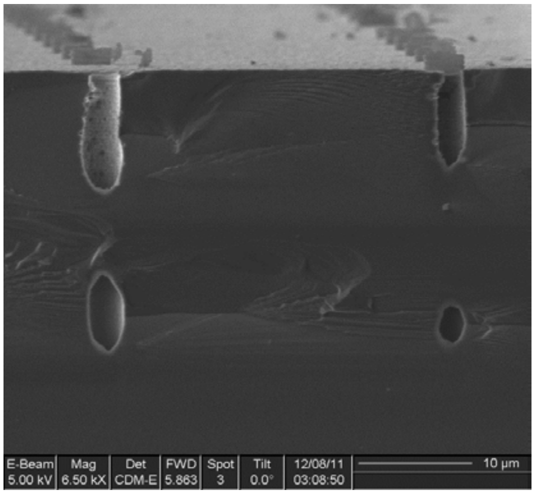


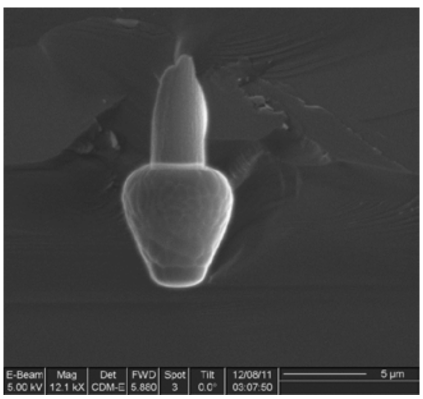


Without SiO_2 on the sidewall, Al_2O_3 film protected sidewall from XeF_2 attacks implying no pinhole. Undercut structure was attacked by XeF_2 and deformed heavily.

3.2.2 100 cycles

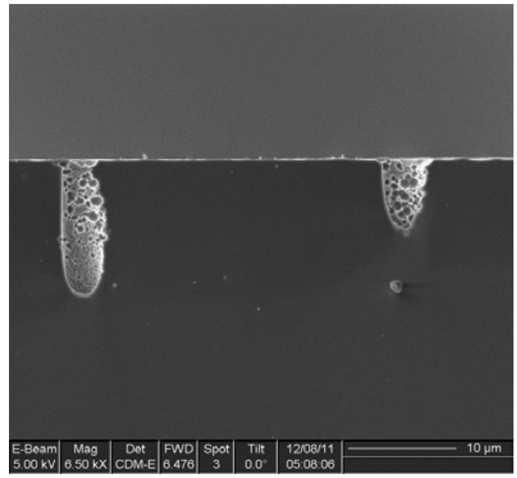
With SiO_2 sidewall





Since sidewall was protected by the combination of SiO_2 and Al_2O_3 film, sidewall protection by ALD cannot be judged. Undercut structure was protected completely by ALD coating. There is no pinhole at all for the entire structure.

Without SiO_2 sidewall

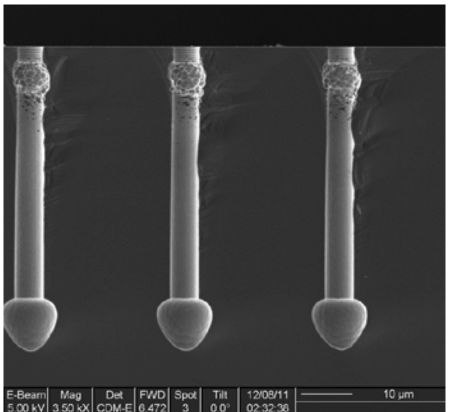


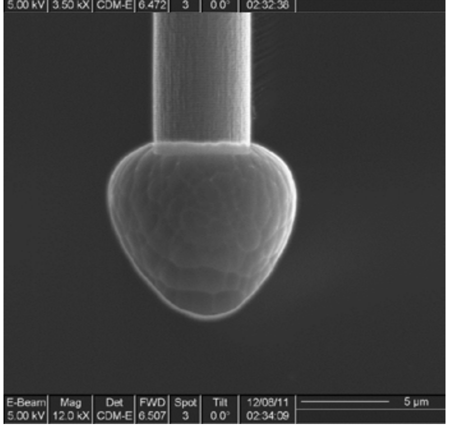
Without SiO_2 on the sidewall, Al_2O_3 film protected sidewall fairly from XeF_2 attacks but there could be some pinholes since Undercut structure remained same and protected by Al_2O_3 film.

3.3 D = 5um

3.3.1 20 cycles

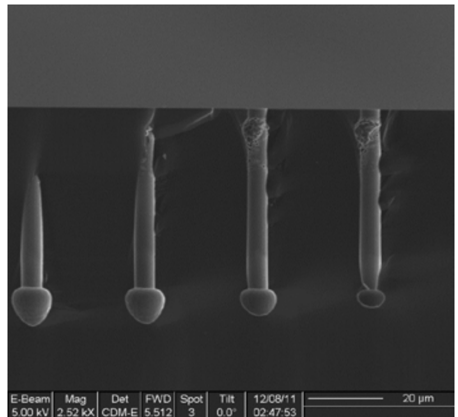
With SiO_2 sidewall

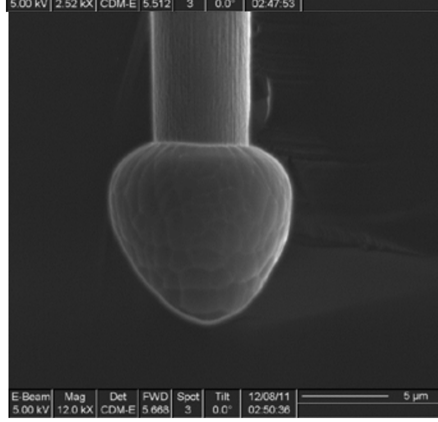




There is no pinhole at all for the entire structure

Without SiO_2 sidewall

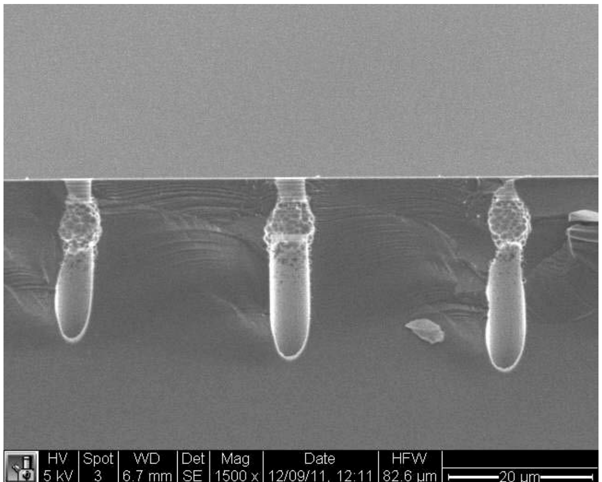


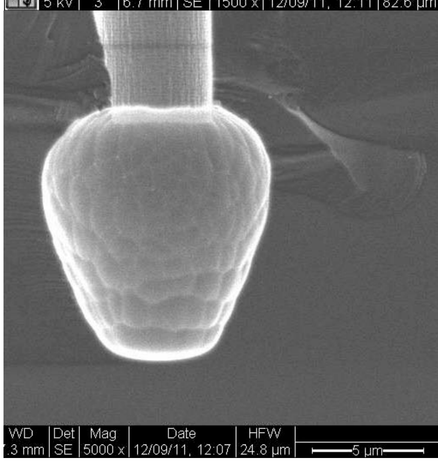


There is no XeF_2 attack observed over the entire structure

3.3.2 100 cycles

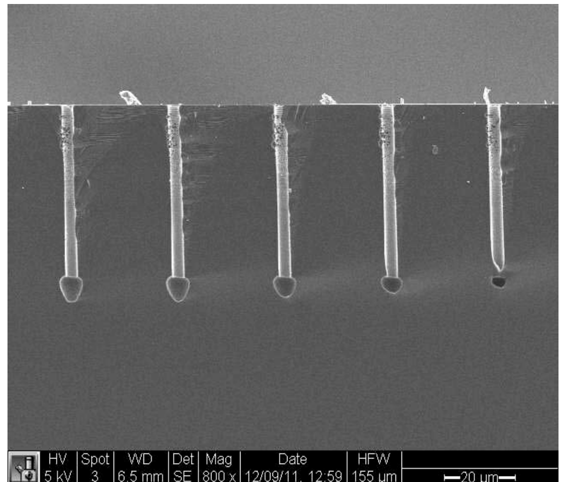
With SiO_2 sidewall

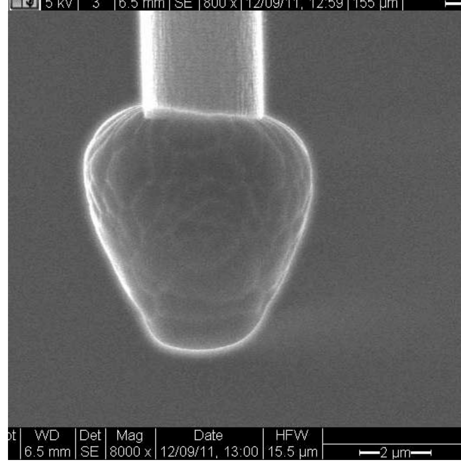




There is no pinhole at all for the entire structure

Without SiO_2 sidewall





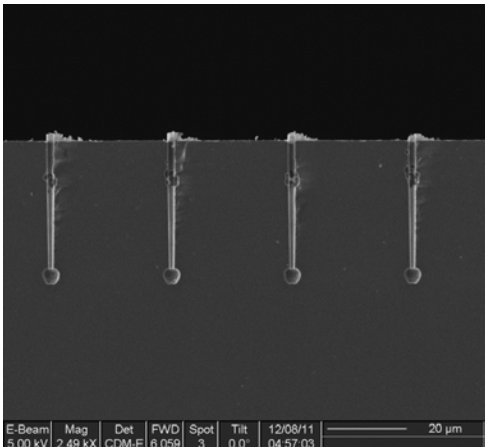
There is no XeF_2 attack observed over the entire structure

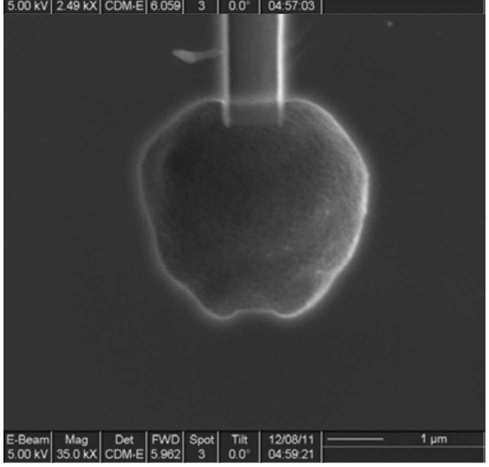
4 TiN

4.1 D = 1um

4.1.1 100cycles

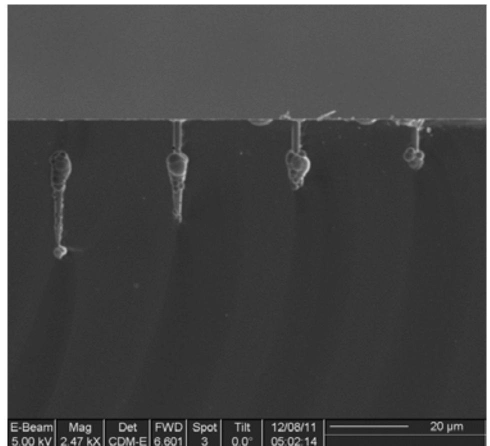
With SiO_2 sidewall





Since sidewall was protected by the combination of SiO_2 and TiN film, sidewall protection by ALD cannot be judged. Undercut structure was attacked by XeF_2.

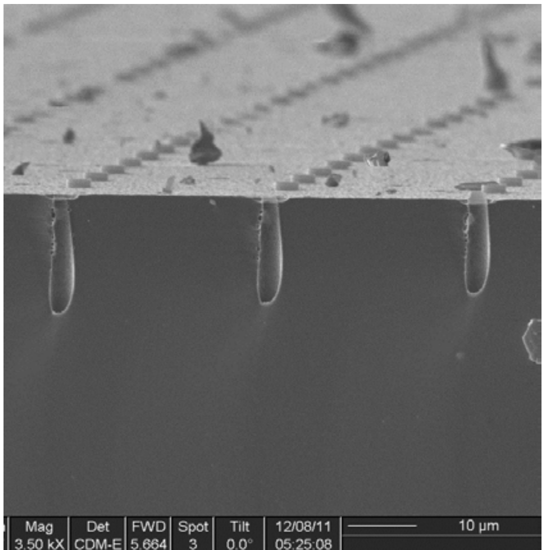
Without SiO_2 sidewall

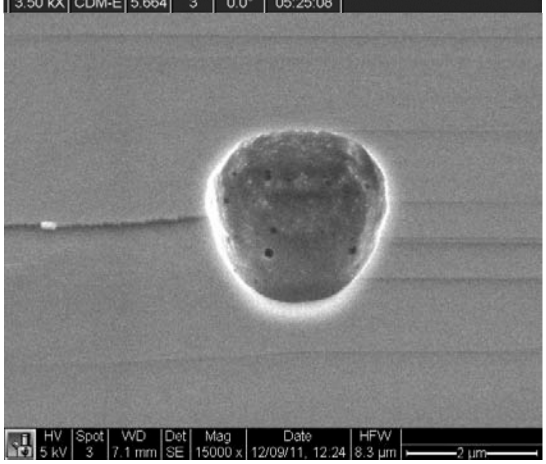


Without SiO_2 on the sidewall, XeF_2 attacks sidewall heavily. Sidewall was deformed by the attack. Undercut structure was attacked by XeF_2 and deformed severely.

4.1.2 400cycles

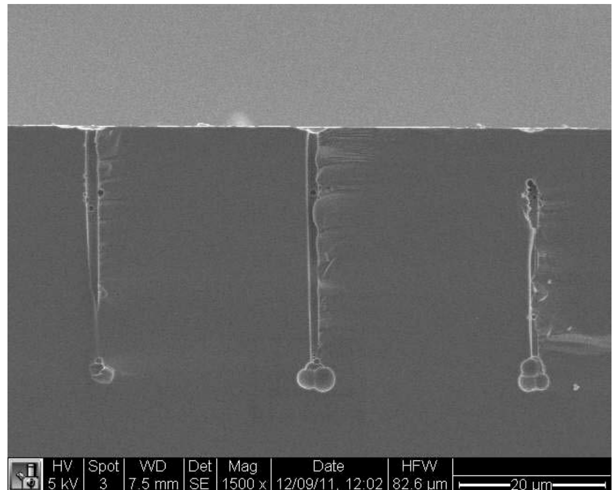
With SiO_2 sidewall

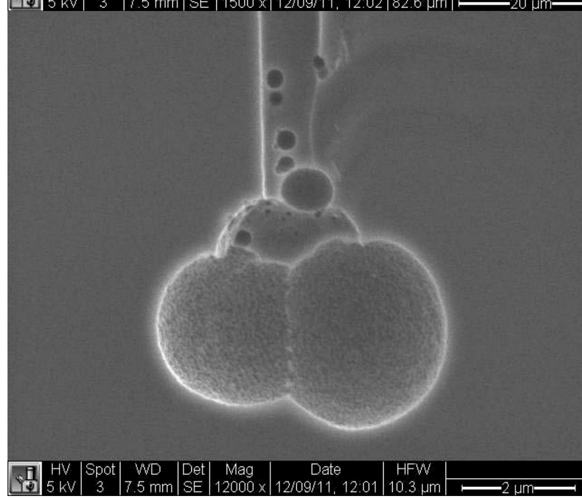




Since sidewall was protected by the combination of SiO_2 and TiN film, sidewall protection by ALD cannot be judged. Undercut structure got some pinholes after XeF_2 attack implying good coverage of TiN film.

Without SiO_2 sidewall



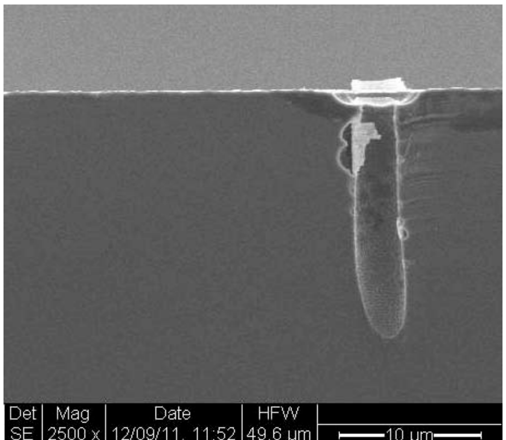


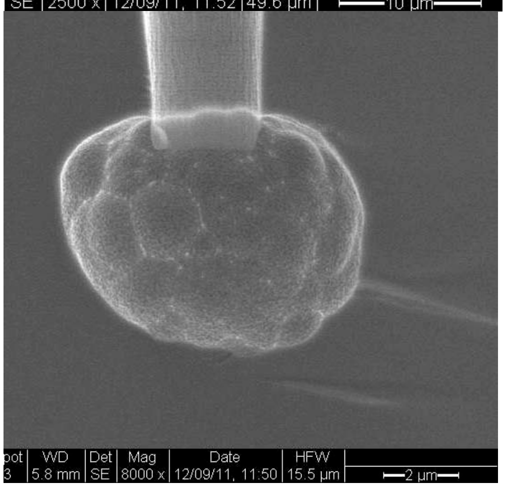
Without SiO_2 on the sidewall, XeF_2 attacks sidewall and creates cavity through pinhole of ALD layer. Sidewall was deformed by the attack. Undercut structure was attacked by XeF_2 and deformed severely.

4.2 D = 3um

4.2.1 100 cycles

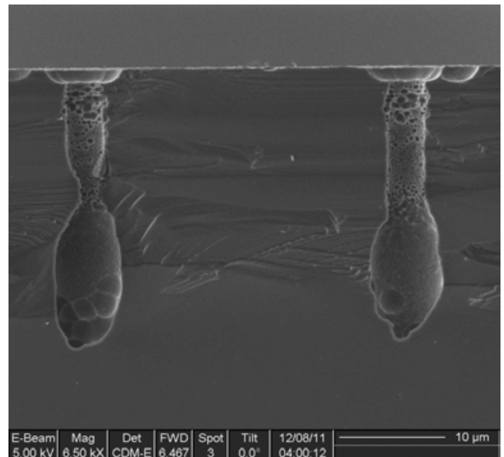
With SiO_2 sidewall





Since sidewall was protected by the combination of SiO_2 and TiN film, sidewall protection by ALD cannot be judged. Undercut structure got exploded by XeF_2 attack through pinhole.

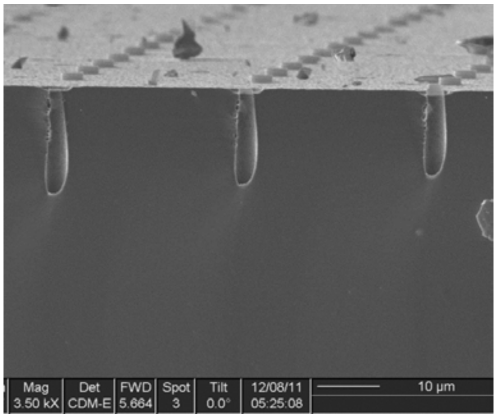
Without SiO_2 sidewall

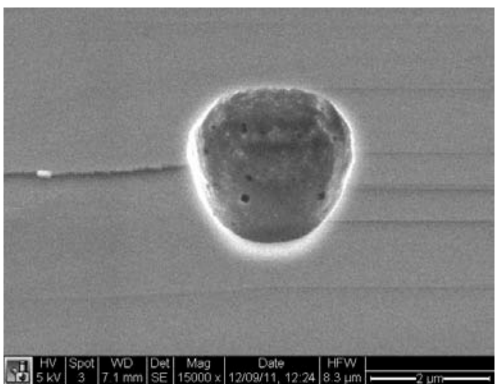


Without SiO_2 on the sidewall, XeF_2 attacks sidewall and deformed sidewall. Sidewall was deformed by the attack. Undercut structure was attacked by XeF_2 and deformed severely.

4.2.2 TiN 400 cycles (D = 3um)

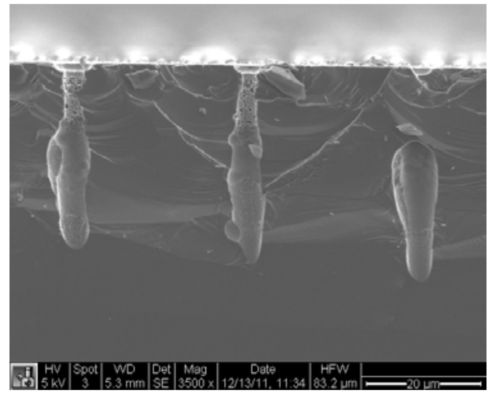
With SiO2 sidewall

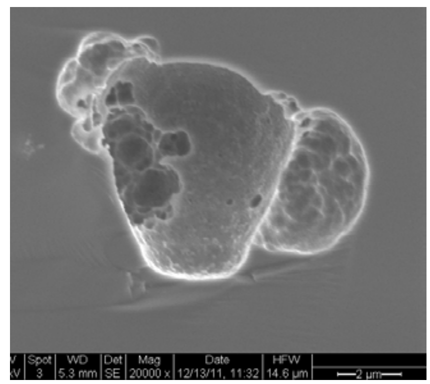




Since sidewall was protected by the combination of SiO_2 and TiN film, sidewall protection by ALD cannot be judged. Undercut structure got some pinholes after XeF_2 attack implying good coverage of TiN film.

Without SiO2 sidewall



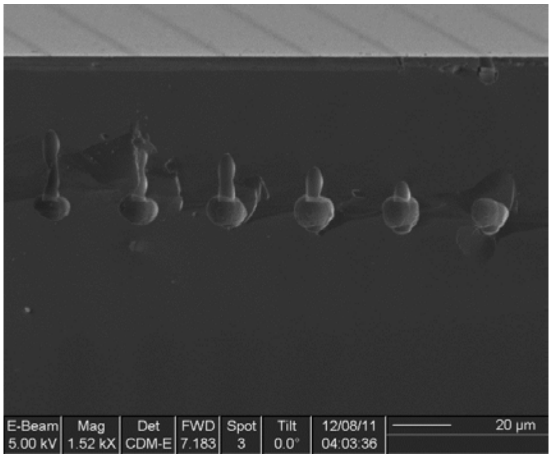


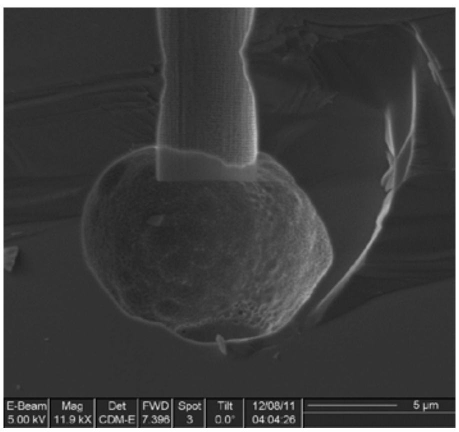
Without SiO_2 on the sidewall, XeF_2 attacks sidewall and deformed sidewall. Sidewall was deformed by the attack. Undercut structure was attacked by XeF_2 and deformed severely.

4.3 D = 5um

4.3.1 TiN 100 cycles (D = 5um)

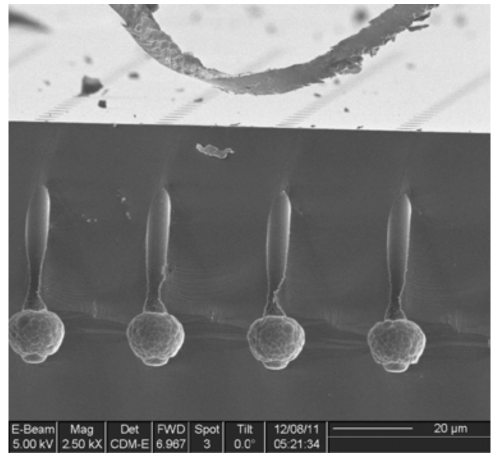
With SiO_2 sidewall

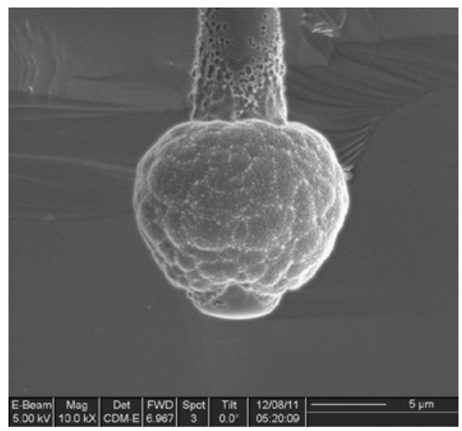




Since sidewall was protected by the combination of SiO_2 and TiN film, sidewall protection by ALD cannot be judged. Undercut structure got exploded by XeF_2 attack through pinhole.

Without SiO_2 sidewall

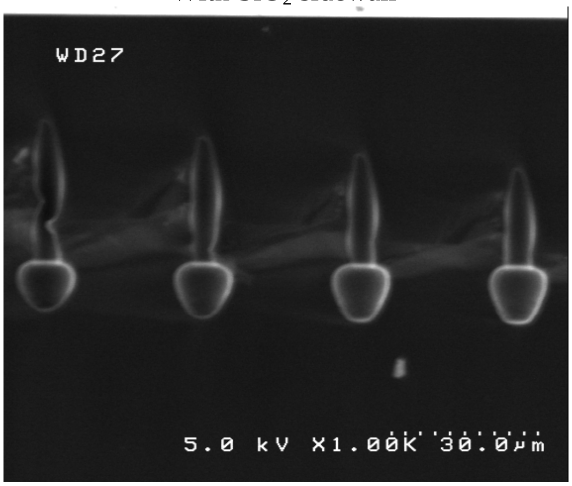




Without SiO_2 on the sidewall, Al_2O_3 film protected sidewall from XeF_2 attacks implying no pinhole. Undercut structure was attacked by XeF_2 and deformed heavily.

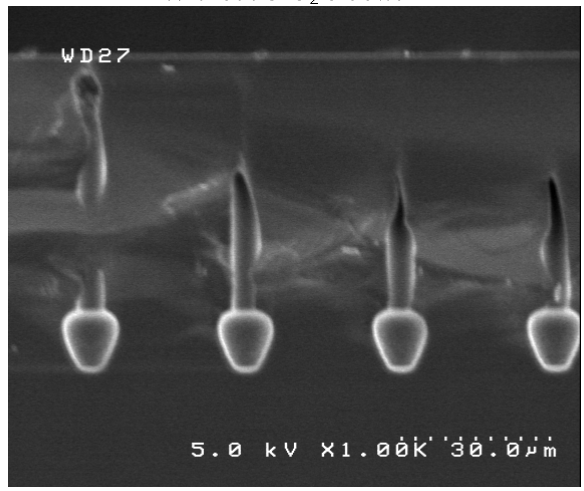
4.3.2 TiN 400 cycles (5um)

With SiO_2 sidewall



Since sidewall was protected by the combination of SiO_2 and TiN film, sidewall protection by ALD cannot be judged. Undercut structure was protected completely by ALD coating. There is no pinhole at all for the entire structure.

Without SiO_2 sidewall

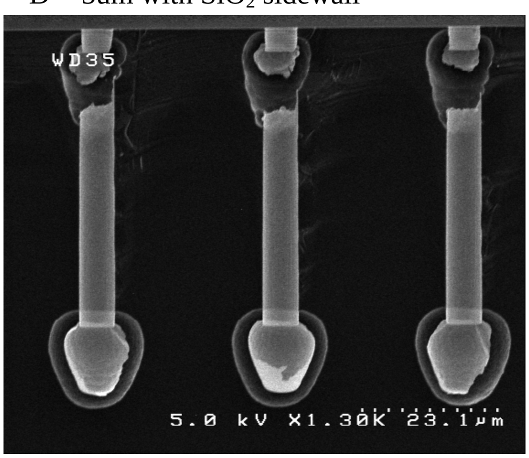


Undercut structure was protected well and there is no sign of attack. For both test samples, TiN ALD film coated the undercut successfully and prevented it from being etched by XeF_2.

5 Pt

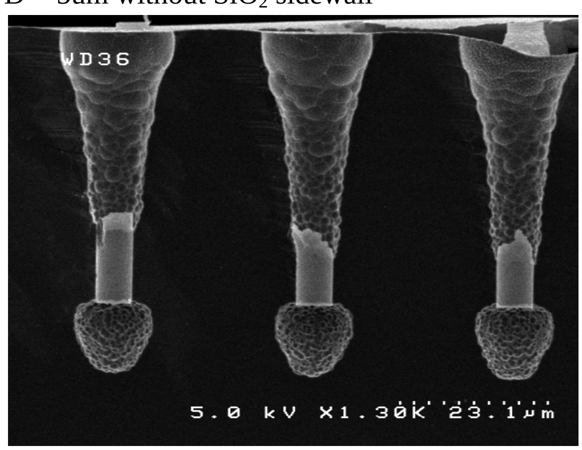
Plasma Platinum was deposited by 300cycles on the substrate having 5um hole with or without SiO_2 sidewall

D = 5um with SiO_2 sidewall



Since sidewall was protected by the combination of SiO_2 and TiN film, sidewall protection by ALD cannot be judged. The undercut structure was attacked by XeF_2 but interestingly the original shape of the structure remained.

D = 5um without SiO_2 sidewall



Without SiO_2 on the sidewall, XeF_2 attacks sidewall and deformed sidewall. Sidewall was deformed by the attack. Undercut structure was attacked by XeF_2 and deformed severely.

Discussion and Summary

We graded the ALD film coverage performance (Good/Fair/Poor) based on number and size of pinholes and overall shape change, and summarized the above SEM results in Table2-5 according to this rule.

Structure (w/ Oxide)	20 cycles		100 cycles	
	Plasma	Thermal	Plasma	Thermal
Undercut	1um: Poor 3um : Good 5um : Poor	1um: Good 3um: Good 5um : Poor	1um: Good 3um: Good 5um : Good	1um: Good 3um: Good 5um: Good

Structure (wo/ Oxide)	20 cycles		100 cycles	
	Plasma	Thermal	Plasma	Thermal
Sidewall	1um: Poor 3um: Good 5um : Good	1um: Good 3um : Good 5um : Good	1um: Good 3um: Good 5um : Good	1um: Good 3um: Good 5um : Good
Undercut	1um: Poor 3um: Poor 5um : Good	1um: Poor 3um: Poor 5um : Good	1um: Good 3um: Good 5um : Good	1um: Good 3um: Good 5um : Good

Table 2: Fiji Al_2O_3 summary table

Structure (w/ Oxide)	20 cycles Thermal	100 cycles Thermal
Undercut	1um: Good 3um : Good 5um : Good	1um: Good 3um: Good 5um : Good

Structure (wo/ Oxide)	20 cycles Thermal	100 cycles Thermal
Sidewall	1um: Poor 3um: Fair 5um : Good	1um: Fair 3um: Fair 5um : Good
Undercut	1um: Poor 3um: Poor 5um : Good	1um: Fair 3um: Good 5um : Good

Table 3: Savannah Al_2O_3 summary table

Structure (w/ Oxide)	100 cycles Thermal	400 cycles Thermal
Undercut	1um: Poor 3um : Poor 5um : Poor	1um: Fair 3um: Good 5um : Good

Structure (wo/ Oxide)	100 cycles Thermal	400 cycles Thermal
Sidewall	1um: Poor 3um: Poor 5um : Good	1um: Good 3um: Poor 5um : Good
Undercut	1um: Poor 3um: Poor 5um : Poor	1um: Poor 3um: Poor 5um : Good

Table 4: Plasma TiN summary table

Structure (w/ Oxide)	300 cycles Thermal
Undercut	5um : Poor

Structure (wo/ Oxide)	300 cycles Thermal
Sidewall	5um : Poor
Undercut	5um : Poor

Table 5: Plasma Pt summary table

The key points we have found out are:

- Performance of thermal Al_2O_3 coating was similar between Savannah and Fiji
- Conformal coating tends to get better with larger entrance
- Sidewall structures can always be coated better than undercut structures
- More cycles always enhances the film coverage over structures with tens of microns deep sidewall and undercut
    - ➢ 100 cycles of Plasma/Thermal Al_2O_3 ALD
    - ➢ 400 cycles of TiN Plasma ALD if hole diameter is larger than 5um
- The SiO_2 sidewall helps the coating of undercut structure
- D=5um, 20 cycles of Plasma/Thermal Al_2O_3 ALD do not match exactly the tendency above
    - ➢ Data shows somehow confusing results
    - ➢ More experiments required for clearer result

Acknowledgements

We appreciate J Provine's kind advice and help throughout the course. We also would like to thank Dr. Mary Tang, Professor Roger Howe, and Professor Olav Solgaard for their guidance and discussion.