Micromachining of Hafnium Carbide

ME-401 Semester Project in Mechanical Engineering

Author:
Fabian BAUER

Supervisors:
Prof. Guillermo VILLANUEVA
Marco LIFFREDO

Lab:
Advanced NEMS Laboratory
EPFL

In collaboration with:
[redacted] Sarl

Abstract

During this project, the microfabrication of Hafnium Carbide (HfC) was investigated. Multiple tools were considered and tested for their compatibility and effectiveness when working with HfC.

The main method of choice for the machining of HfC thin films was Ion Beam Etching (IBE). In the first step, the etch rate and selectivity were determined using three different methods. Then IBE was used successfully together with a standard photolithography process to create a pattern etched through the HfC film. The dependence of the etch recipe on the etch rate and sidewall verticality was then explored. Additionally, a second process using a Silicon Oxide (SiO) hard mask was tested to improve the results with regard to sidewall verticality and fencing. It was found to lead to a slight improvement.

Two methods of releasing Micro-Electro-Mechanical-Systems (MEMS) devices were also investigated. For the release from a Si substrate Xenon difluoride (XeF2) was tested and judged a suitable means. For the release of devices from a SiO release layer vapour HF was tested but found not to be compatible with HfC and leading to undesirable reactions on the surface.

Ellipsometry was checked as a method of determining the thickness of the HfC film but was found to be unsuitable due to material being too absorptive.

Contents

Introduction

The goal of this semester's project was to investigate the micromachining of HfC in the context of MEMS devices. The project was done in collaboration with [redacted] Sarl who provided the materials.

To ensure the fabrication of a MEMS device is possible, the following three processes need to be verified.

- **Photolithography:** To pattern the different layers necessary in most MEMS devices there must be a Photolithography (PL) process that is compatible with the material. Compatible here means that the Photoresist (PR) needs to stick to the material and stay put during the further steps (i.e. etching). There then needs to be a way to remove the PR without damaging the material. In this report the methods investigated are the standard processes of the Center of MicroNanoTechnology (CMi) using the AZ ECI 3027 resist [1] as well as the use of a hard mask made from sputtered SiO. To remove the PR wet removal was investigated together with oxygen plasma.
- **Etching of the material:** There needs to be a way of etching the material of interest. In this report, the etching method investigated is IBE. This was chosen as IBE is known to be a universal etchant[2].
- **Selective etching of the substrate/release layer:** To release future devices an etching method has to be found that selectively etches the substrate (Si in this case) or release layer (SiO in this case) without attacking the material. In this report, the methods investigated are XeF2 gas etching as well as vapour HF etching. XeF2 was an interesting candidate for the selective etching of Si as it is known to be isotropic and to have a high selectivity for many different mask materials[3]. No reports were found for the selectivity with regards to HfC. Vapour HF was an interesting candidate for the removal of SiO as it is known to etch SiO isotropically[4]. Again no reports were found for vapour HF in combination with HfC.

In order to validate these processes suitable metrological tools have to be found. The main tools used in this report are optical microscopy, SEM, mechanical profilometry as well as spectroscopic reflectometry.

State of the Art

Hafnium Carbide is a material that is interesting for many different applications such as field emitters[5][6] and solar absorbers [7].

HfC could also be of interest in the fabrication of MEMS and NEMS devices. Given my best effort I was unable to find any examples of people micromachining or etching HfC. I was also not able to find examples of the use of HfC in any actualMEMS.

There is a lot of information available on the deposition of HfC. Methods that have been investigated are sputtering [7][6], electron beam evaporation [5] and chemical vapour deposition [8].

There is also a lot of information available on IBE in general. In particular it is known that ion beam etching is a universal etchant that is able to etch nearly all materials[9][2].

Fabrication of Samples

This section will give an overview of the fabrication of the samples used for the validation of the different processes. A detailed description with all steps listed for all samples as well as the exact process parameters for every step can be found in appendix D. The work was performed between October and December 2022. Details of what work was done and when can be found in appendix B.

All fabrication work mentioned in this report was performed in the cleanrooms at EPFLs CMi. More information on what machines were used can be found in appendix I.

For the tests different pieces of wafers were provided by [redacted] with HfC films of different thicknesses on them. An overview of the provided samples can be found in appendix E.

3.1 Photolithography

Nearly all samples were subjected to a PL step during their fabrication. The PL step consists of the sub steps: Surface Preparation, coating, exposure, development and resist removal. The details of the steps are different depending on if the PR is applied directly to the HfC or if a SiO hardmask is used.

3.1.1 Surface Preparation

For the samples not using a hardmask the samples were simply dehydrated on a hotplate. The exact times and temperatures can be found in appendix D. For the first sample using a hardmask a simple dehydration step was also used but this lead to the PR delaminating. Therefore all following samples with a hardmask had a vapour HMDS step done to make the surface hydrophobic. This eliminated the problem of the PR delaminating. The vapour HMDS step was performed using the ATMsse VB20 vapour prime module integrated into the same machine as the ATMsse SB20 manual coater.

3.1.2 Coating

All samples were coated with AZ ECI 3027 resist using the ATMsse SB20 manual coater. After coating the samples were subjected to a softbake. The spin coater recipe as well as the bake temperature and time for each sample can be found in appendix D.

3.1.3 Exposure

All samples were exposed using the Heidelberg Instruments MLA150 laser writer. The exact details of the exposure can be found in appendix D. This tool was chosen as it allows for great flexibility as no mask is needed. The smallest feature size possible of ca. $1\mu m$ was sufficient for our application.

3.1.4 Development

All samples were developed manually in a beaker using AZ 726 MIF developer solution. They were then rinsed with water in another beaker and then dried using nitrogen. Some of the samples were subjected to a Post Development Bake (PDB) to improve the resist adhesion. The exact development times can be found in appendix D.

3.1.5 Removal

After the PR is no longer needed it was removed using a combination of oxygen plasma and wet removal. For the oxygen plasma either the Tepla 300 or Tepla GiGAbatch Microwave plasma ashers were used. Wet removal was done using heated baths of remover 1165 in a wetbench.

3.2 Deposition of SiO

The SiO used as a hardmask was deposited using the Pfeiffer SPIDER 600 sputter cluster system. The recipe and time used can be found in appendix D

3.3 SiO Etching

The etching of the SiO hardmask was done using SPTS APS Dielectric Etcher. This machine uses an inductively coupled plasma and fluorine chemistry[10]. The process parameters used can be found in appendix D.

3.4 IBE

All IBE was performed using the Veeco Nexus IBE350. The process parameters used can be found in appendix D. The results of the IBE are further discussed in chapter 5.

3.5 XeF2 etching

The XeF2 etching was performed using the SPTS Xactix X4 silicon etching system. Details on the recipes used can be found in appendix D. The results of the XeF2 etching are further discussed in chapter 6.

3.6 Vapour HF Etching

The machine used for the vapour HF etching was the SPTS μEtch HF vapour phase etcher. Details on the recipes used can be found in appendix D. The results of the vapour HF etching are further discussed in chapter 7.

Metrology

4.1 Optical Microscopy

Optical microscopy was used as a tool to quickly verify many of the steps done during the fabrication. The microscope models used were the Leica DM800 as well as the Nikon LV150. Both of them are equiped with digital cameras. All images obtained can be found in appendix F.

4.2 Scanning Electron Microscopy

To get more detailed images SEM was used. The instrument of choice was the Zeiss Leo 1550. The samples were usually cleaved in half in order to expose the cross-section before being imaged using a section holder. All SEM images obtained can be found in appendix F.

4.3 Mechanical Profilometry

For mechanical profilometry a Bruker Dektak XT surface profiler was used. The measurement results are provided in appendix H.

4.4 Spectroscopic Reflectometry

A FilMetrics F20-UV was used to try to measure the thickness of the HfC film (see section 5.1.1). It was found that a reliable measurement is only possible for very thin films (≈100nm). For thicker films fitting the measured data was no longer possible. Data from the literature [11] was used to fit the measured spectrums.

4.5 Ellipsometry

Ellipsometry was investigate as an alternative method of measuring the HfC film thickness and to get better values of n and k for the use in the FilMetrics UV20.

Ellipsometry measures the change of polarization incurred when light reflects or transmits from a material structure. By then fitting a model to this data different material parameters such as the film thickness and optical constants can be determined. [12]

Using the Woolam RC2 it was tried to measure the thickness of the HfC film on a piece of the "100 nm" wafer. It was not possible to fit the data to a model. It was therefore concluded that the film is too absorbent to be measured for the thickness of 100nm. As no thinner films were available this route of investigation was abandoned. This result is not very surprising as HfC is known to be very absorbent in the wave length range used by the Woolam RC2[7][13].

Ion Beam Etching of HfC

This chapter deals with different aspects of IBE of HfC.

5.1 Etch Rate Determination

This section describes how the etch rate for HfC was determined. Three different methods were used: Global etching of a chip in combination with a spectroscopic measurement, patterned etching in combination with SEM imaging and patterned etching in combination with mechanical profilometry.

5.1.1 Method 1: Global Etching and using Spectroscopic Measurement

The first experiment was done on a piece of roughly 1 cm^2 (sample 1.0) that was cleaved of from the wafer with the 100 nm film of HfC. The sample was then mounted on a carrier wafer using QuickStick™135 (QS135). The thickness of the HfC film was then determined at multiple spots using a FilMetrics™F20-UV spectroscopic reflectometer. The location of the spots is shown in Figure 5.1. Note that point 1 and point 6 coincide. This was done to determine the repeatability of the measurement. The sample was then etched for 1 minute. The full details of the fabrication can be found in appendix D. The thickness was then measured again. The measured values together with their Goodness of Fit (GOF) can be found in Table 5.1.

	Before IBE		After IBE		
Point	Thickness [nm]	GOF [%]	Thickness [nm]	GOF [%]	Difference [nm]
1	98.59	99	65.32	98	33.27
2	90.31	95	62.97	99	27.34
3	91.16	95	64.34	99	26.82
4	91.44	96	65.98	98	25.46
5	90.78	96	66.48	99	24.30
6	91.74	96	63.22	99	28.52
	Average Difference (excluding point 1)				26.5

Table 5.1: Thickness of sample 1.0 before and after 1 minute of IBE at medium power and -10° in the Veeco Nexus IBE350,
Note: the thickness value for point 1 before IBE is much higher than the value for point 6 which corresponds to the same measurement location. It is therefore assumed that this value is erroneous. It is likely that instead of the thickness the GOF got noted two times. Point 1 has therefore been excluded from the average.

From this measurements the etch rate can be estimated using the difference between the thickness before and after the IBE. The average of this difference is then taken. It found to be 26.5 nm. As the etch time was one minute it follows that the etch rate is approximatively 26.5 nm/min.

5.1.2 Method 2: Using patterned Etching and SEM

The second test was performed on a sample (sample 2.1) of roughly $15mm$x$15mm$ cleaved of from the wafer with the $600nm$ film. Lines of various widths were etched into it. The details of this can again be found in appendix D. Next the sample was cleaved perpendicular to the etched lines and the cross section was imaged under the SEM. The resulting image can be seen in Figure 5.2.

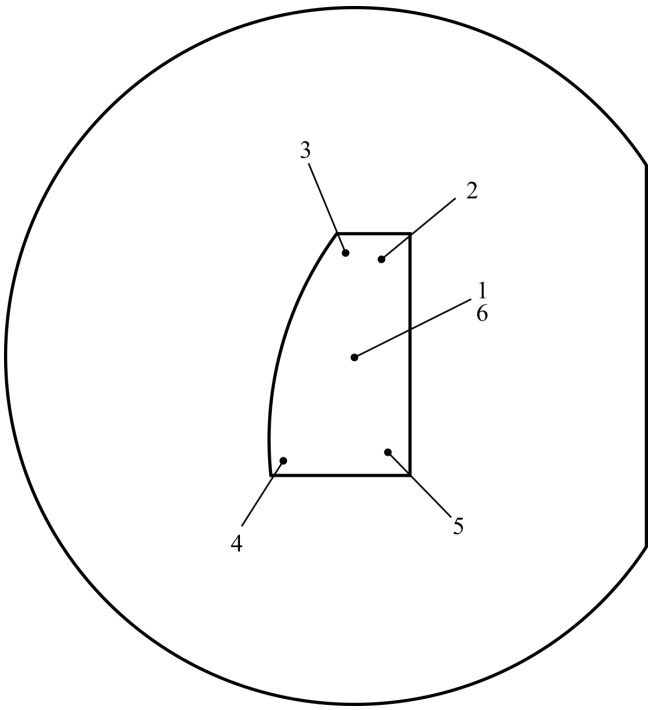


Figure 5.1: Measurement positions on sample 1.0 used for the determination of the thickness pre and post IBE.

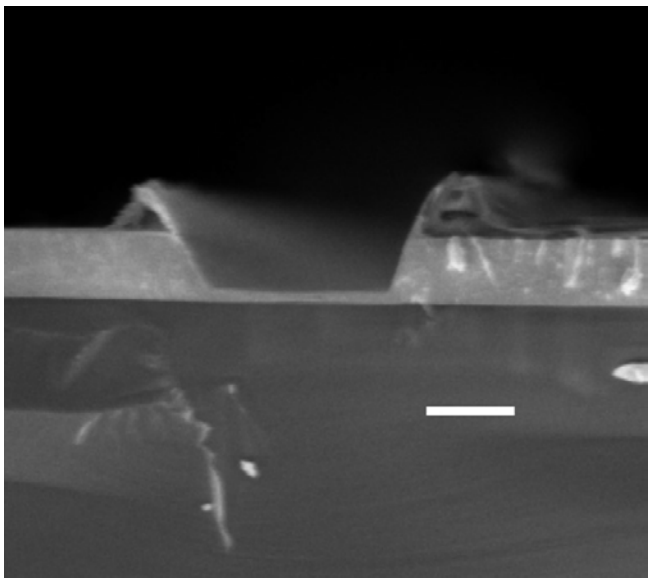
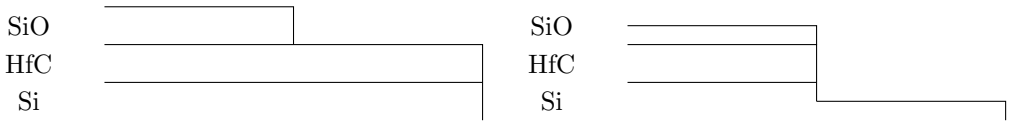


Figure 5.2: Cross section of sample 2.1. Scale bar 1 μm

Measuring on the image gives a thickness of the HfC film of ca. 750 nm. The measurement for the remaining HfC at the bottom of the trench gives a thickness of ca. 150 nm. Therefore ca. 600 nm of HfC have been etched within 25 minutes. This gives an average etch rate of ca. 24 $\frac{nm}{min}$ which is close to the value obtained by the first method.

5.1.3 Method 3: Using patterned Etching and Mechanical Profilometry

A third experiment was done in order to validate the etch rate. A step feature on sample 2.6 was measured after the etching of the hardmask and then again after the etching of the HfC. Because we wanted to keep the possibility of a release open the HfC layer was overetched in order to expose the underlying Si. The resulting situation is presented in Figure 5.3.



(a) before IBE (b) after IBE

Figure 5.3: Step used for profilometry (not to scale)

The step size before IBE (h_0) was ca. 1900 nm. The step size after IBE (h_f) was ca. 1700nm. The final step (h_f) height is constituted of the remaining thickness of the SiO mask (h_{SiO}),the height of the HfC layer (h_{HfC}) and the step height in the Si layer (h_{Si}). Therefore we can write

$$h_f = h_{SiO} + h_{HfC} + h_{Si} \tag{5.1}$$

Because we know the etchrate of SiO (r_{SiO}) and Si (r_{Si}) we can write

$$h_{SiO} = h_0 - r_{SiO}t \tag{5.2}$$

$$h_{Si} = r_{Si}t_{Si} \tag{5.3}$$

where t is the total etch time and t_{Si} is the time the Si was exposed to the etching which is not known. Combining (5.1),(5.2) and (5.3) gives

$$h_f = h_0 - r_{SiO}t + r_{Si}t_{Si} + h_{HfC} \tag{5.4}$$

As we know h_{HfC} from the previous experiment we can solve this for t_{Si}

$$t_{Si} = \frac{1}{r_{Si}}(h_f - h_0 - h_{HfC} + r_{SiO}t) \tag{5.5}$$

We also know that

$$t = t_{Si} + t_{HfC} \tag{5.6}$$

Combining (5.5) and (5.6) gives

$$t_{HfC} = t - \frac{1}{r_{Si}}(H_f - h_0 - h_{HfC} + r_{SiO}t) \tag{5.7}$$

We can calculate the etch rate of the HfC as

$$r_{HfC} = h_{HfC}/t_{HfC} \tag{5.8}$$

Inserting (5.7) gives

$$r_{HfC} = h_{HfC}(t - \frac{1}{r_{Si}}(h_f - h_0 - h_{HfC} + r_{SiO}t))^{-1} \tag{5.9}$$

h_{HfC}	750nm
r_{Si}	39.00 nm/min[1]
r_{SiO}	37.44 nm/min[2]
h_f	1700 nm
h_0	1900 nm
t	23 min

Table 5.2: Numerical values used in the determination of the HfC etch rate

Inserting the numerical values presented in Table 5.2 gives us an etch rate of 29.7 nm/min.

[1]Given by Veeco for the IBE350
[2]Given by Veeco for the IBE350

5.1.4 Results

The results for the three different methods are reported in table 5.3

Method 1	Method 2	Method 3
26.5	24.0	29.7

Table 5.3: Etch rates as determined by the three different methods

5.1.5 Discussion

One can note that the values obtained by the three different methods are close to each other.

The etch rate obtained using the SEM image of the cross section is probably the most reliable as it does not rely on external data such as the etch rates in method three or the spectral data in method one.

It is important to note that the etch rate in IBE is highly dependant on the angle of incidence [2][9]. The etch rate found here is therefore only valid for an angle of incidence of 10°. This fact also has affected our result in method three as the test was performed at -10° but the values given by the manufacturer for the etch rates of Si and SiO are for normal incidence.

An even more reliable measure for the etch rate could be obtained by using method three without overetching so the etch rate of Si does not need to be known. Combining this with a similar experiment to determine the etch rate of SiO at the same angle could provide a very reliable and exact result.

5.2 Effect of the Etching Recipe

After the etch rate had been determined the next thing of interest was the side wall verticality and the effect of the etching recipe on it.

5.2.1 Experiment Performed

Two samples were fabricated (sample 2.2 and 2.3). The details of this can be found in appendix D. Sample 2.2 got etched at -10° for 30 min. Sample 2.3 was first etched at medium power and -10° for 15 minutes followed by 1 minute of cooling. Then the angle got changed to -40° and the sample was etched for 7.5 minutes followed again by 1 minute of cooling. Then the angle got changed once again this time to -70° and the sample was etched again for 7.5 minutes followed again by 1 minute of cooling[3]. After the etching the samples were removed from the carrier and cleaved perpendicular to the etched lines. They were then imaged using the SEM.

5.2.2 Results

The result can be seen in Figure 5.4.

(a) Sample 2.2

(b) Sample 2.3

Figure 5.4: Samples 2.2 and 2.3 after IBE

[3]This recipe was suggested by Nan XU.

5.2.3 Discussion

It can be seen from the cross section images that fencing is present. Fencing is caused by redeposition of material onto the sidewall of the PR mask. It is a well known phenomena in IBE [2]. One can see that the thickness of the PR has been selected a bit to thick as in the ideal case the facet of the PR would meet up with the top corner of the HfC Sidewall. It can also be seen that changing the recipe to the second variant does not significantly improve the verticality of the sidewalls.

5.3 Effect of a SiO Hardmask

5.3.1 Experiment Performed

In an effort to improve the side wall verticality the use of a SiO hardmask was investigated. To do so SiO was deposited onto the remainder of the "600nm" wafer using the Pfeiffer SPIDER 600 [4]. The exact process parameters used can be found in appendix D Table D.5.

From this wafer three pieces were cleaved off (samples 2.5,2.6 and 2.7). They were then patterned before being etched in the SPTS APS Dielectric Etcher[5]. The parameters used can be found in appendix D Table D.6. Exact fabrication details can be found in appendix D.

It was then verified that the APS etching did not attack the HfC by looking at the cross section using the SEM. One resulting image can be seen in Figure 5.5. The height of the SiO hardmask was then verified on sample 2.6 using the DekTak XT profilometer. The resulting measurement can be found in Figure 5.6. It can be seen that the SiO layer has a thickness of approximatively 2μm.

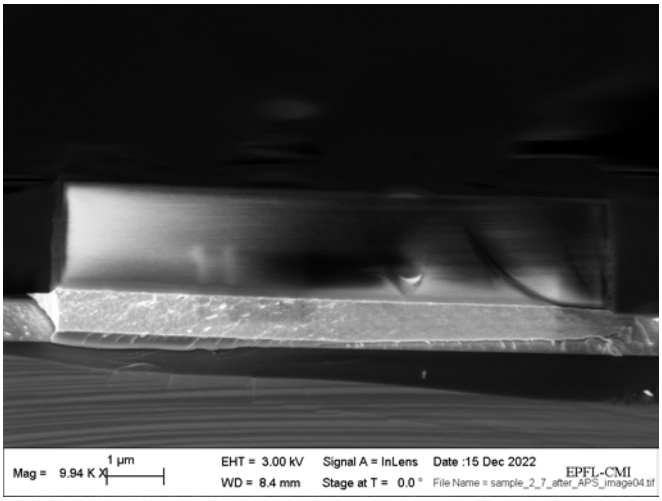


Figure 5.5: Sample 2.7 after etching in APS

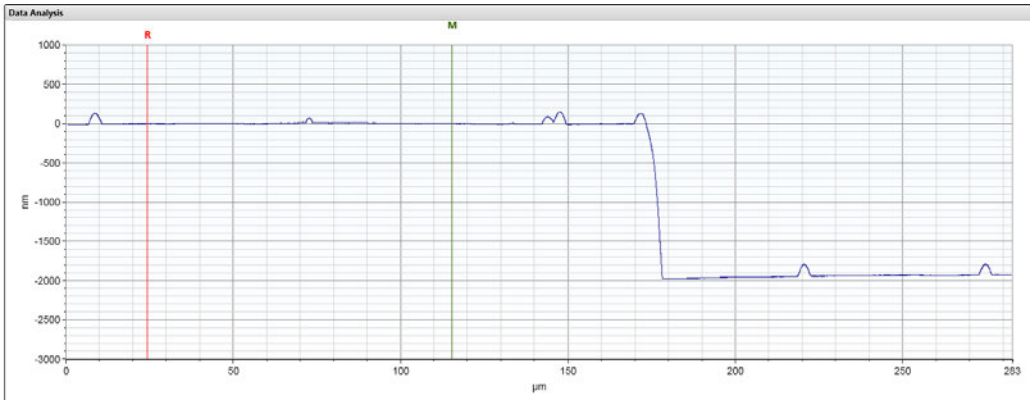


Figure 5.6: Profile measurement of sample 2.6 after etching in APS

[4]Note: The deposition was performed by Marco LIFFREDO as I did not have the appropriate training for the machine.

[5]Note: The etching was performed by Frederico PERETTI as due to machine and trainer availability it was not possible for me to get the training until later in the project.

In a next step samples 2.5 and 2.6 were etched using IBE. The settings that were used can be found in appendix D. End Point Detection (EPD) was used. As can be seen in Figure 5.7 the endpoint was visible at 20 minutes. To ensure that we got completely through the HfC even in the small trenches 3 more minutes were added to the etch and then it was stopped manually. Overetching is important in this case as we want to release the device later on so we have to have access to the underlying Si.

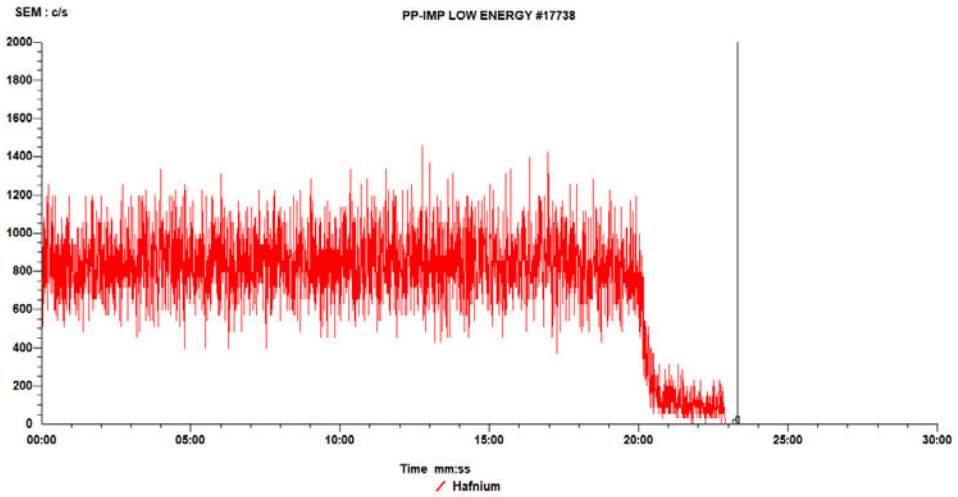


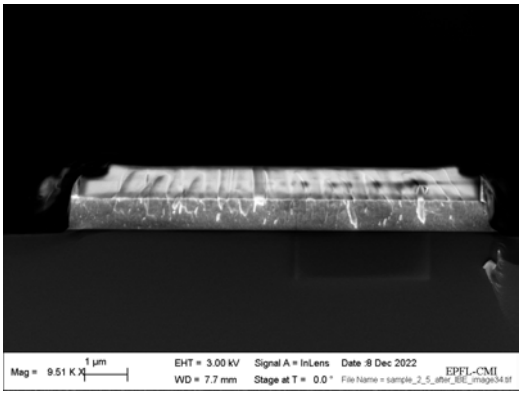
Figure 5.7: EPD graph of IBE of sample 2.5

5.3.2 Results

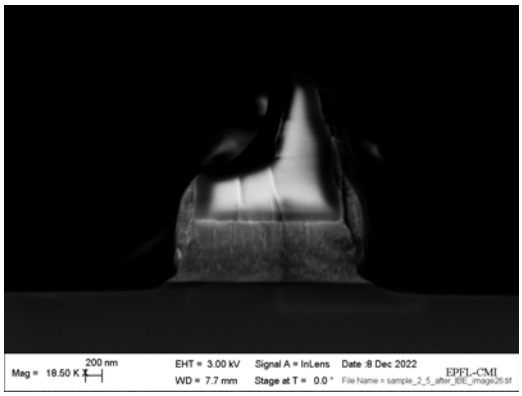
A selection of the resulting cross sections can be found in Figure 5.8. The full set of obtained cross section images is available in appendix F.

5.3.3 Discussion

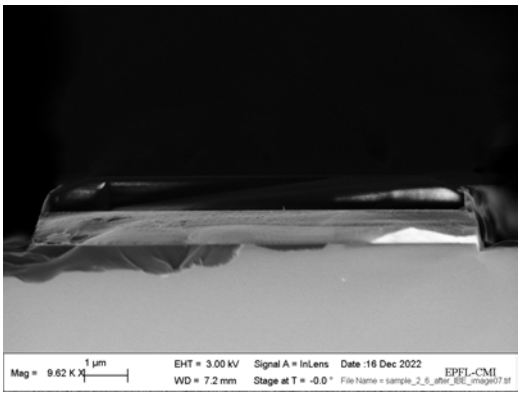
It can be seen that for all the cross sections the side wall verticality is similar or slightly better then for the case without a hardmask. Fencing is present in all samples.



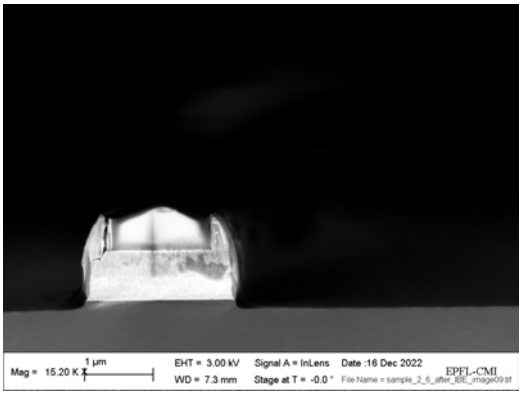
(a) Sample 2.5



(b) Sample 2.5



(c) Sample 2.6



(d) Sample 2.6

Figure 5.8: Samples 2.5 and 2.6 after IBE

XeF2 Release

For the potential release of a future device it was interesting to investigate the behaviour of HfC when exposed to XeF2. To do this sample 2.2 was used the fabrication of which is explained in appendix D. The etching was performed using the SPTS Xactix X4 system in zone 11. The parameters for the recipe used can be found in appendix D Table D.8.

After the etch it could be seen under the optical microscope that the structures on the sample got damaged. It is suspected that this is because of a high stress level in the HfC layer.

The cross section of the sample was then examined using the SEM. One of the images can be found in Figure 6.1.

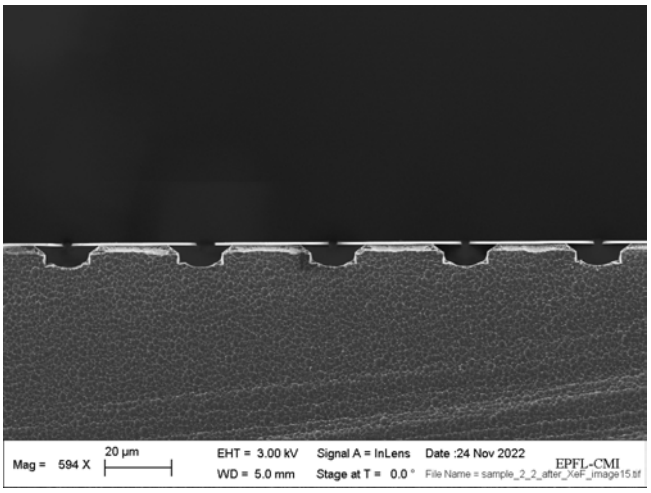


Figure 6.1: Cross section of sample 2.2 after XeF2 etching.

Measuring the thickness of the HfC layer on the image shows that its thickness remains unchanged to within the measuring tolerance. It can therefore be concluded that HfC is not attacked by XeF2. This makes XeF2 etching a suitable method for releasing HfC layers from a Si substrate. It is necessary though to control the stress present in the layer so that it does not tear itself apart on release.

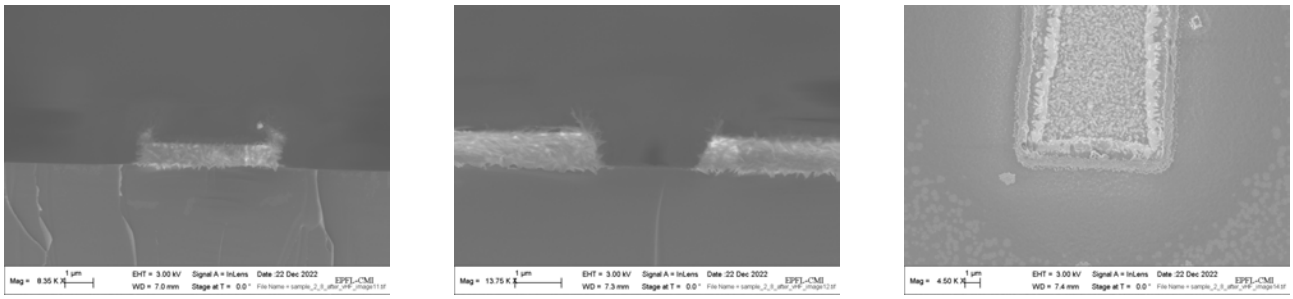
Vapour HF Mask Removal

7.0.1 Experiment Performed

The SiO hardmask needed to be removed to allow for imaging of potential fencing present on the sample 2.8. To do so vapour HF was chosen as this method could also be used to release future devices if the HfC is on top of a SiO release layer. The etching was performed using the SPTS uEtch vapour phase etcher in zone 2. Before the etching the sample was baked at 170°C for ca. 5 minutes using the hotplate to the left of the etcher. This baking was performed in order to make sure no moisture was present on the sample surface. The sample was then etched using the parameters shown in appendix D Table D.7.

7.0.2 Results

After the etching the sample was inspected using the SEM. Some resulting images can be seen in Figure 7.1.



(a) Wall (b) Trench (c) Tip of a cantilever

Figure 7.1: Sample 2.8 after vapour HF etching

It can clearly be seen from these images that some needle like structures have formed on the surface of the HfC. It can therefore be concluded that there was some sort of reaction. It follows that vapour HF is not a process that is compatible with HfC.

Conclusion

Form the work done during this project we can conclude the following.

- The standard PL recipe can be used for AZ ECI 3027 on top of HfC without adhesion issues.
- IBE is a suitable method for machining HfC. The etch rate is similar to that for PR giving us a selectivity of about 1:1. The selectivity for a SiO mask is about 1:1.5. A side wall verticality of about 10° was achieved for a PR mask with a slight improvement for a SiO mask. Fencing is present but can be minimized by a good choice of recipe.
- XeF2 does not attack HfC and is therefore a suitable method of releasing devices made from HfC from a Si substrate.
- Vapour HF does react with HfC it is therefore not a suitable method to release a device from a SiO release layer.
- The available optical measurement instruments at CMi are not suitable for the thickness measurement of a HfC film with a thickness of more than a few nanometers.

Recommended next Steps

If the project is to be continued I recommend the following steps to be performed next.

1. **Investigate Deposition:** The samples that were available for this project proofed unsuitable to fabricate a released device this is suspected to be due to a high surface stress in the HfC layer. The recommendation would therefore be to try to find a suitable method for the deposition of HfC thin films with a lower stress.
2. **Selective Etching of SiO:** A future device will need to be released from an underlying release layer (that needs to be an insulator as well). Therefore a method of selectively etching SiO needs to be found that does not attack the HfC.
3. **Wire Bonding:** For a future device it will be necessary to verify that it is possible to wire bond to the HfC layer.
4. **Verification of Composition:** To remove the PR from the sample oxygen plasma needed to be used. It should be verified that this does not lead to an undesirable change in surface composition.

Bibliography

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[2] Robert E Lee. "Microfabrication by ion-beam etching". In: ().

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List of Acronyms

CMi Center of MicroNanoTechnology

EPD End Point Detection

GOF Goodness of Fit

HF Hydrofluoric acid

HfC Hafnium Carbide

IBE Ion Beam Etching

MEMS Micro-Electro-Mechanical-Systems

PDB Post Development Bake

PEB Post Exposure Bake

PL Photolithography

PR Photoresist

QS135 QuickStick™135

SEM Scanning Electron Microscope

SiO Silicon Oxide

XeF2 Xenon difluoride

List of Figures

List of Tables

Lab Journal Summary

The following table gives an overview of what work was done in the clean room and on what date.

Date	Work Done
13. Oct. 2022	Coating of Si test wafer with PR, training MLA 150 on test wafer, training manual PR development
14. Oct. 2022	Training resist removal using wetbench.
17. Oct. 2022	Training Tepla
18. Oct. 2022	Cleaving and mounting of sample 1.0, thickness measurement sample 1.0 using FilMetrics UV 20
19. Oct. 2022	IBE of sample 1.0 using Veeco Nexus IBE350, thickness measurement sample 1.0
21. Oct. 2022	Cleaving of sample 2.0
24. Oct. 2022	Photolithography on sample 2.0, IBE of sample 2.0, resist removal sample 2.0
25. Oct. 2022	Failed imaging session on sample 2.0
27. Oct. 2022	Cleaving of sample 2.1
31. Oct. 2022	Photolithography of sample 2.1, IBE of sample 2.1, resist removal 2.1
02. Nov. 2022	SEM inspection sample 2.1
08. Nov. 2022	Cleaving of samples 2.2 and 2.3, Training DekTakXT
09. Nov. 2022	SEM inspection sample 2.0
14. Nov. 2022	Photolithography samples 2.2 and 2.3
15. Nov. 2022	IBE samples 2.2 and 2.3, SEM inspection sample 2.2
17. Nov. 2022	SEM inspection sample 2.3
21. Nov. 2022	Cleaving of sample 3.0, Photolithography samples 2.4 and 3.0
22. Nov. 2022	IBE of sample 3.0
23. Nov. 2022	Resist removal samples 2.2 and 3.0, Training SPTS Xactix X4 with sample 2.2
24. Nov. 2022	SEM inspection sample 2.2
28. Nov. 2022	Resist removal sample 2.4
29. Nov. 2022	IBE sample 2.4
30. Nov. 2022	Cleaving of samples 2.5/2.6/2.7, Photolithography samples 2.5,2.6 and 2.7
01. Dec. 2022	QS135 of samples 2.5,2.6 and 2.7
02. Dec. 2022	SEM inspection of sample 2.4
06. Dec. 2022	Resist removal 2.5/2.6/2.7, DekTak XT measurement sample 2.6
07. Dec. 2022	QS135 samples 2.5 and 2.6, IBE of sample 2.5 and 2.6, DekTak XT measurement sample 2.5 and 2.6
08. Dec. 2022	SEM inspection sample 2.5
09. Dec. 2022	Photolithography samples 2.8,2.9 and 2.10
12. Dec. 2022	Training Ellipsometry
13. Dec. 2022	Resit removal sample 2.3, DekTak XT measurement sample 2.3
14. Dec. 2022	QS135 samples 2.8/2.9/2.10, Training uEtch sample 2.5
15. Dec. 2022	SEM inspection sample 2.5 and 2.7, Training APS using samples 2.8/2.9/2.10
16. Dec. 2022	Resist removal samples 2.8, 2.9 and 2.10, SEM inspection sample 2.6
19. Dec. 2022	IBE of sample 2.8 and 2.9
20. Dec. 2022	SEM inspection sample 2.8
21. Dec. 2022	uEtch of sample 2.8
22. Dec. 2022	SEM inspection of sample 2.8
23. Dec.2022	SEM inspection of sample 2.9

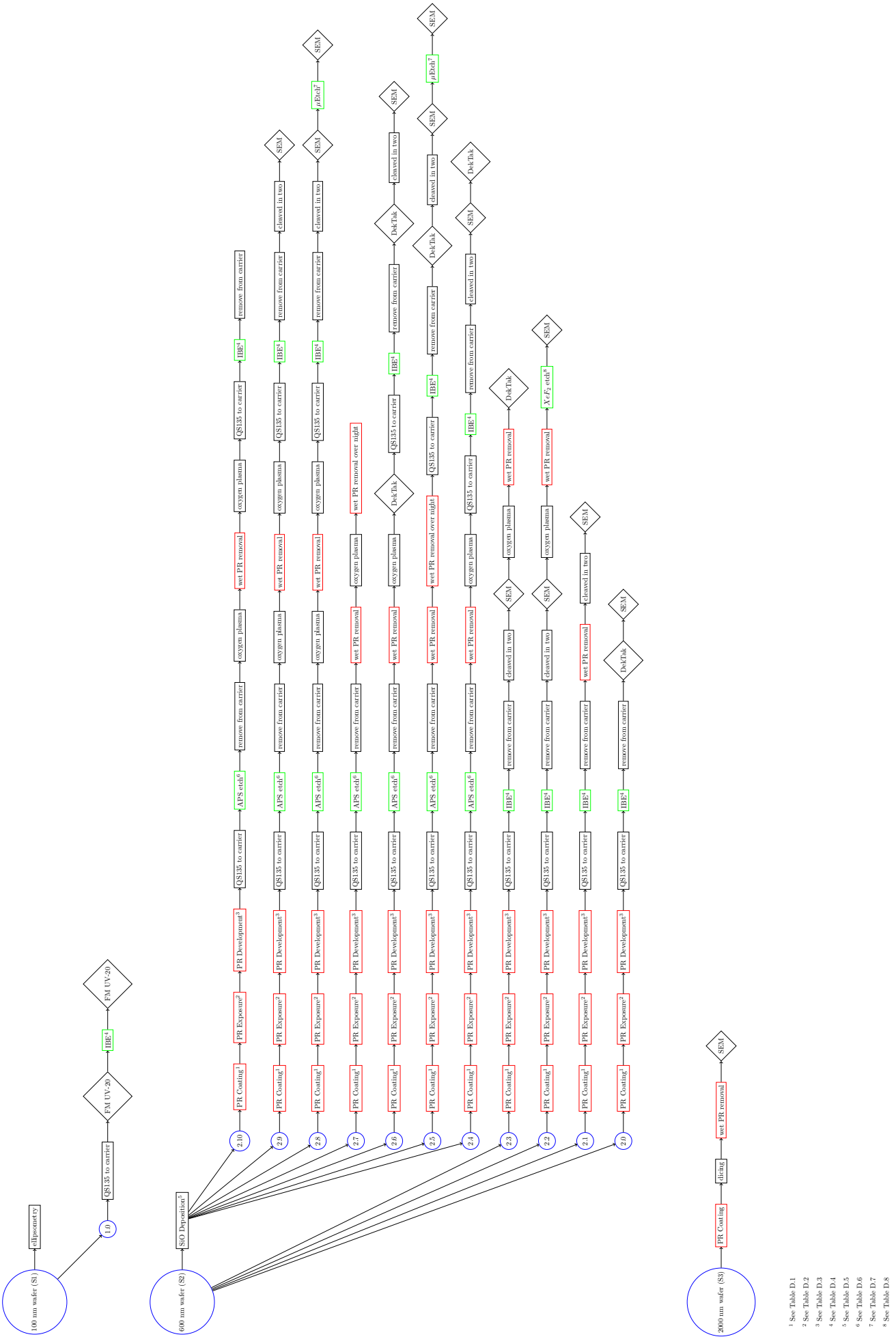
Literature Research Log

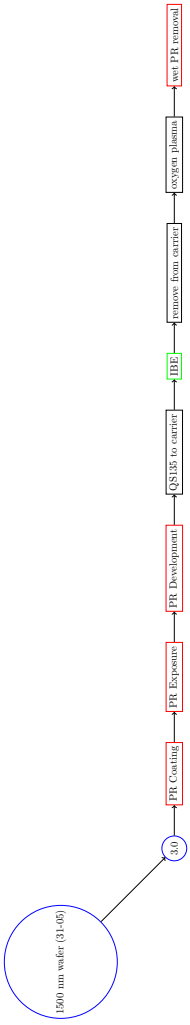
The following table gives a list of papers that were found during the literature research while determining the state of the art that were found to be at least somewhat relevant to the task at hand.

Key	Title	Found using	Keywords	Found on
1	Hafnium carbide based solar absorber coatings with high spectral selectivity	Google Schoolar (Hafnium carbide etching)	Sputtering, Absorbtance	28.12.2022
2	Fabrication of molybdenum carbide and hafnium carbide field emitter arrays	Google Schoolar (Hafnium carbide etching)	XPS, electron beam evaporation	28.12.2022
3	Fabrication of Polycrystalline Silicon Field Emitter Arrays with Hafnium Carbide Coating for Thin-Film-Transistor Controlled Field Emission Displays	Google Schoolar (Hafnium carbide etching)	ICP-Sputtering	28.12.2022
4	Chemical Vapor Deposition of Hafnium Carbide	Google Schoolar (Hafnium carbide "chemical vapor deposition")	CVD	28.12.2022
5	Microfabrication by ion-beam etching	Google Schoolar (etch rates ion beam etching)	IBE	4.1.2023
6	Ion-beam etching	Google Schoolar (etch rates ion beam etching)	IBE	4.1.2023
5	Etch rates for micromachining processing	Google Schoolar (xenon difluoride etching hafnium)	XeF2, vHF	5.1.2023
6	Controlled pulse-etching with xenon difluoride	Google Schoolar (xenon difluoride etching)	XeF2	5.1.2023

Details of Fabrication

The following pages present the exact details of the fabrication of all the samples. The first two pages give a graphical overview of all the steps performed on all the samples. The following pages show tables with all the process parameters used per sample per step.


100 mm wafer (S1)
ellipsometry
1.0
QS135 to carrier
FM UV-20
IBE[4]
FM UV-20
600 mm wafer (S2)
SiO Deposition[5]
2.10
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
PR Coating[1]
PR Exposure[2]
PR Development[3]
QS135 to carrier
APS etch[6]
remove from carrier
oxygen plasma
wet PR removal
wet PR removal over night
QS135 to carrier
IBE[4]
remove from carrier
cleaved in two
SEM
DekTak
μEtch[7]
SEM
2000 nm wafer (S3)
PR Coating
dicing
wet PR removal
SEM
[1] See Table D.1
[2] See Table D.2
[3] See Table D.3
[4] See Table D.4
[5] See Table D.5
[6] See Table D.6
[7] See Table D.7
[8] See Table D.8


1500 nm wafer (31-05)
3.0
PR Coating
PR Exposure
PR Development
QS135 to carrier
IBE
remove from carrier
oxygen plasma
wet PR removal

Sample	2.0	2.1	2.2	2.3	2.4	2.5	2.6	2.7	2.8	2.9	2.10	3.0
Surface preparation	Dehydration	Dehydration	Dehydration	Dehydration	Dehydration	HMDS	HMDS	HMDS	HMDS	HMDS	HMDS	Dehydration
Dehydration time [min]	10	10	10	10	10	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	10
Dehydration temperature [°C]	135	135	135	135	135	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	135
Vapour HMDS recipe	N.A.	N.A.	N.A.	N.A.	N.A.	HMDS Standard	HMDS Standard	HMDS Standard	HMDS Standard	HMDS Standard	HMDS Standard	N.A.
Coater recipe	CHIP_6000	CHIP_6000	CHIP_6000	CHIP_6000	CHIP_6000	CHIP_6000	CHIP_6000	CHIP_6000	CHIP_6000	CHIP_6000	CHIP_6000	CHIP_6000
Softbake temperature [°C]	90	90	90	90	90	90	90	90	90	90	90	90
Softbake time [sec]	90	90	90	90	90	100	100	100	90	90	90	90

Table D.1: Details surface preparation and PR Coating per sample

Sample	2.0	2.1	2.2	2.3	2.4	2.5	2.6	2.7	2.8	2.9	2.10	3.0
Dose [mJ/cm^2]	215	215	215	215	215	215	215	215	215	215	215	215
Defocus	-1	-1	-1	-1	-1	-1	-1	-1	-1	-1	-1	-1
Layout	1 device	lines	advanced lines	advanced lines	advanced lines	advanced lines and cantilevers	advanced lines and cantilevers	advanced lines and cantilevers	advanced lines and cantilevers	advanced lines and cantilevers	advanced lines and cantilevers	advanced lines

Table D.2: Details of the PR Exposure per sample

Sample	2.0	2.1	2.2	2.3	2.4	2.5	2.6	2.7	2.8	2.9	2.10	3.0
Post Exposure Bake (PEB) temperature [°C]	100	100	100	100	100	100	100	100	100	100	100	100
PEB time [sec]	90	90	90	100	90	90	90	90	90	90	90	90
Develoment time [sec]	90	80	80	80	60	60	60	60	60	60	60	60
Rinse time [sec]	60	60	60	60	60	60	60	60	60	60	60	60
PDB Temperature [°C]	N.A.	N.A.	N.A.	N.A.	N.A.	100	100	100	100	100	100	N.A.
PDB Time [min]	N.A.	N.A.	N.A.	N.A.	N.A.	5	5	5	5	5	5	N.A.

Table D.3: Details of the PR Development per sample

Sample	1.0	2.0	2.1	2.2	2.3	2.4	2.5	2.6	2.8	2.9	3.0
Power	Med	Med	Med	Med	Med	Med	Med	Med	Med	Med	Med
Angle [°]	-10	-10	-10	-10	-10 (15'), -40 (7.5'), -70 (7.5')	-10	-10	-5	-10	-10 (16'), -40 (8.5'), -70 (8.5')	-10
Total etch time [min]	1	25	25	30	30	23	23	23	24	33	50

Table D.4: Details of the IBE per sample

Recipe	SiO2-F
Deposition time	95 min

Table D.5: Parameters used in the deposition of the SiO layer used as a hardmask.

Sample	2.4	2.5	2.6	2.7	2.8	2.9	2.10
Recipe	Sio2_PR_3:1	Sio2_PR_3:1	Sio2_PR_3:1	Sio2_PR_3:1	Sio2_PR_1:1	Sio2_PR_1:1	Sio2_PR_1:1
Time [min]	9	9	9	9	10	10	10

Table D.6: Parameters used for etching the SiO hardmask

Sample	2.5	2.8
Recipe	3	3
Etch time [sec]	300	900
Number of cycles	1	1
Needle valve position	12	12

Table D.7: Settings used for the vapour HF etching

Sample	2.2
Expansion chamber pressure	1 Torr
Number of cycles	14

Table D.8: Parameters used for the XeF2 etching

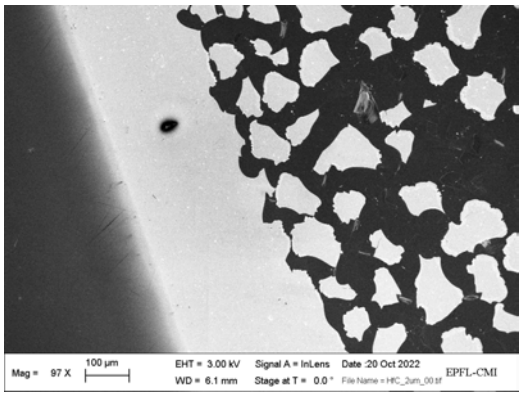
Samples Provided

The following table shows the materials received from [redacted]

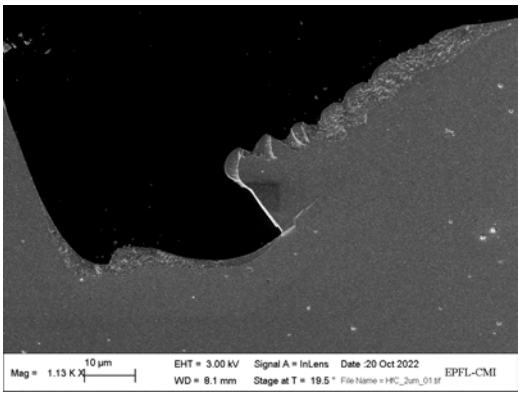
Sample #	Sample name	Film Thickness Measurement as provided by [redacted]	Used during this project
S1	100 nm wafer	109 nm	Yes
S2	600 nm wafer	553 nm	Yes
S3	2000 nm wafer	2300 nm	Yes
31-05	1500 nm wafer	1500 nm	Yes
03-06	03-06	1500 nm	No
04-06	04-06	1500 nm	No
07-06	07-06	1500 nm	No
08-06	08-06	1500 nm	No
09-06	09-06	1500 nm	No

SEMImages

F.1 Sample 0.0



(a) $2\mu m$ wafer piece after dicing

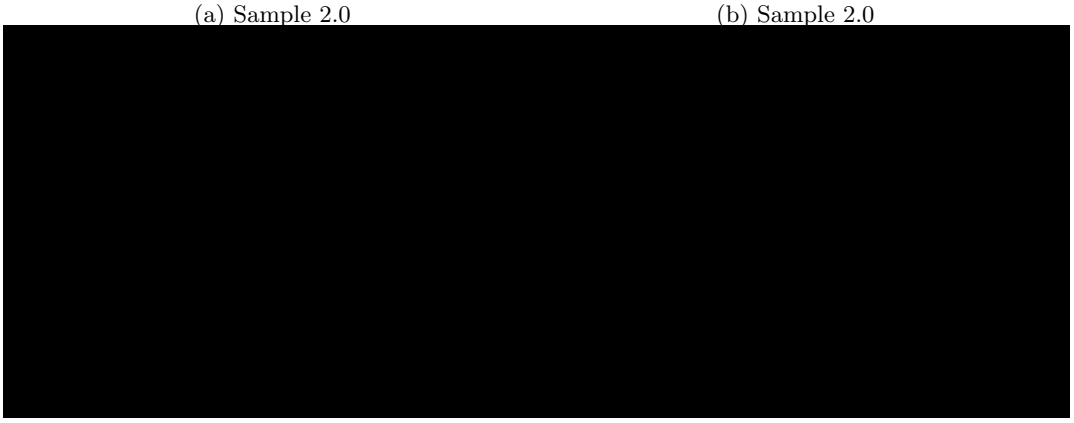


(b) $2\mu m$ wafer piece after dicing

F.2 Sample 2.0

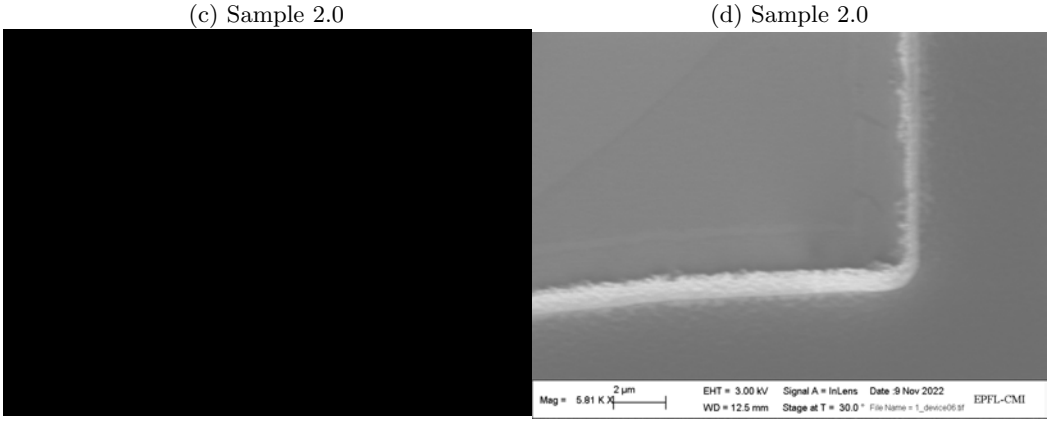
(a) Sample 2.0

(b) Sample 2.0



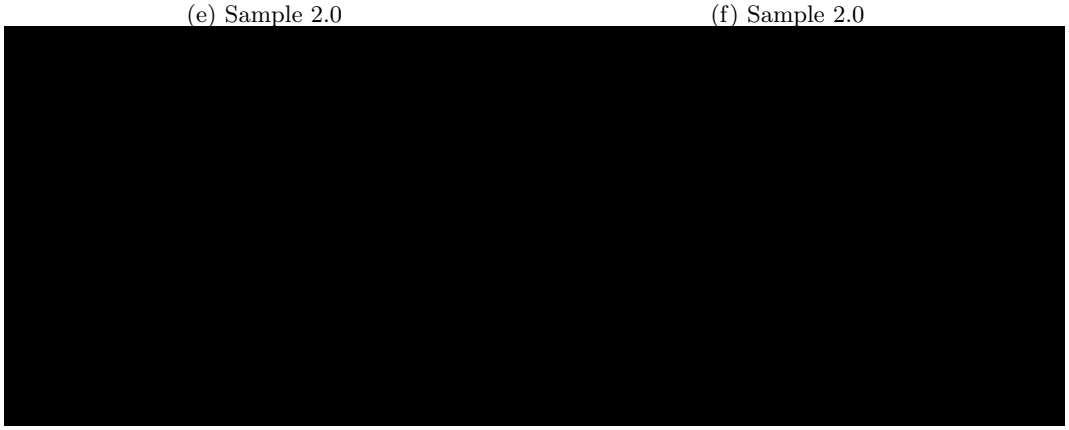
(c) Sample 2.0

(d) Sample 2.0



(e) Sample 2.0

(f) Sample 2.0

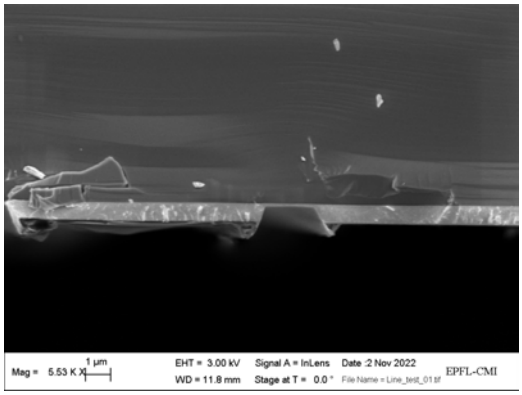


(g) Sample 2.0

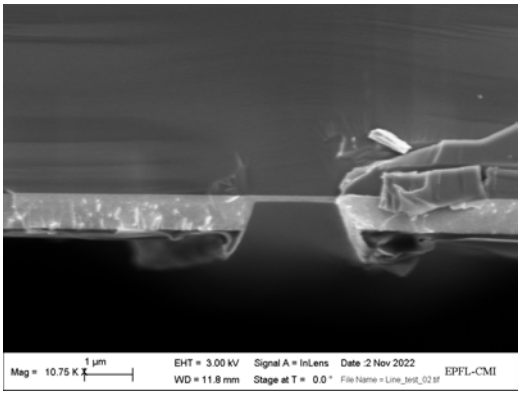
(h) Sample 2.0

(a) Sample 2.0 (b) Sample 2.0

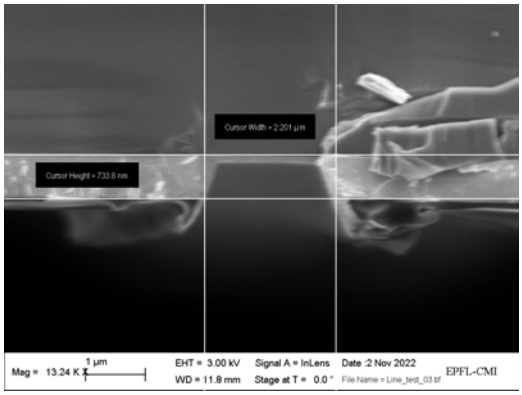
F.3 Sample 2.1



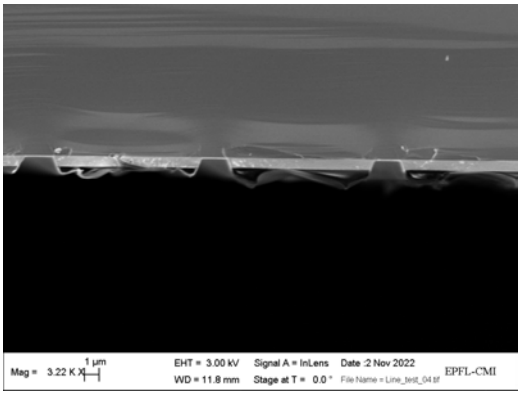
(a) Sample 2.1



(b) Sample 2.1



(c) Sample 2.1



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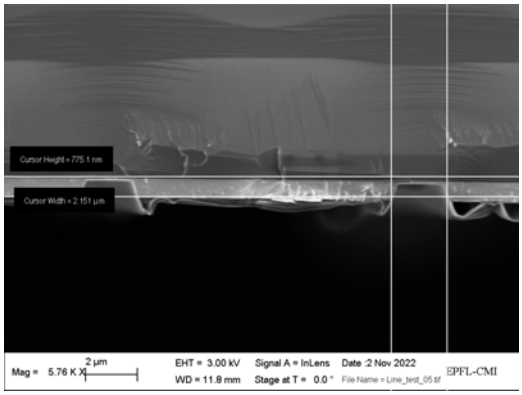

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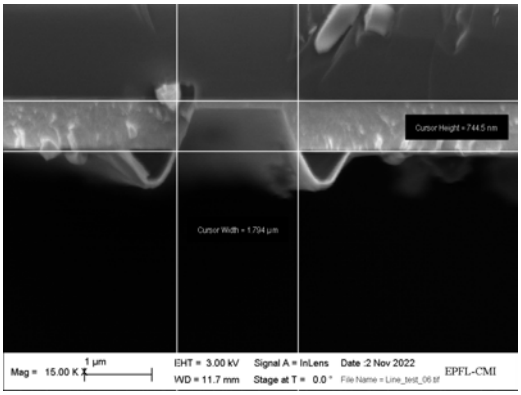

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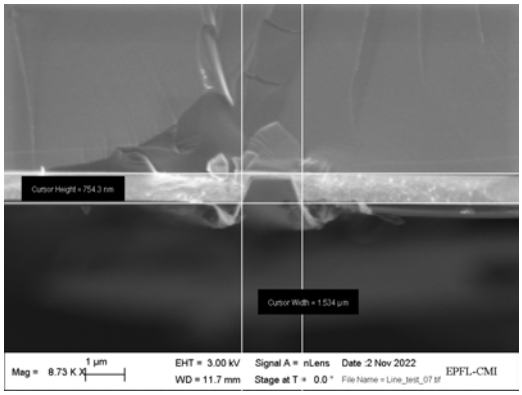
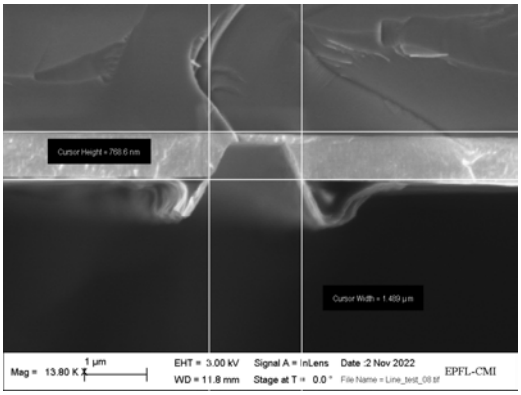
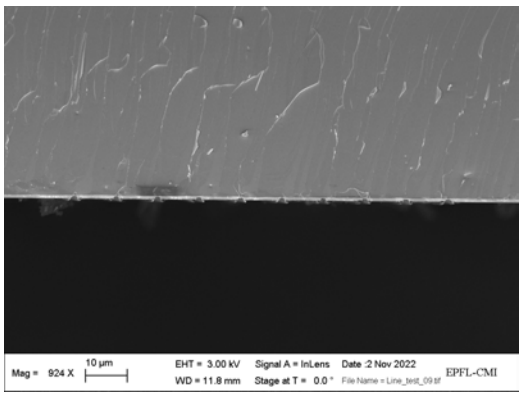


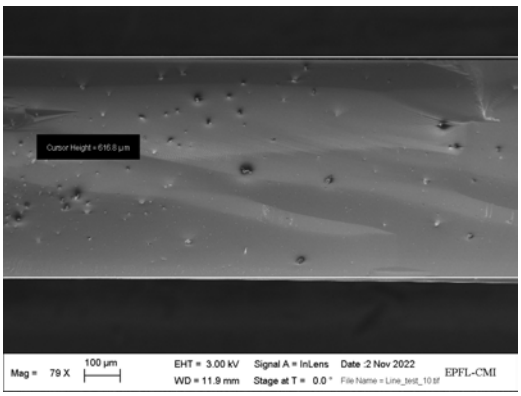
(g) Sample 2.1



(h) Sample 2.1



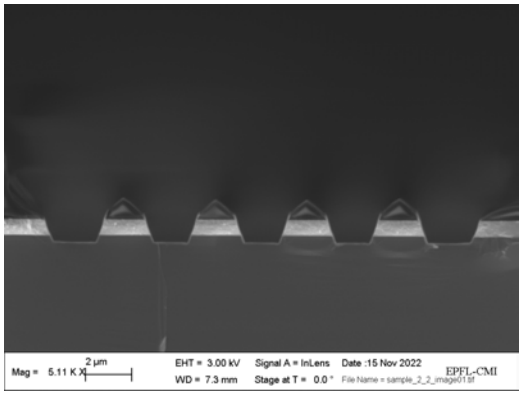
(a) Sample 2.1



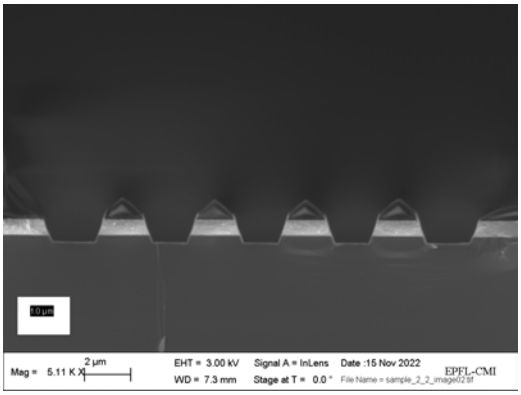
(b) Sample 2.1

F.4 Sample 2.2

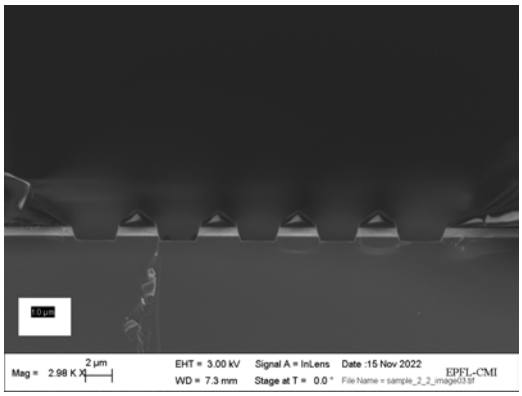
F.4.1 After IBE



(a) Sample 2.2



(b) Sample 2.2



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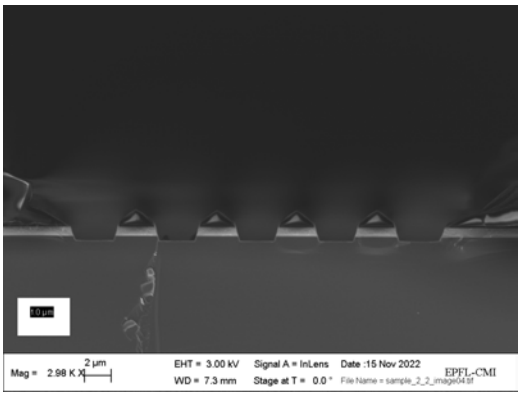

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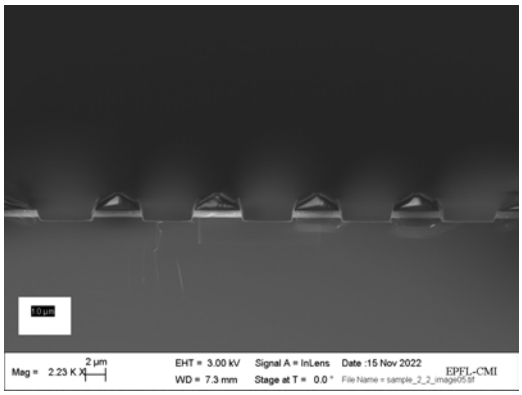
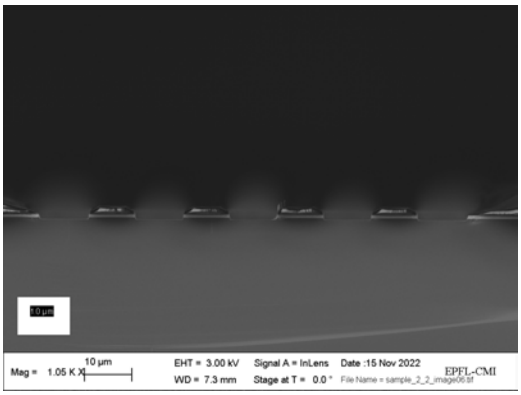


(e) Sample 2.2



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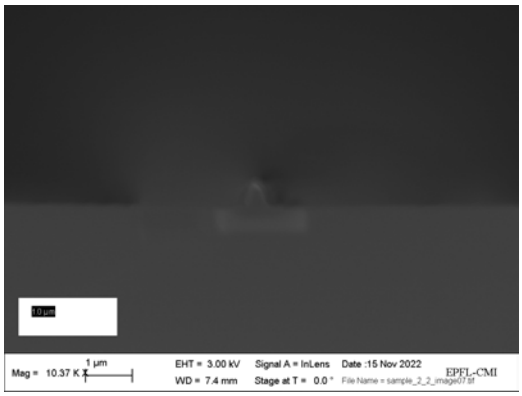
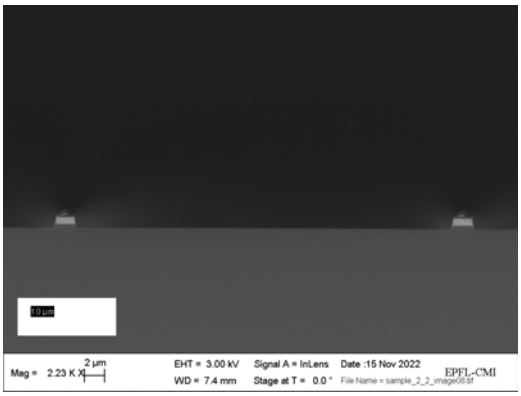
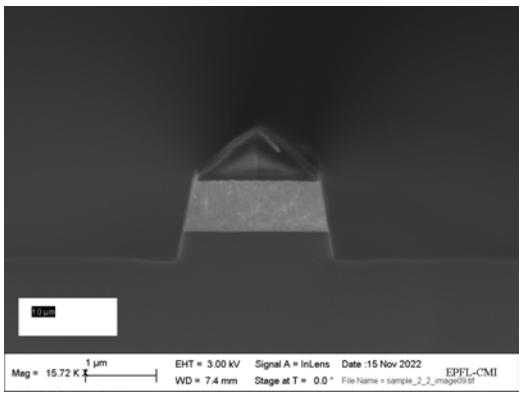


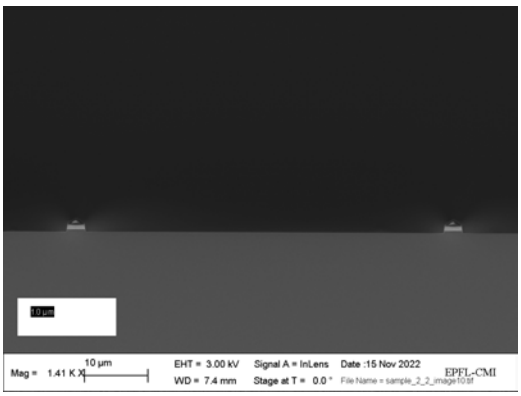
(g) Sample 2.2



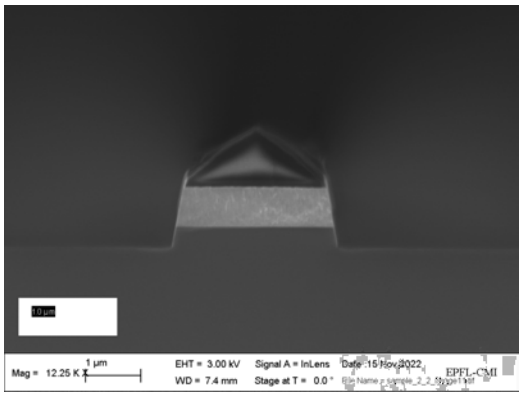
(h) Sample 2.2



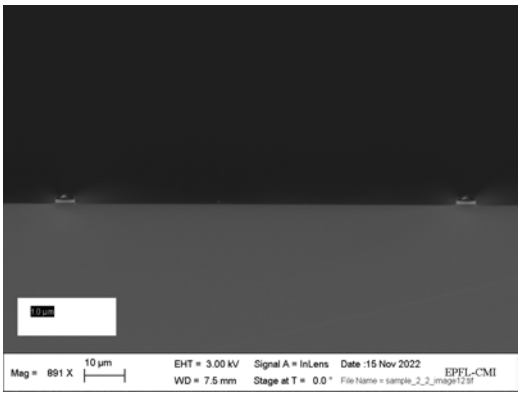
(a) Sample 2.2



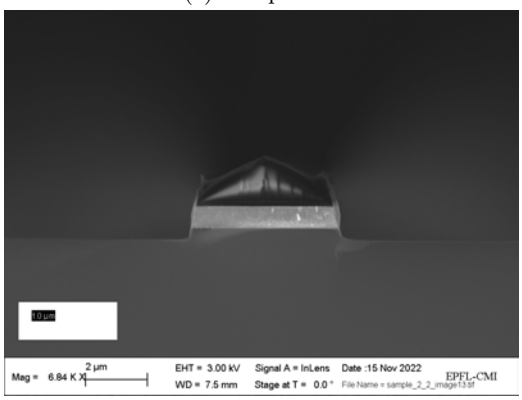
(b) Sample 2.2



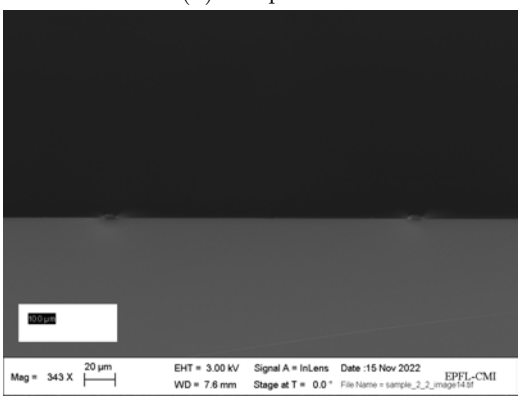
(c) Sample 2.2



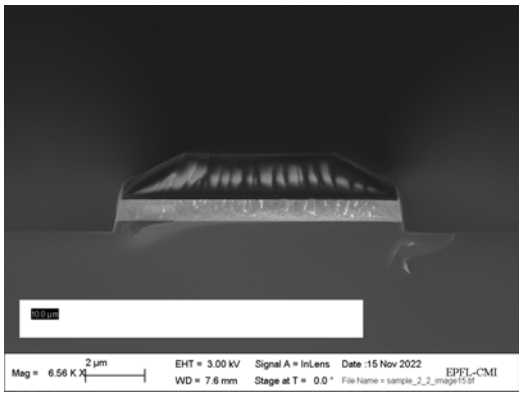
(d) Sample 2.2



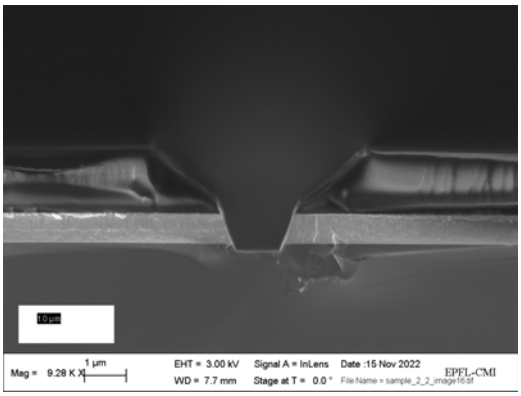
(e) Sample 2.2



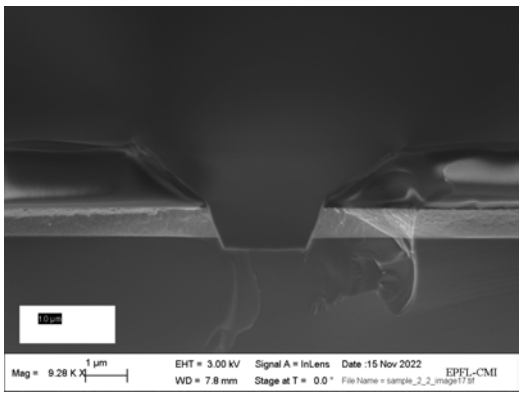
(f) Sample 2.2



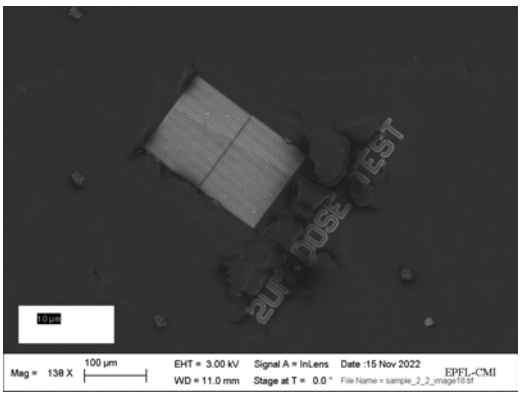
(g) Sample 2.2



(h) Sample 2.2



(a) Sample 2.2

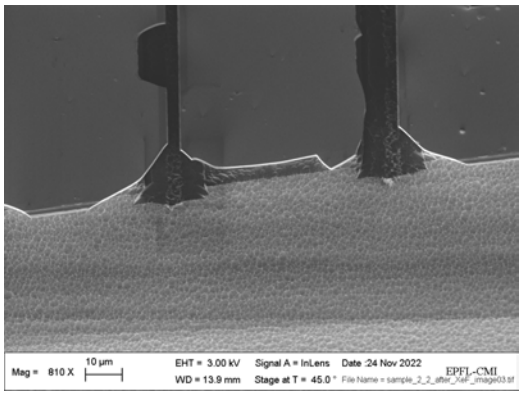


(b) Sample 2.2

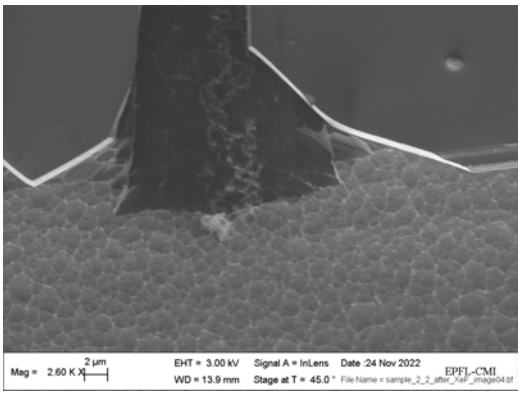
F.4.2 After XeF2 Etching

(a) Sample 2.2

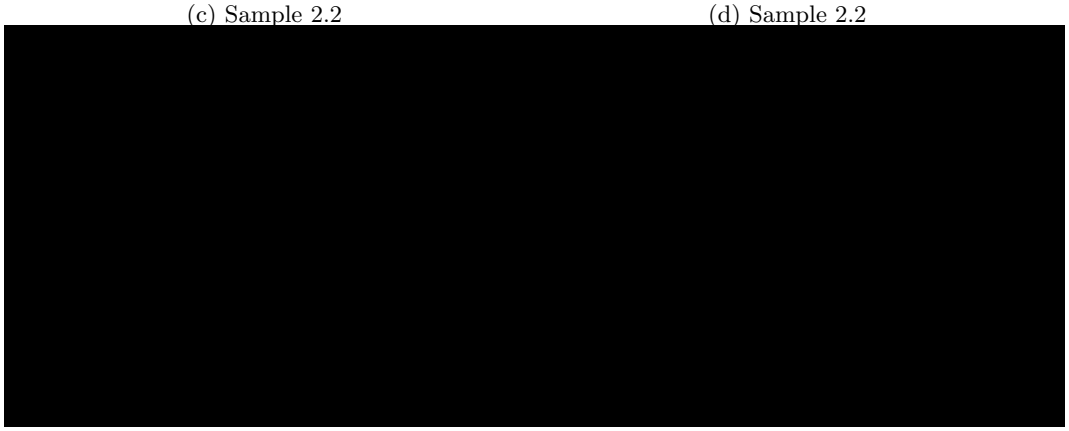
(b) Sample 2.2



(c) Sample 2.2



(d) Sample 2.2

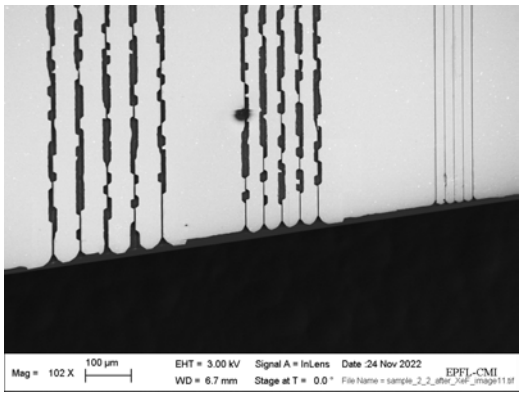


(e) Sample 2.2

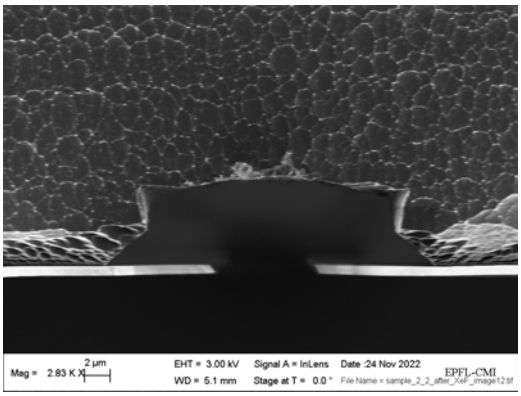
(f) Sample 2.2

(a) Sample 2.2

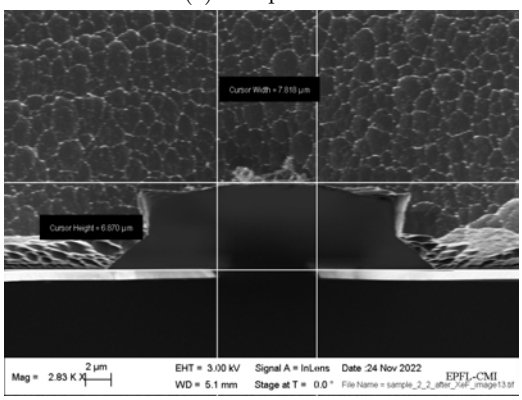
(b) Sample 2.2



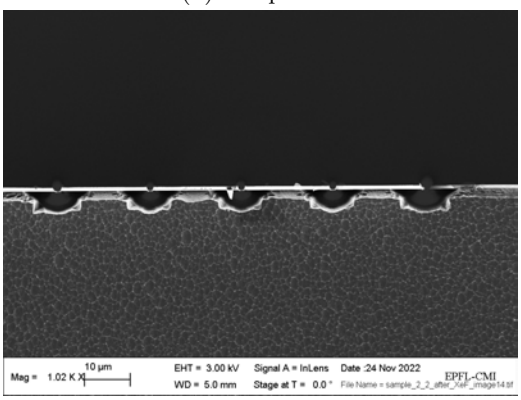
(c) Sample 2.2



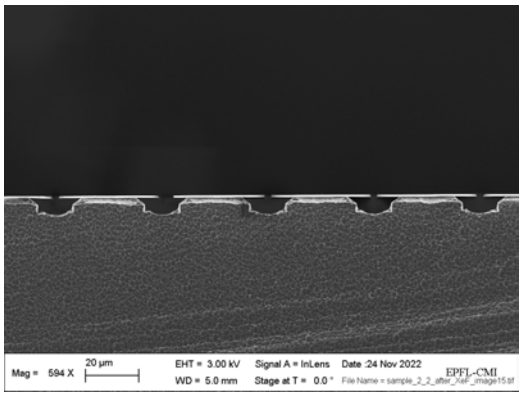
(d) Sample 2.2



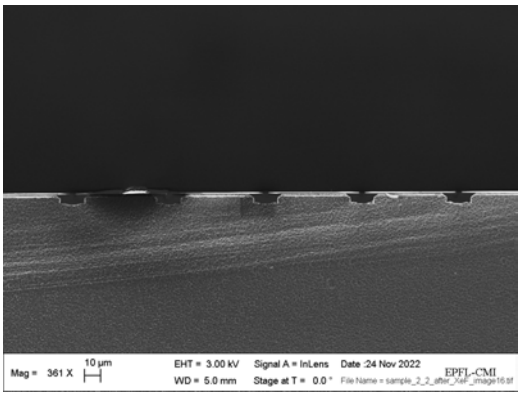
(e) Sample 2.2



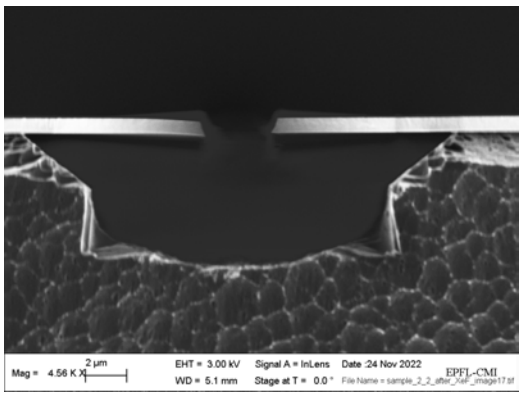
(f) Sample 2.2



(g) Sample 2.2

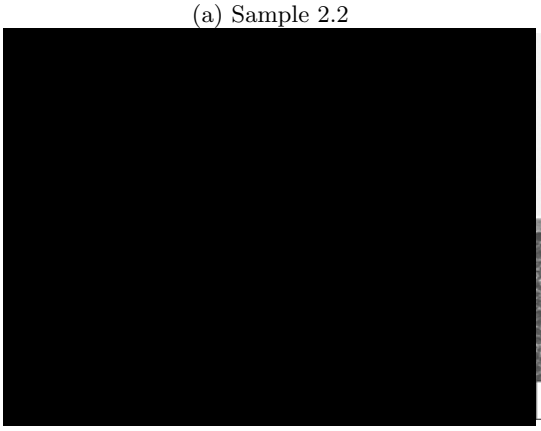


(h) Sample 2.2

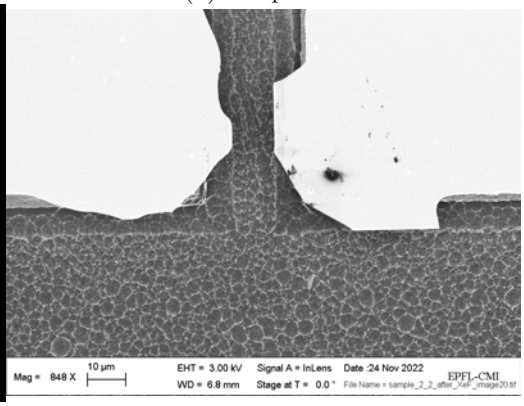


(a) Sample 2.2

(b) Sample 2.2

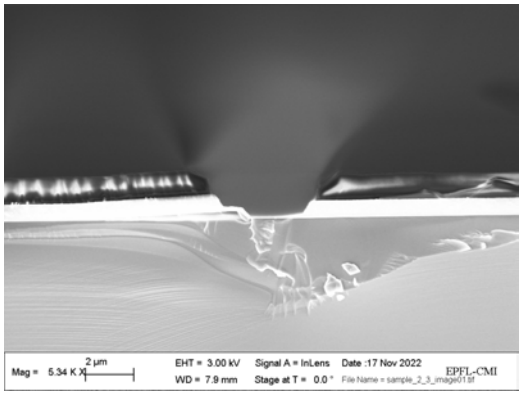


(c) Sample 2.2

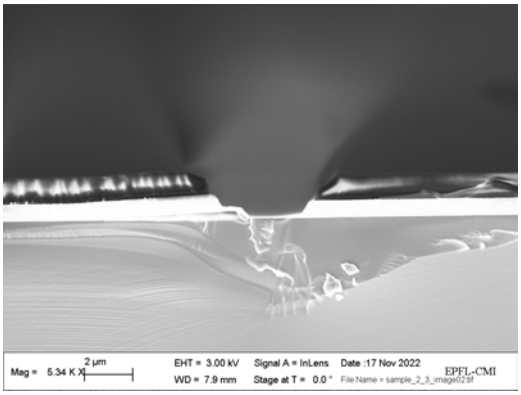


(d) Sample 2.2

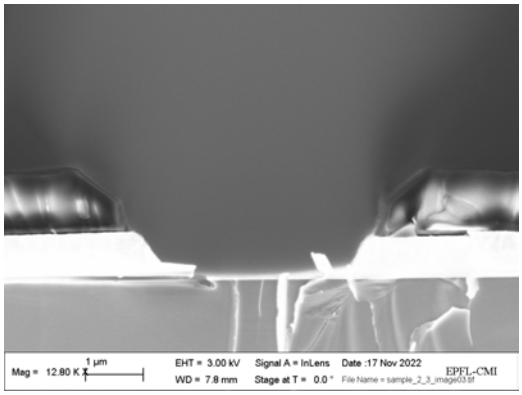
F.5 Sample 2.3



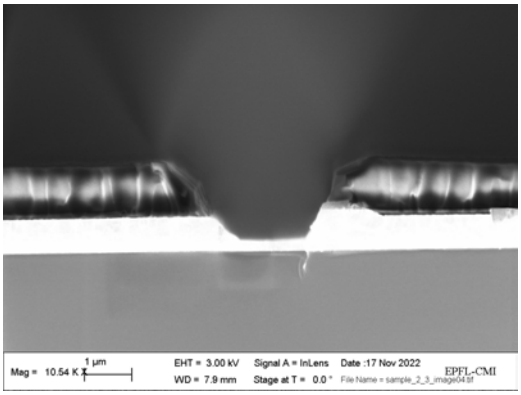
(a) Sample 2.3



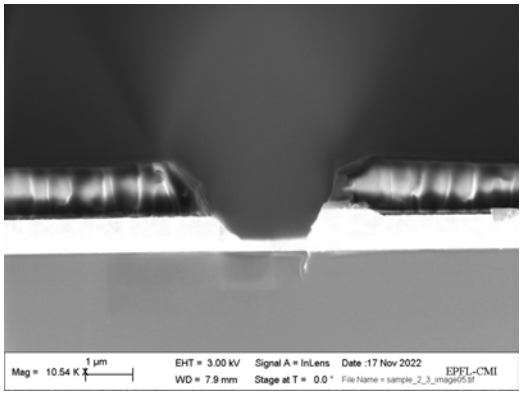
(b) Sample 2.3



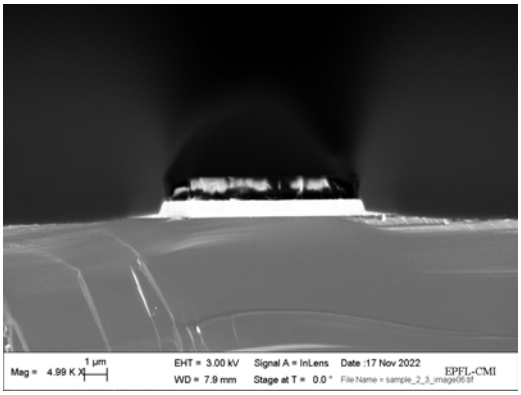
(c) Sample 2.3



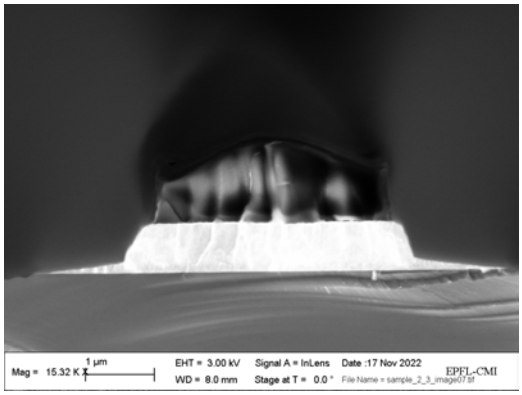
(d) Sample 2.3



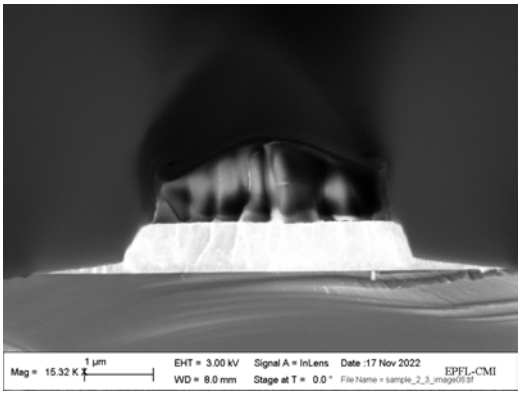
(e) Sample 2.3



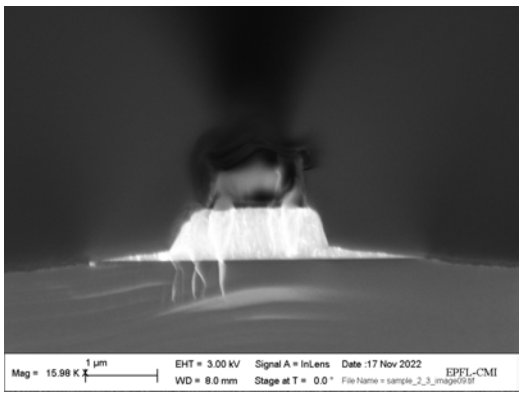
(f) Sample 2.3



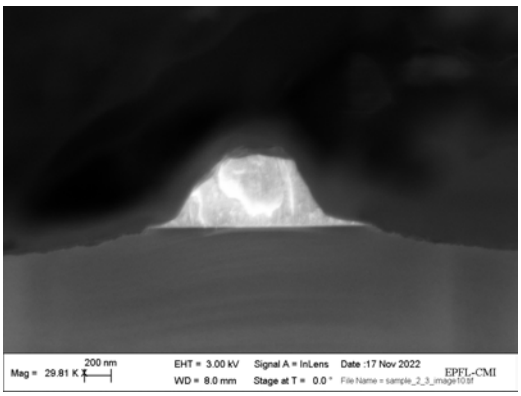
(g) Sample 2.3



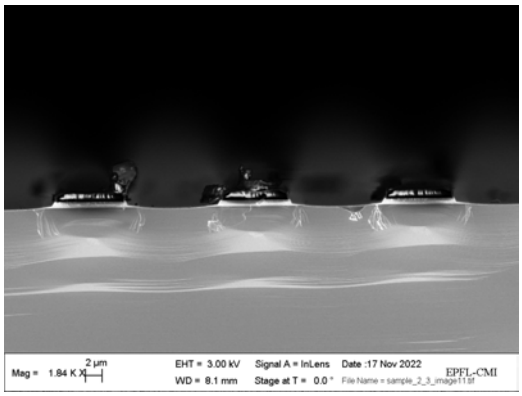
(h) Sample 2.3



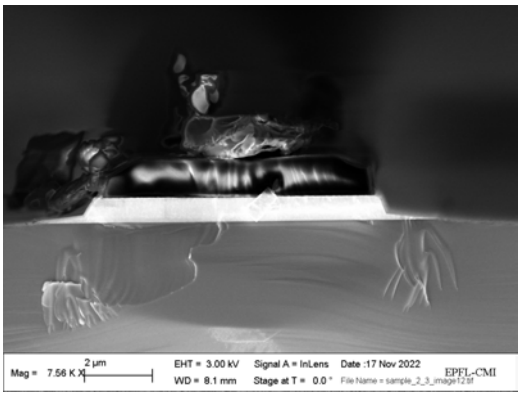
(a) Sample 2.3



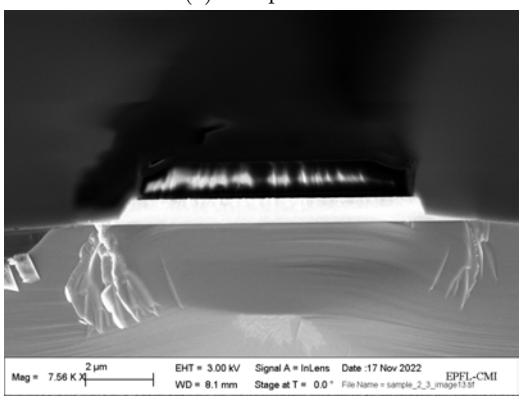
(b) Sample 2.3



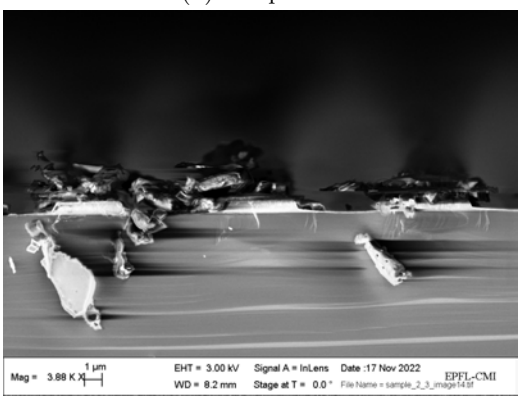
(c) Sample 2.3



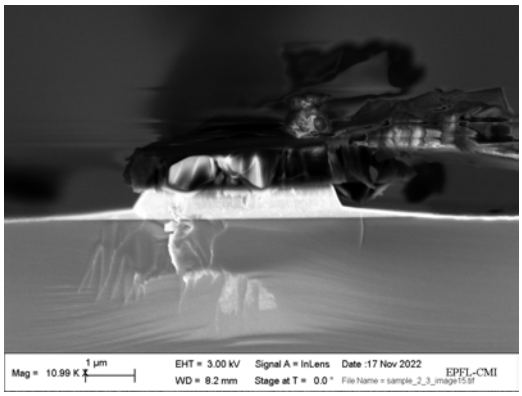
(d) Sample 2.3



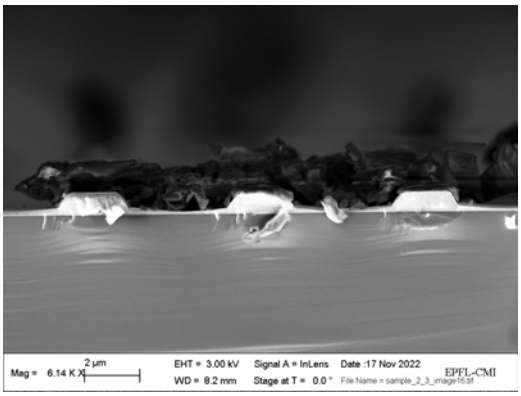
(e) Sample 2.3



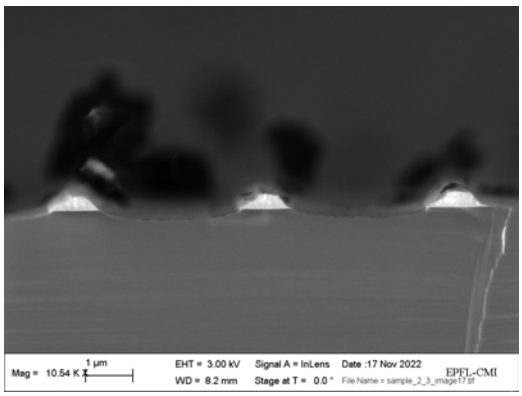
(f) Sample 2.3



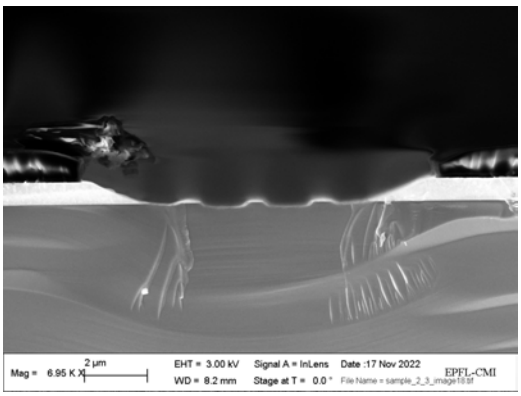
(g) Sample 2.3



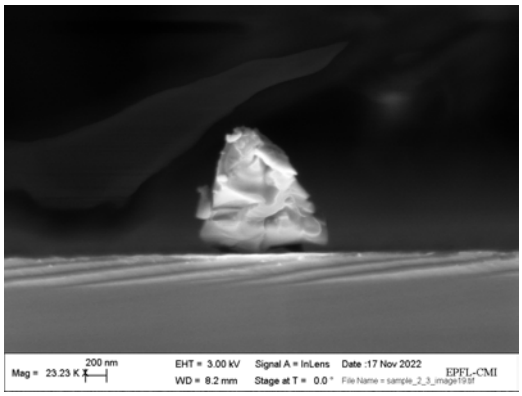
(h) Sample 2.3



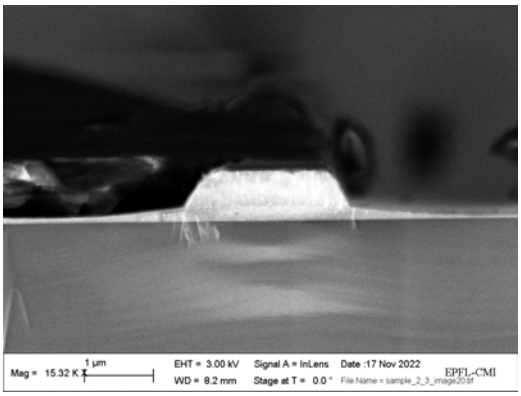
(a) Sample 2.3



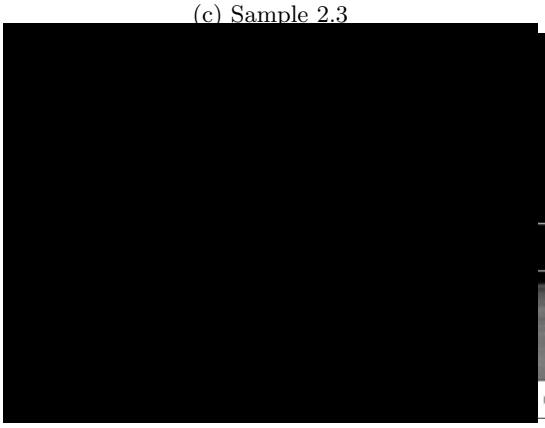
(b) Sample 2.3



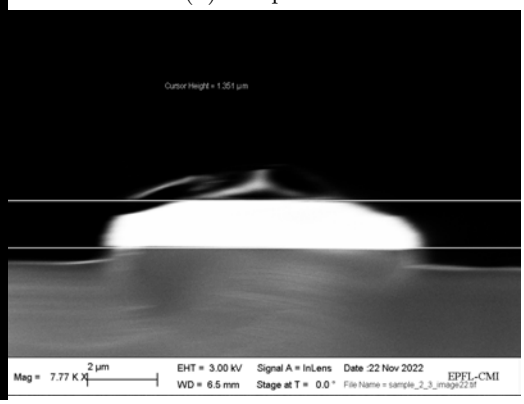
(c) Sample 2.3



(d) Sample 2.3

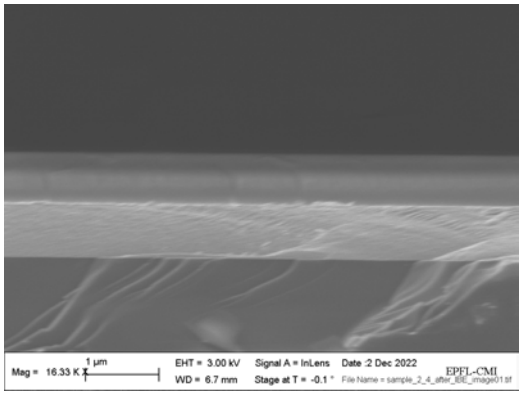


(e) Sample 2.3

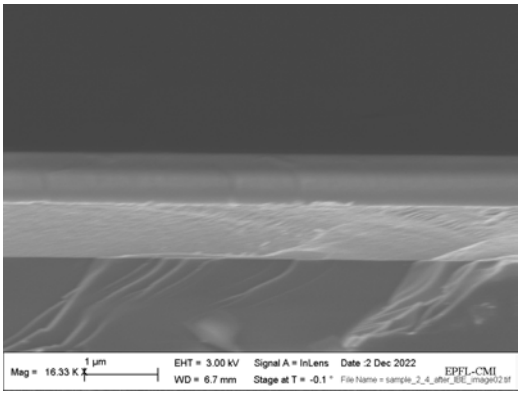


(f) Sample 2.3

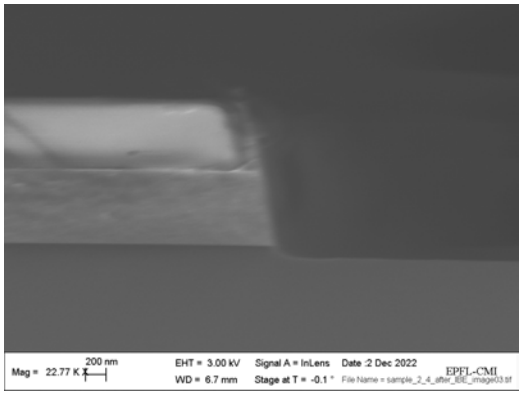
F.6 Sample 2.4



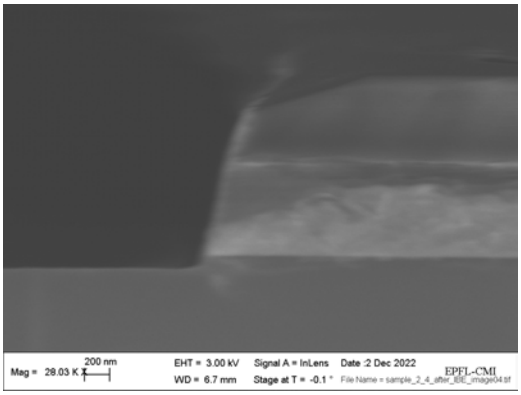
(a) Sample 2.4



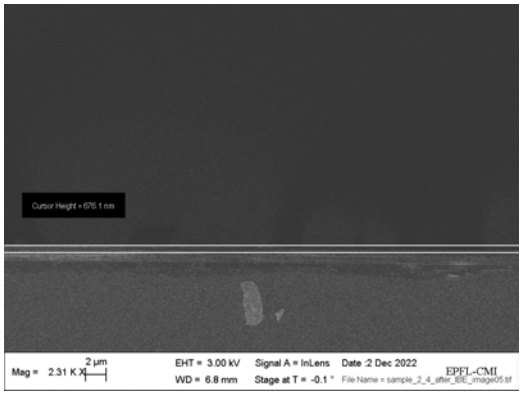
(b) Sample 2.4



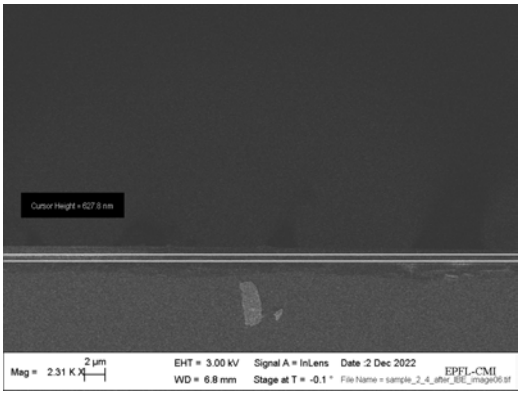
(c) Sample 2.4



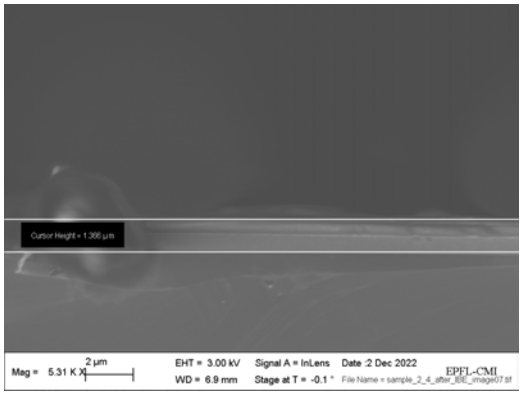
(d) Sample 2.4



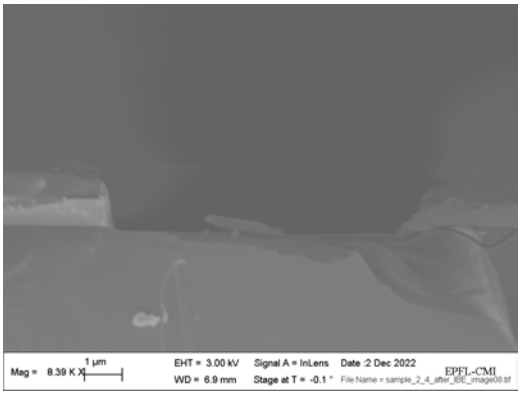
(e) Sample 2.4



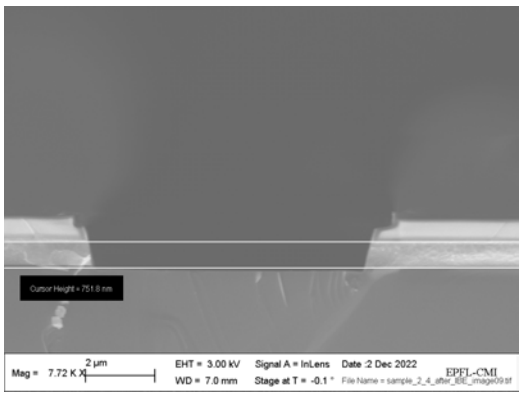
(f) Sample 2.4



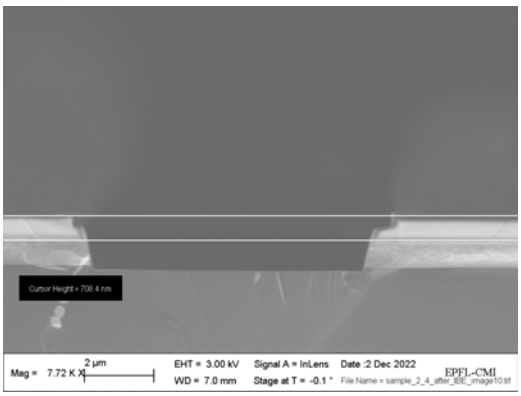
(g) Sample 2.4



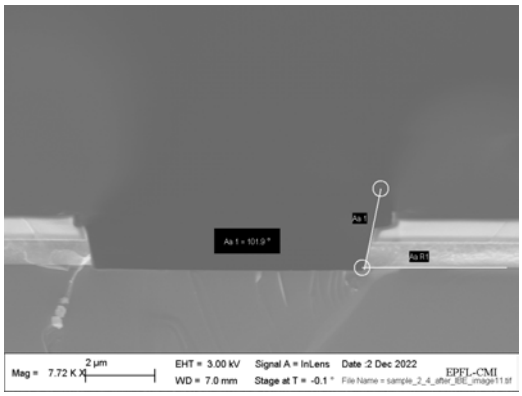
(h) Sample 2.4



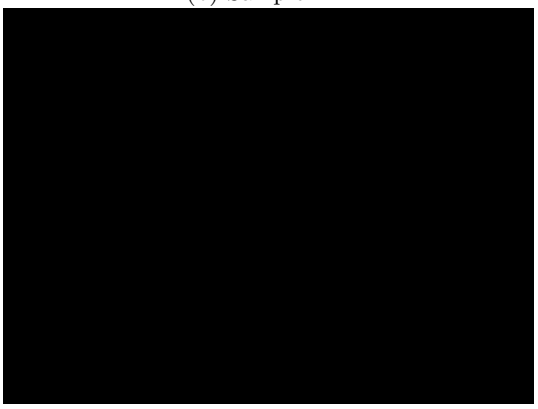
(a) Sample 2.4



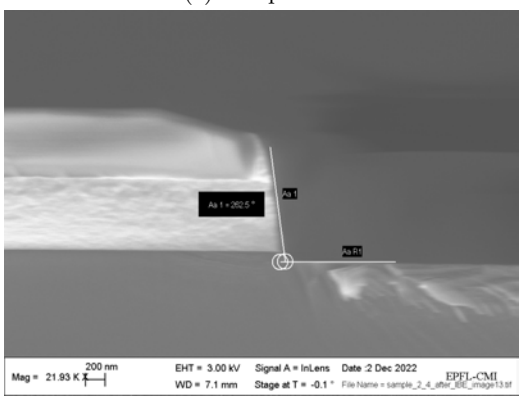
(b) Sample 2.4



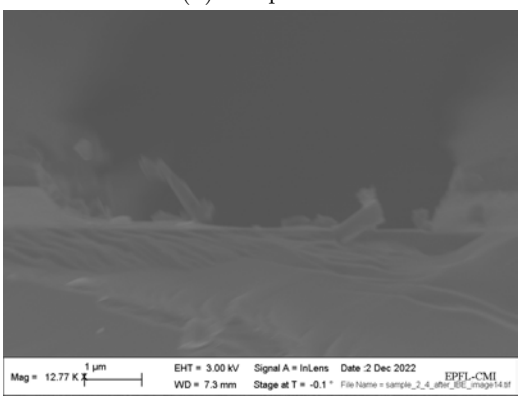
(c) Sample 2.4



(d) Sample 2.4



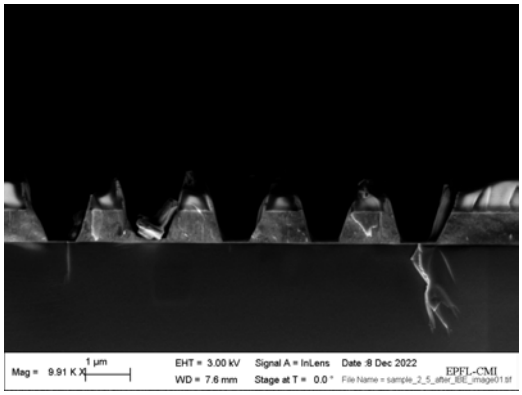
(e) Sample 2.4



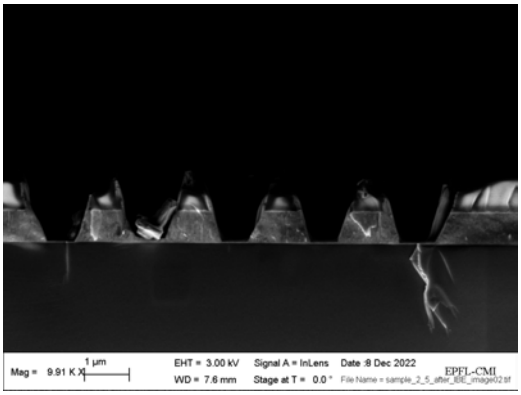
(f) Sample 2.4

F.7 Sample 2.5

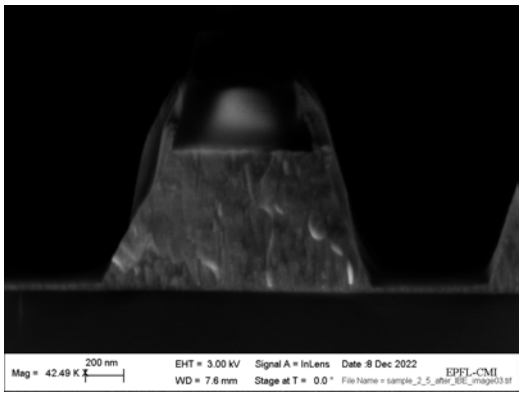
F.7.1 After IBE



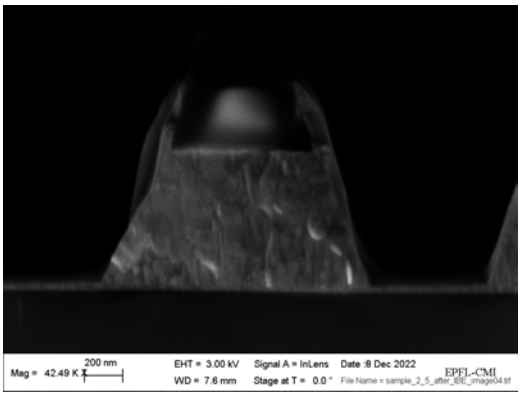
(a) Sample 2.5



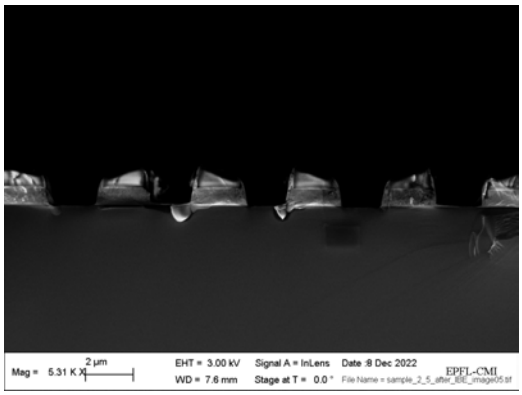
(b) Sample 2.5



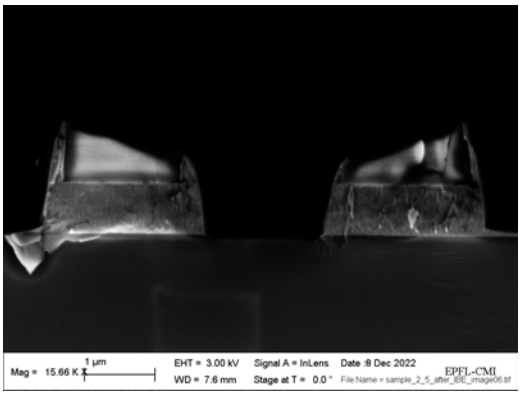
(c) Sample 2.5



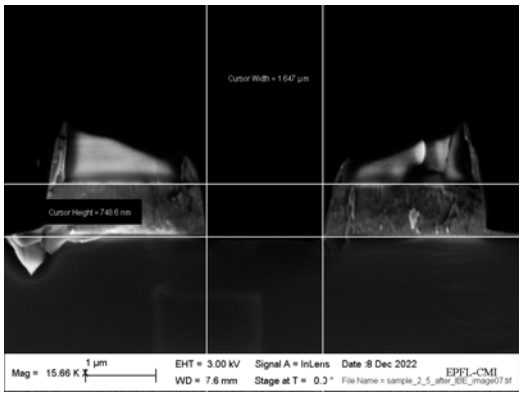
(d) Sample 2.5



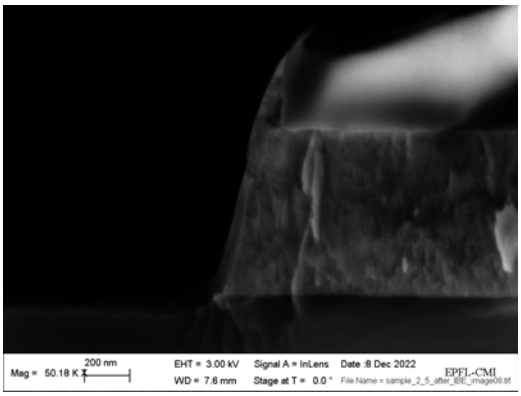
(e) Sample 2.5



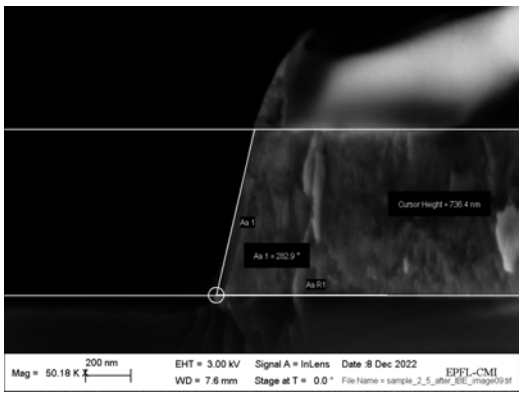
(f) Sample 2.5



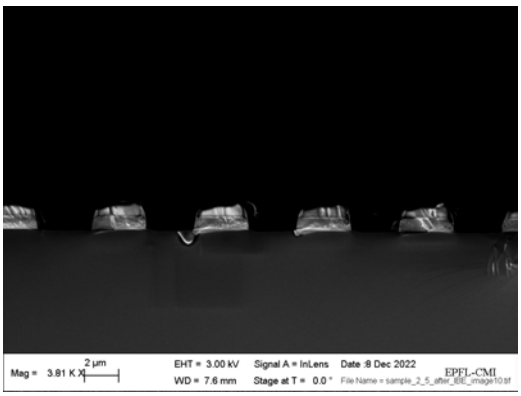
(g) Sample 2.5



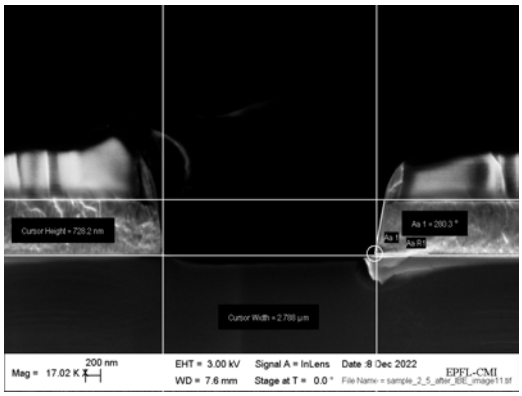
(h) Sample 2.5



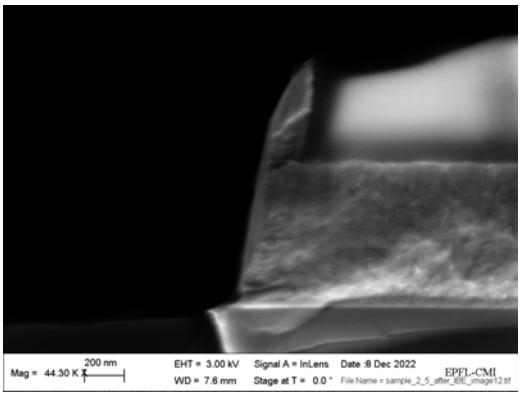
(a) Sample 2.5



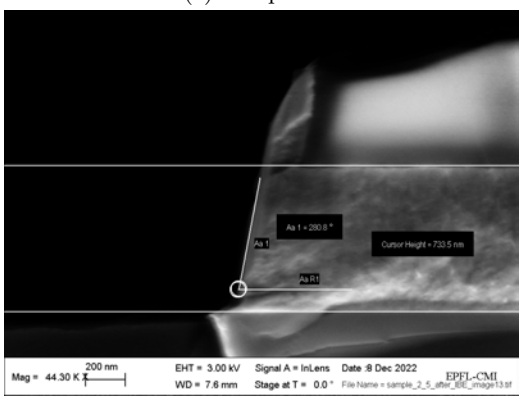
(b) Sample 2.5



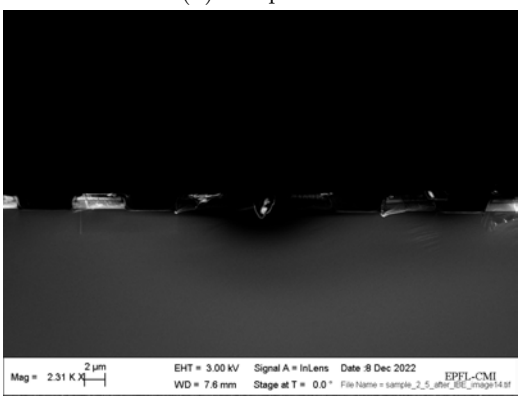
(c) Sample 2.5



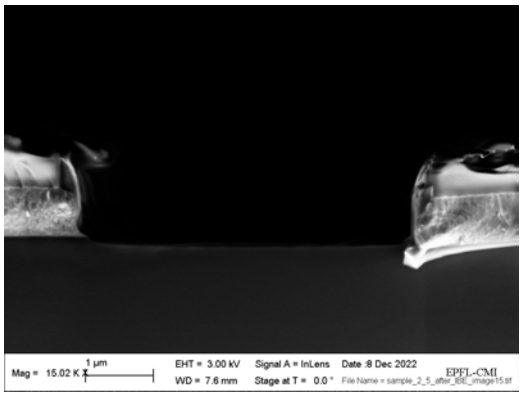
(d) Sample 2.5



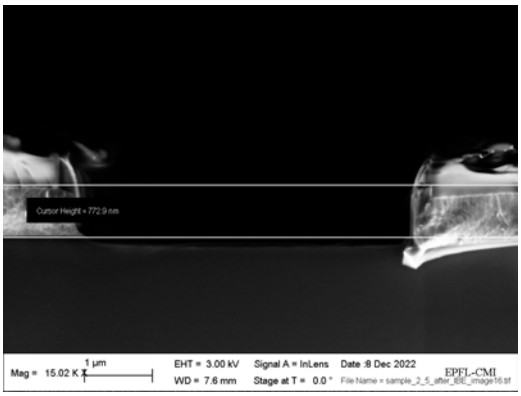
(e) Sample 2.5



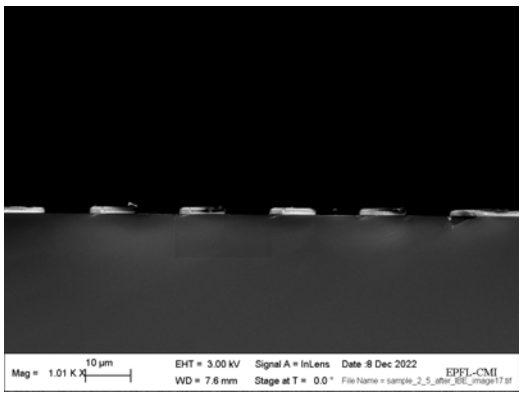
(f) Sample 2.5



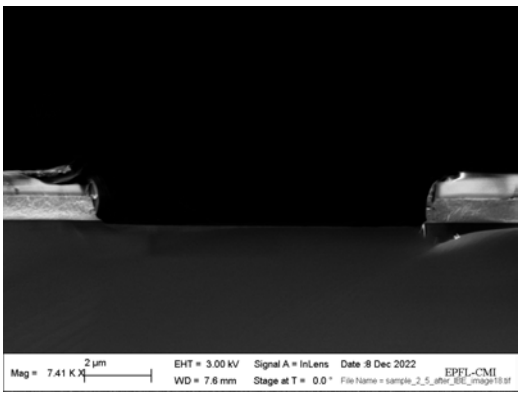
(g) Sample 2.5



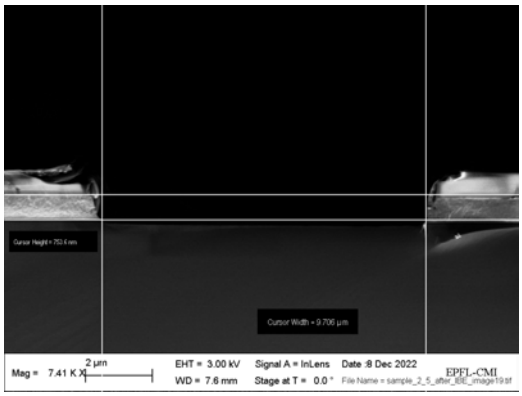
(h) Sample 2.5



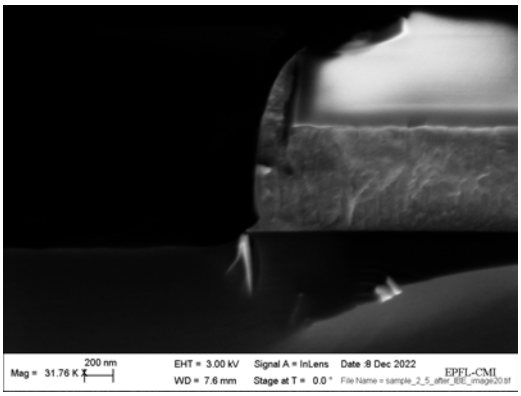
(a) Sample 2.5



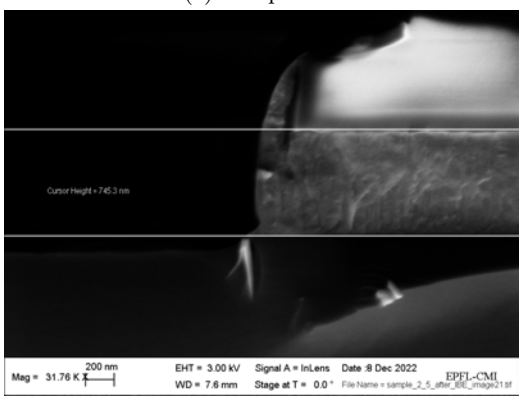
(b) Sample 2.5



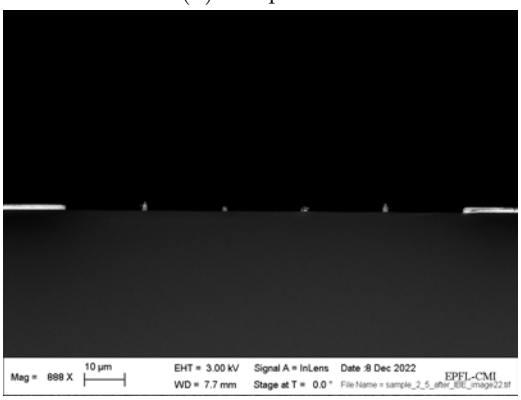
(c) Sample 2.5



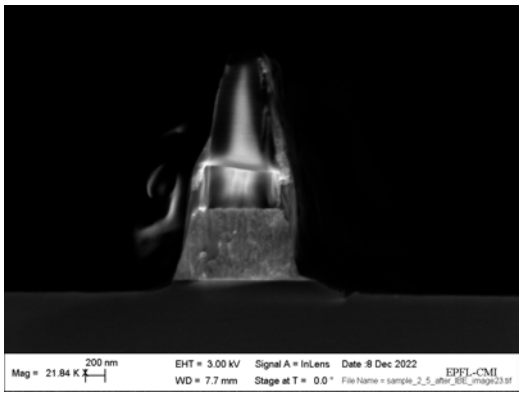
(d) Sample 2.5



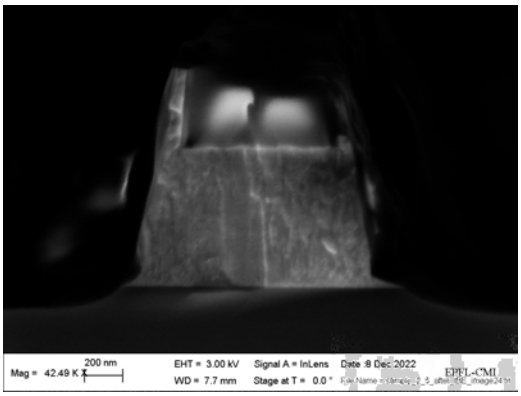
(e) Sample 2.5



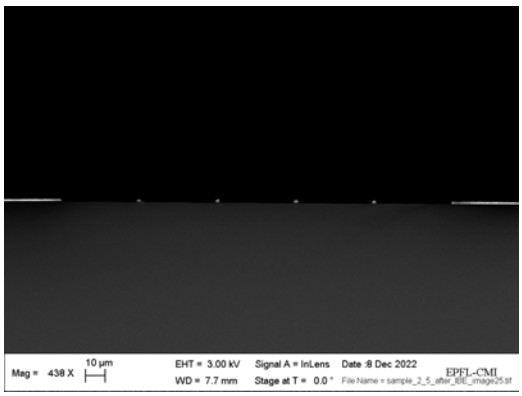
(f) Sample 2.5



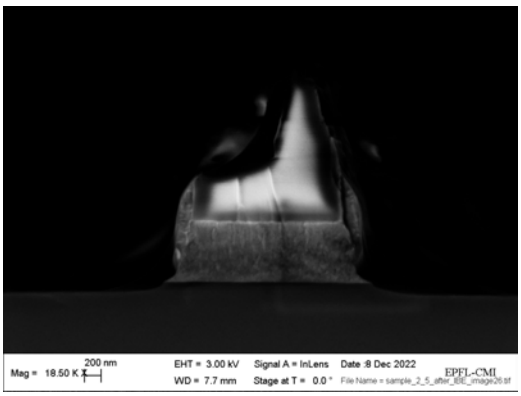
(g) Sample 2.5



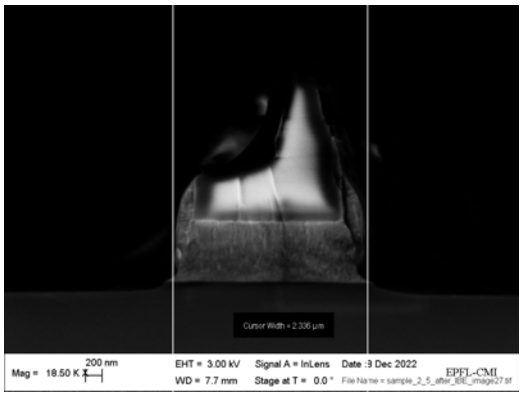
(h) Sample 2.5



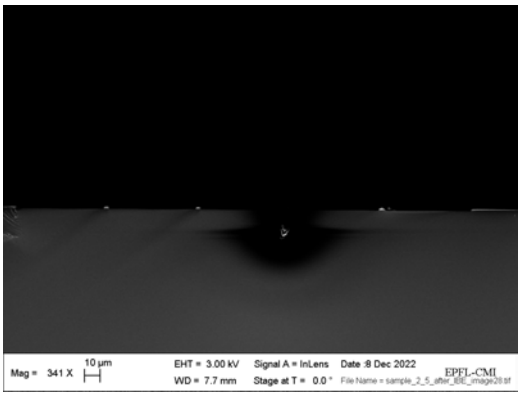
(a) Sample 2.5



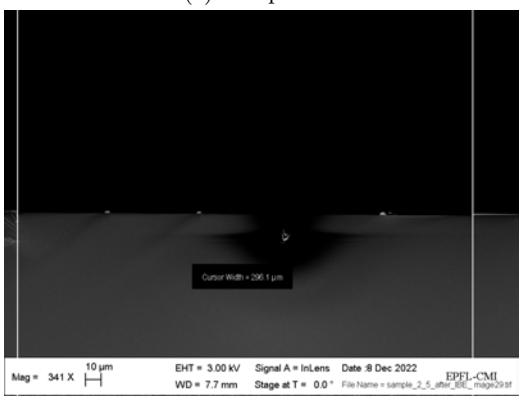
(b) Sample 2.5



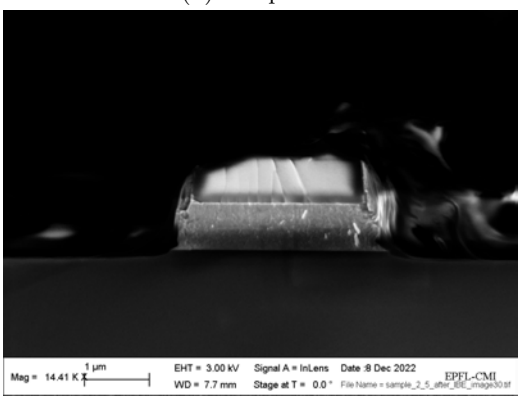
(c) Sample 2.5



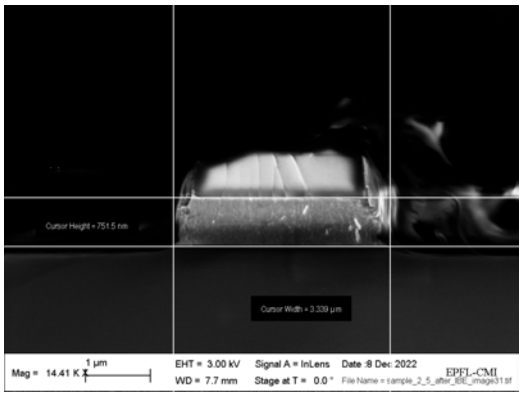
(d) Sample 2.5



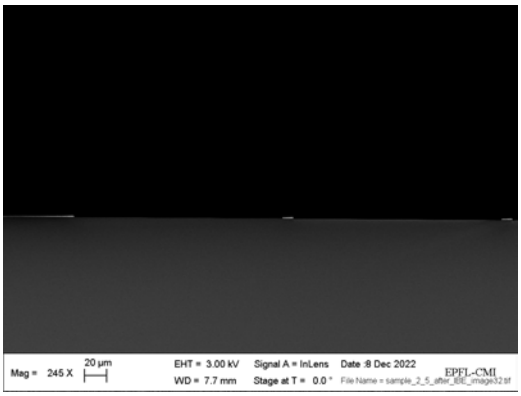
(e) Sample 2.5



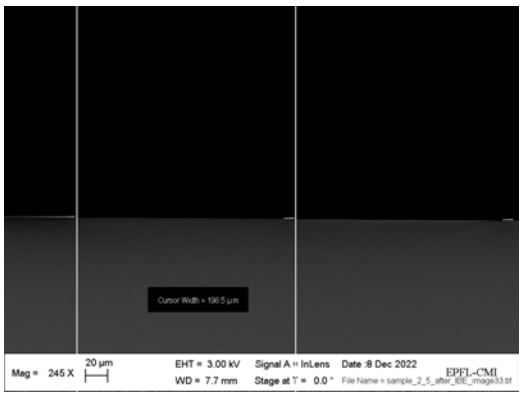
(f) Sample 2.5



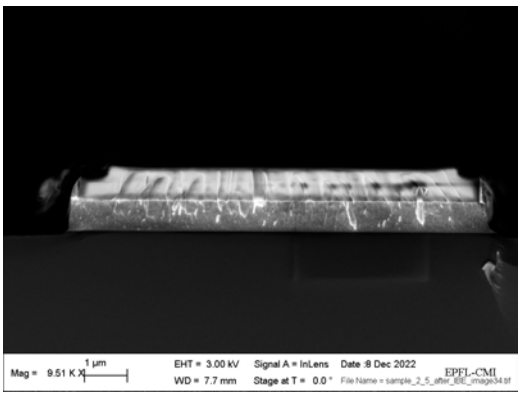
(g) Sample 2.5



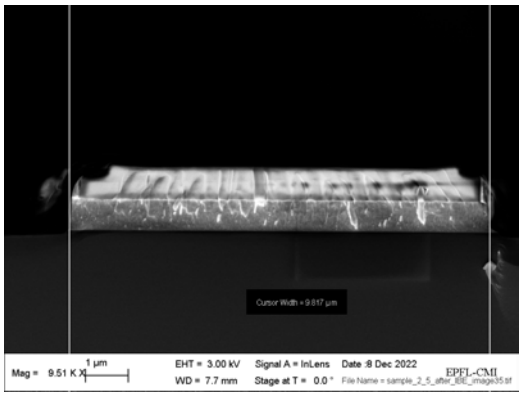
(h) Sample 2.5



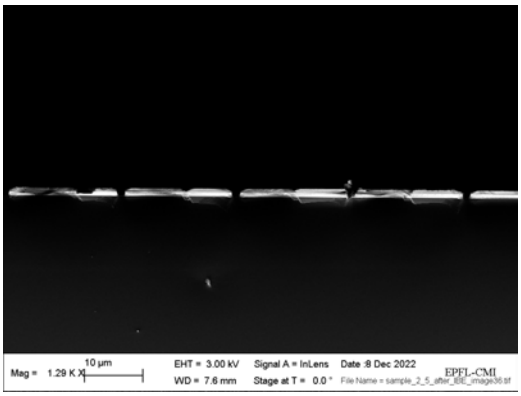
(a) Sample 2.5



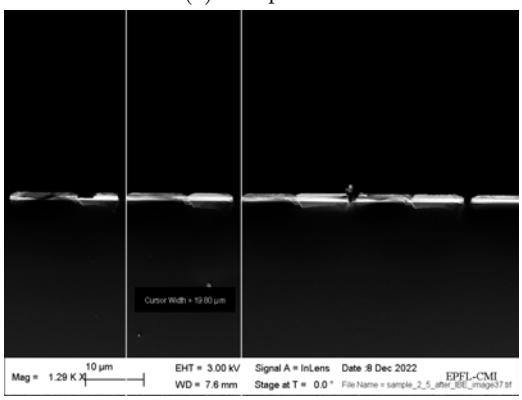
(b) Sample 2.5



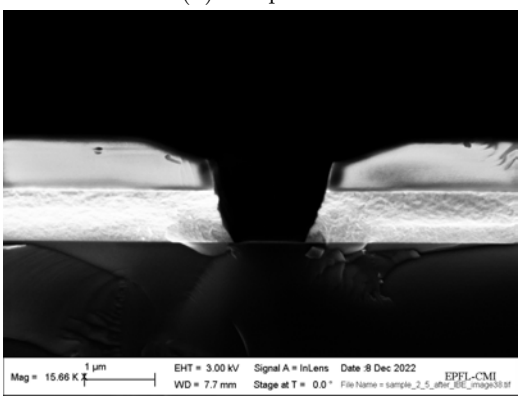
(c) Sample 2.5



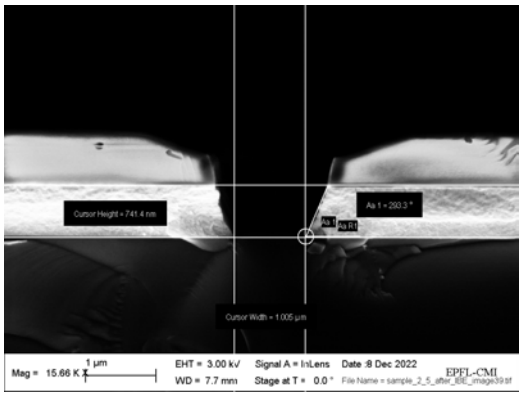
(d) Sample 2.5



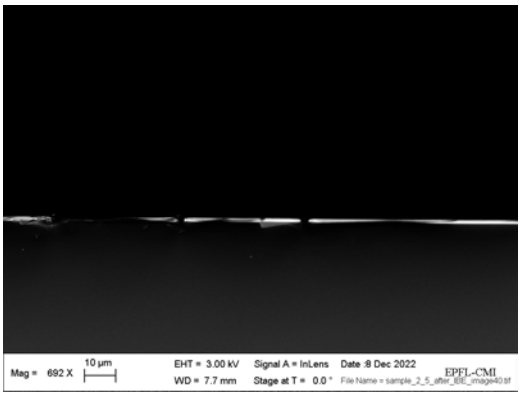
(e) Sample 2.5



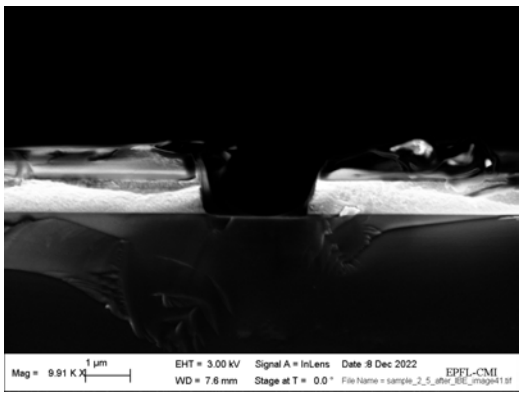
(f) Sample 2.5



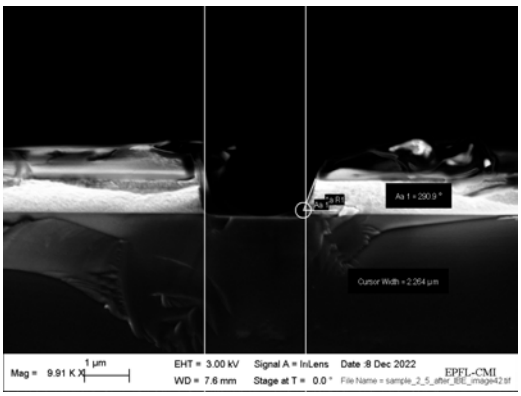
(g) Sample 2.5



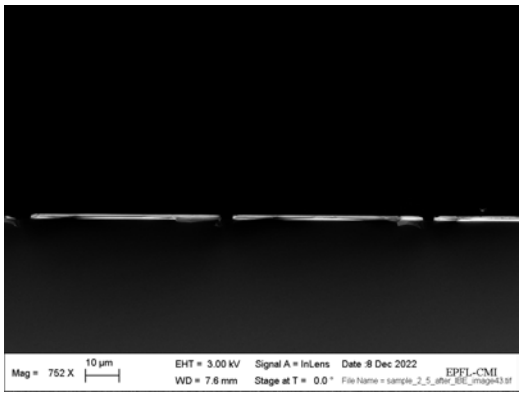
(h) Sample 2.5



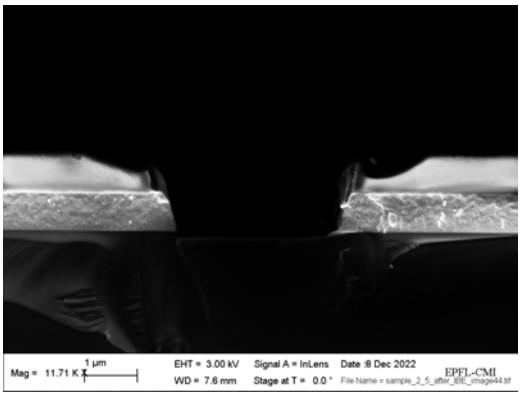
(a) Sample 2.5



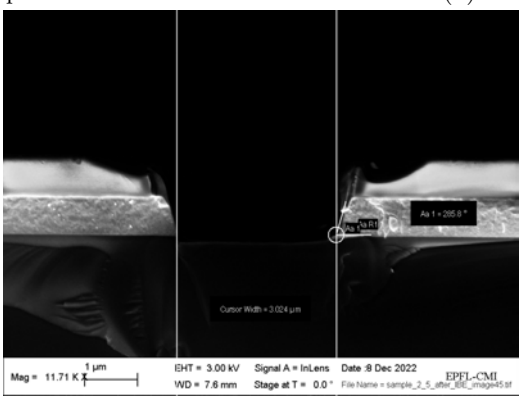
(b) Sample 2.5



(c) Sample 2.5

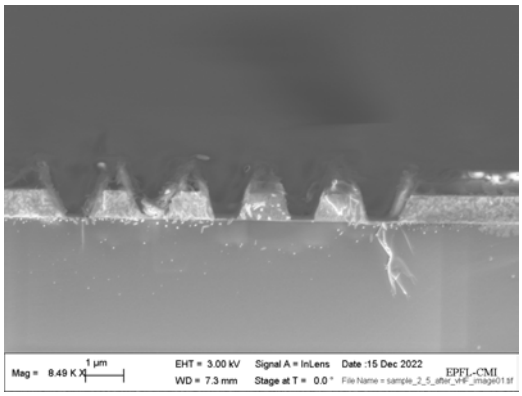


(d) Sample 2.5

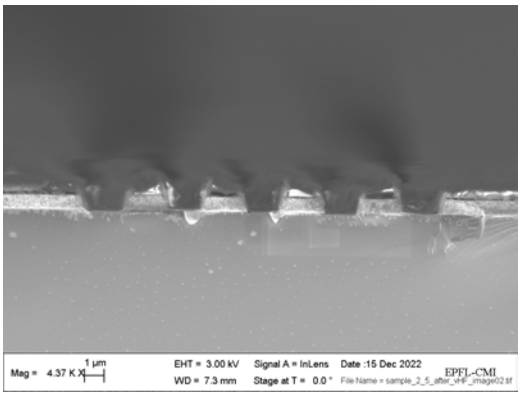


(e) Sample 2.5

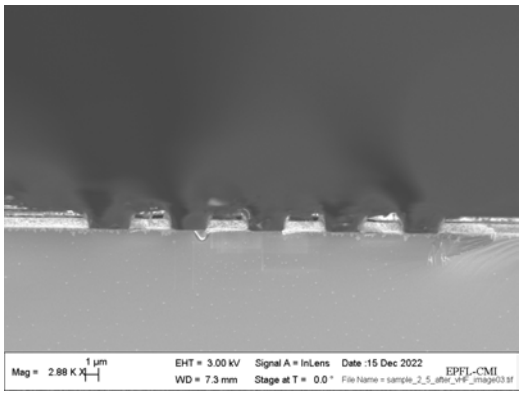
F.7.2 After vapour HF



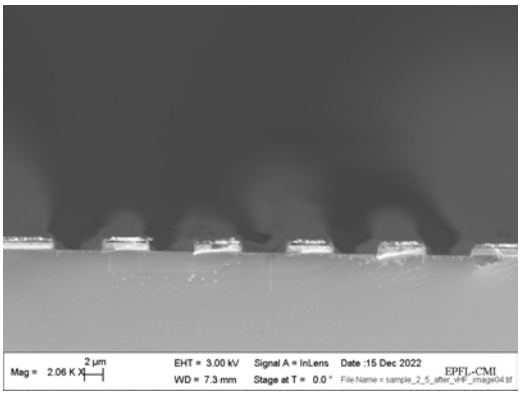
(a) Sample 2.5



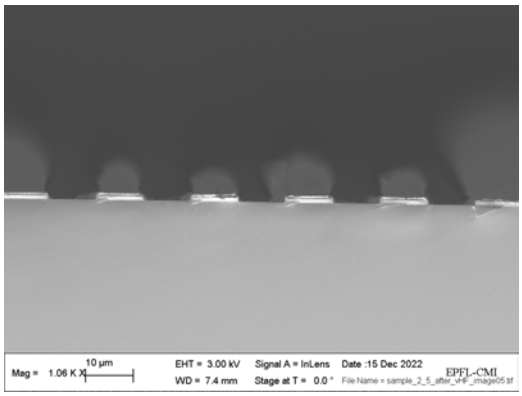
(b) Sample 2.5



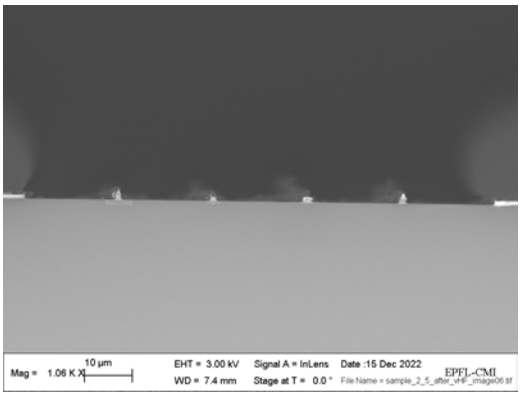
(c) Sample 2.5



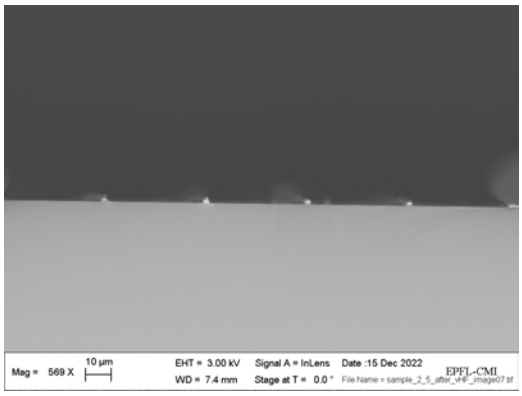
(d) Sample 2.5



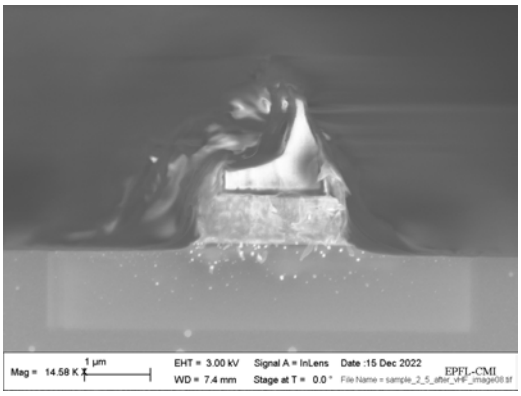
(e) Sample 2.5



(f) Sample 2.5

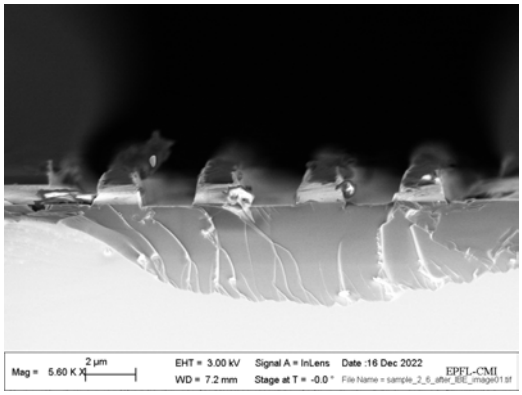


(g) Sample 2.5

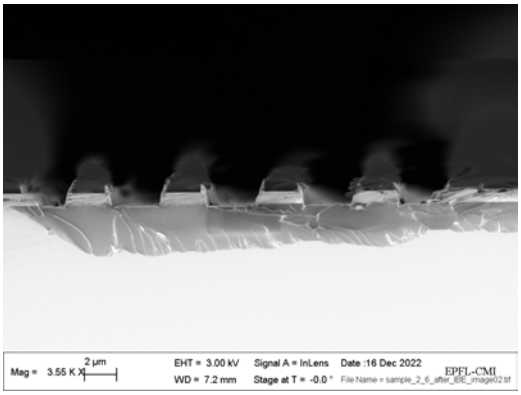


(h) Sample 2.5

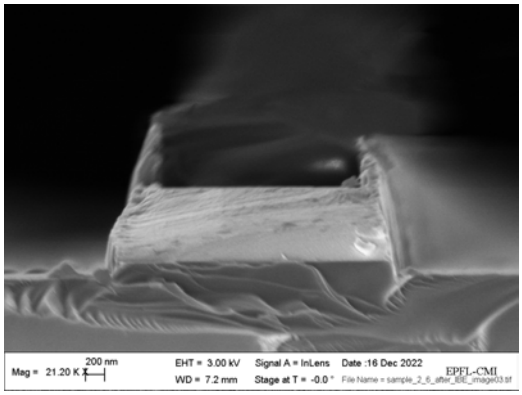
F.8 Sample 2.6



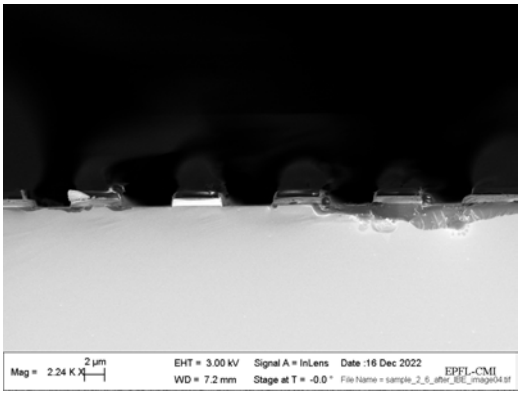
(a) Sample 2.6



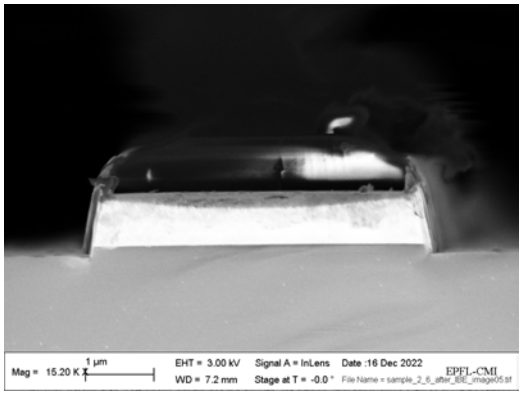
(b) Sample 2.6



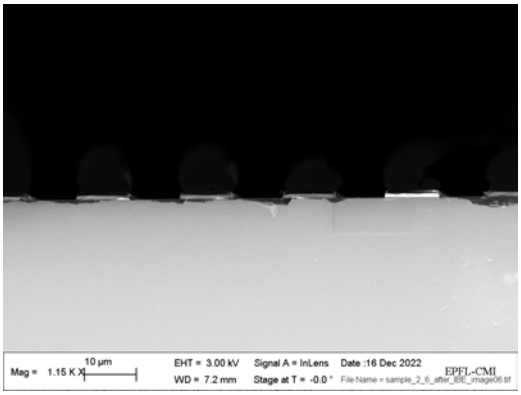
(c) Sample 2.6



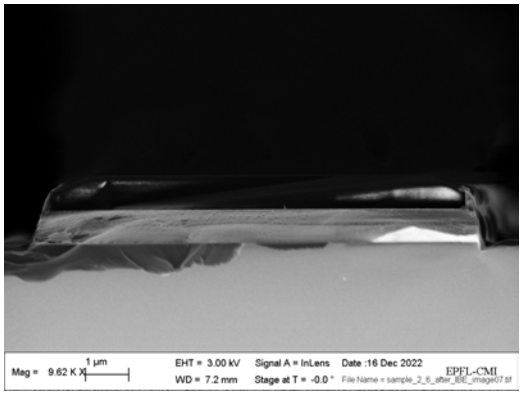
(d) Sample 2.6



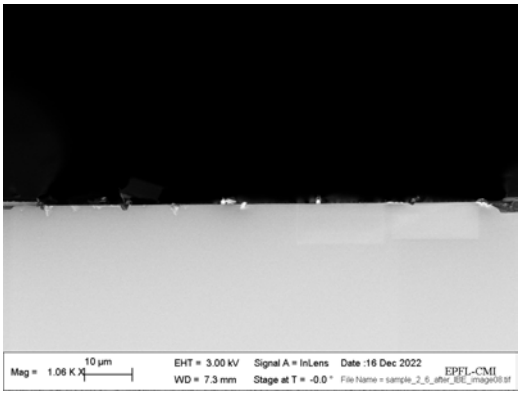
(e) Sample 2.6



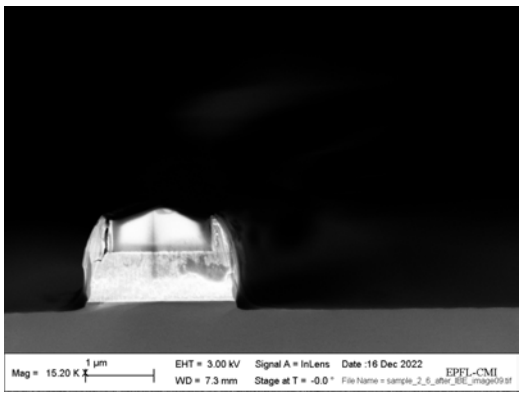
(f) Sample 2.6



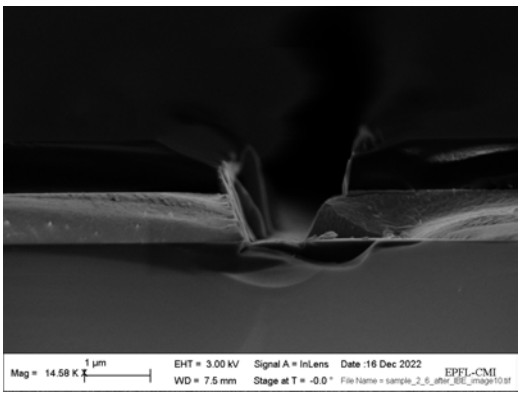
(g) Sample 2.6



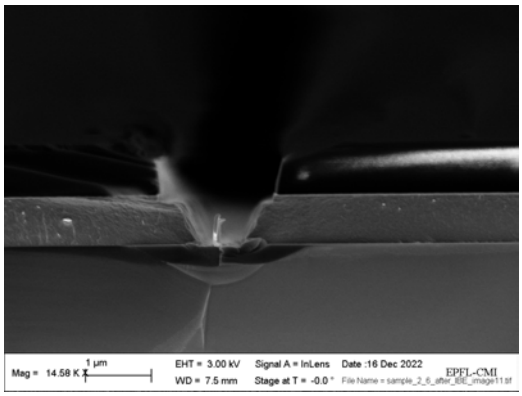
(h) Sample 2.6



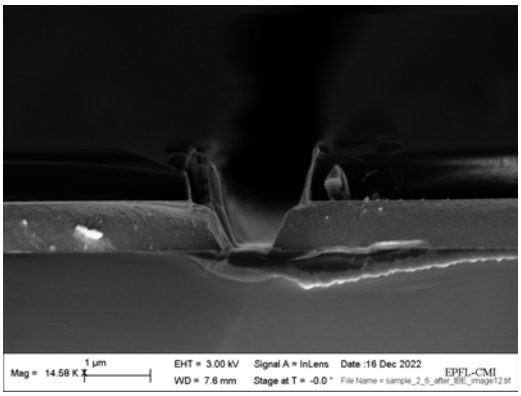
(a) Sample 2.6



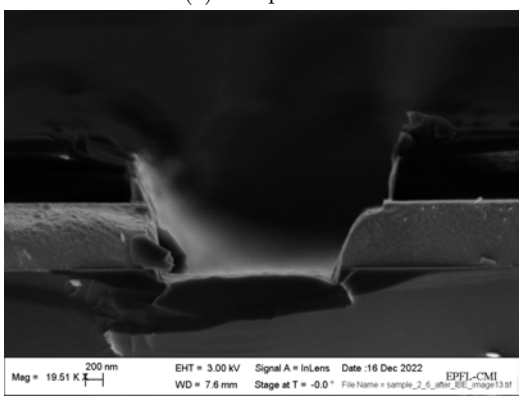
(b) Sample 2.6



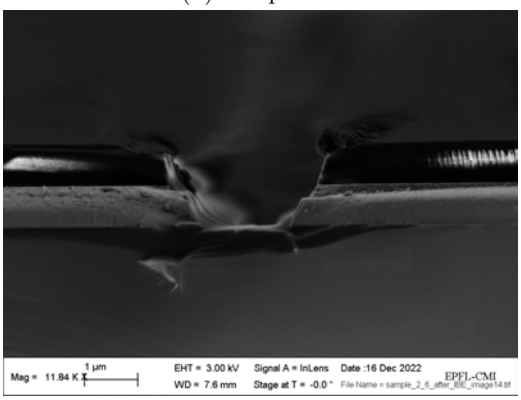
(c) Sample 2.6



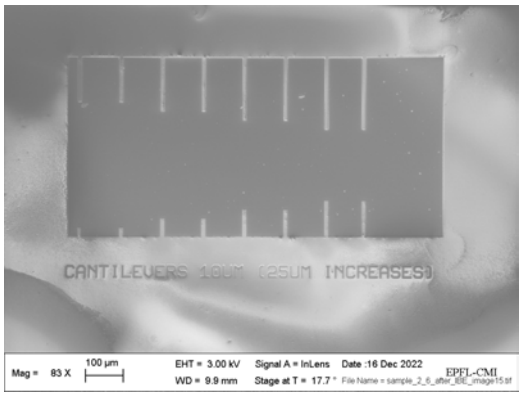
(d) Sample 2.6



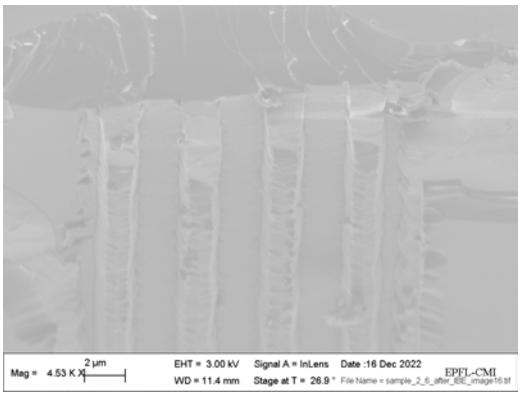
(e) Sample 2.6



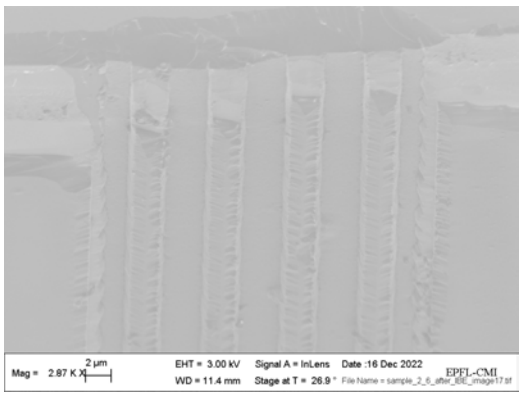
(f) Sample 2.6



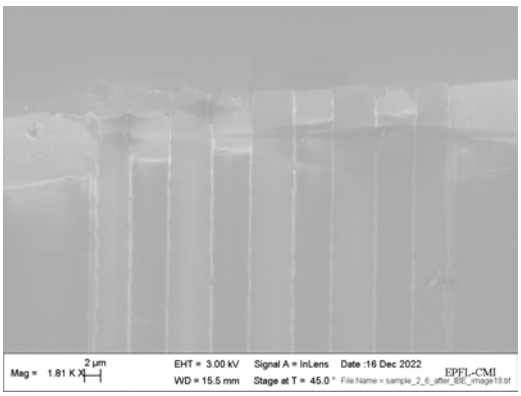
(g) Sample 2.6



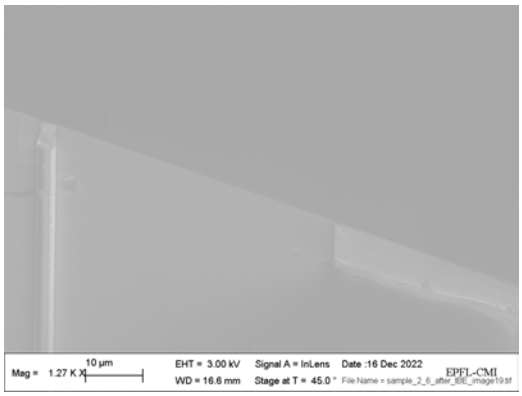
(h) Sample 2.6



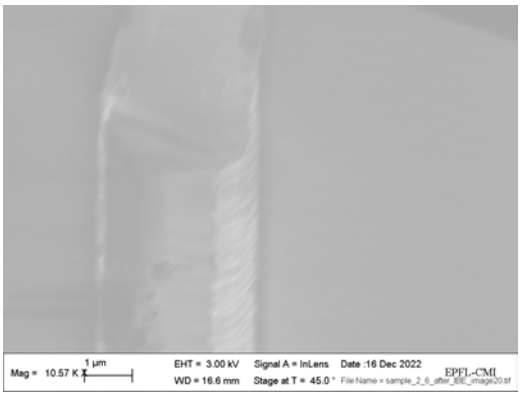
(a) Sample 2.6



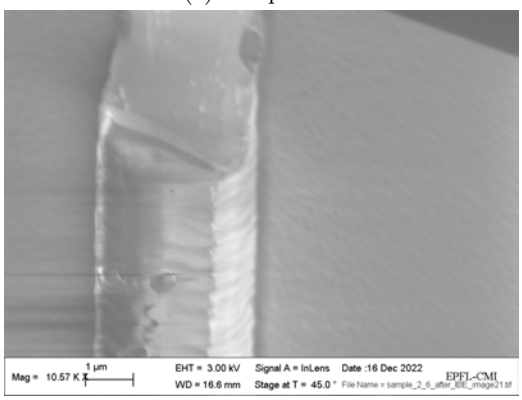
(b) Sample 2.6



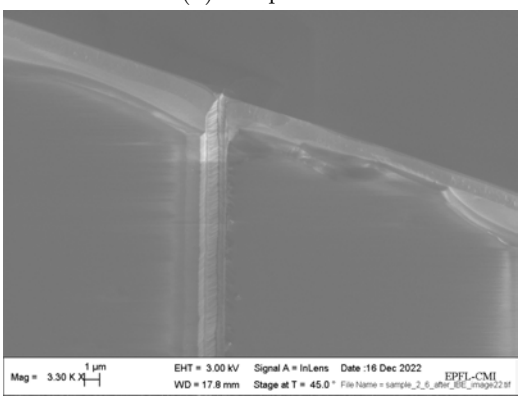
(c) Sample 2.6



(d) Sample 2.6



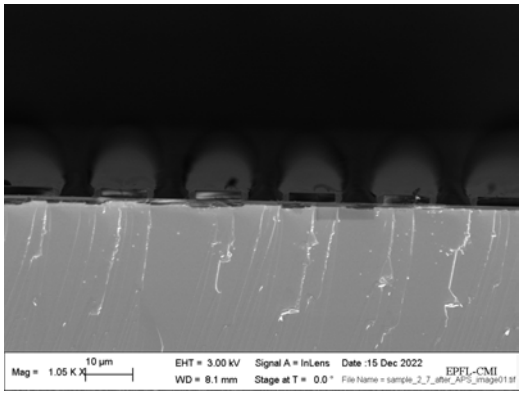
(e) Sample 2.6



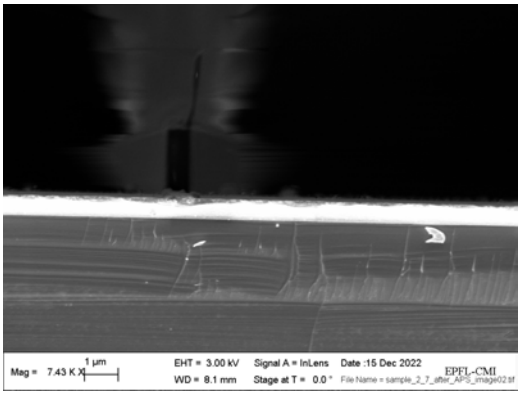
(f) Sample 2.6

F.9 Sample 2.7

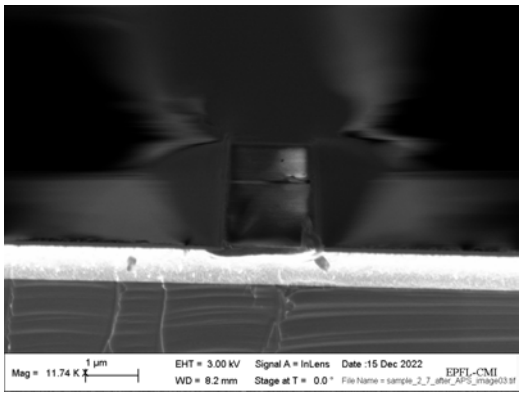
F.9.1 After APS



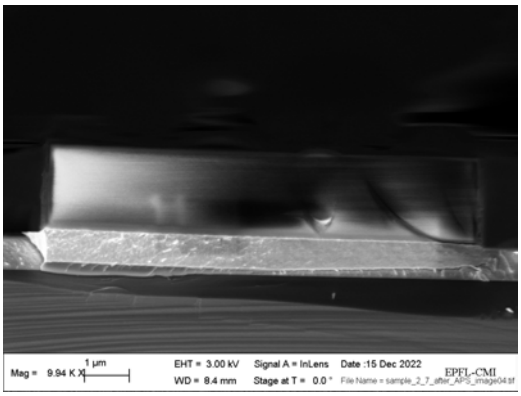
(a) Sample 2.7



(b) Sample 2.7



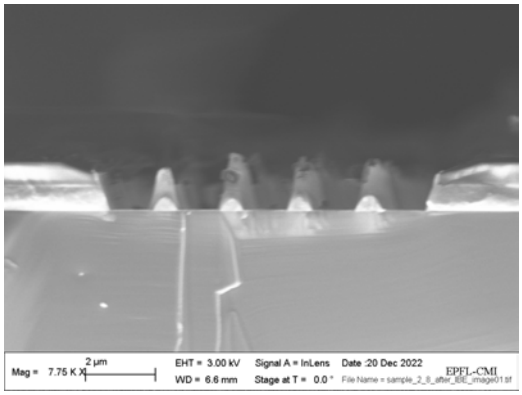
(c) Sample 2.7



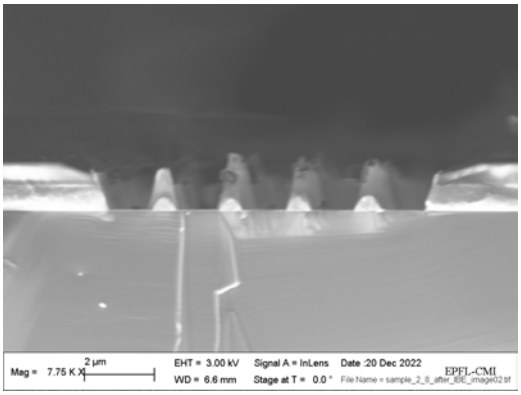
(d) Sample 2.7

F.10 Sample 2.8

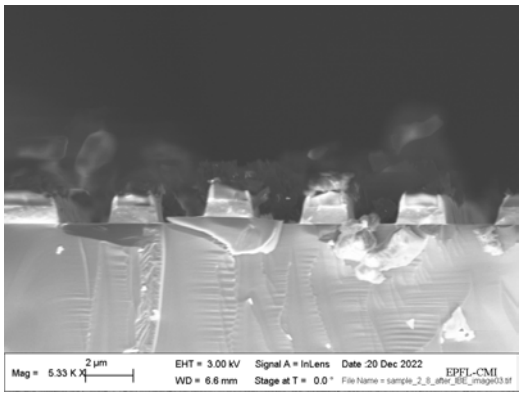
F.10.1 After IBE



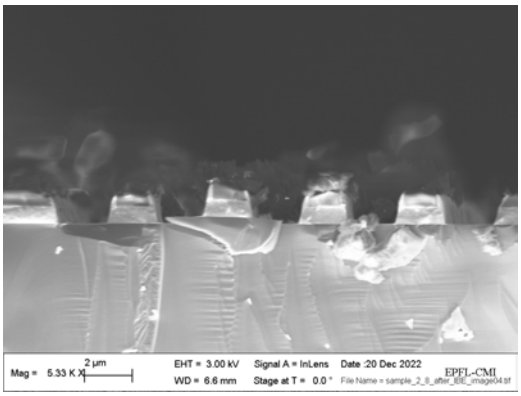
(a) Sample 2.8



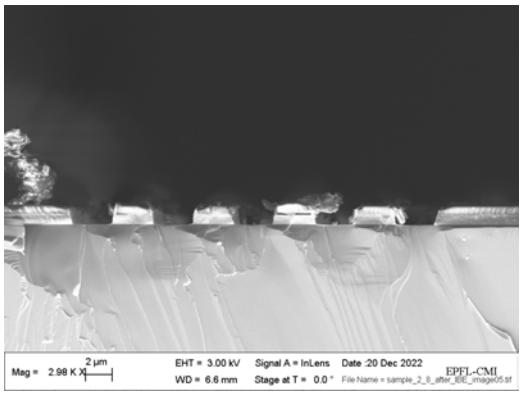
(b) Sample 2.8



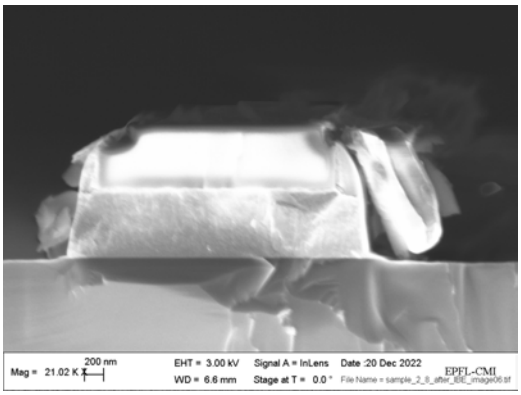
(c) Sample 2.8



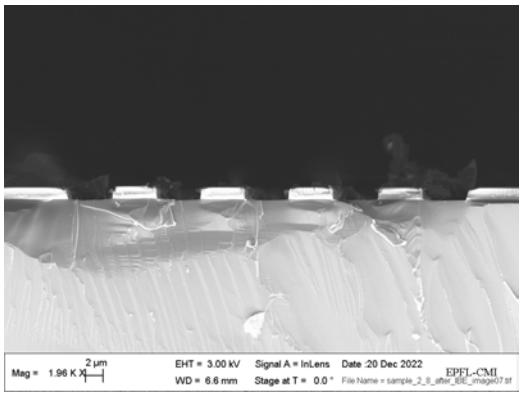
(d) Sample 2.8



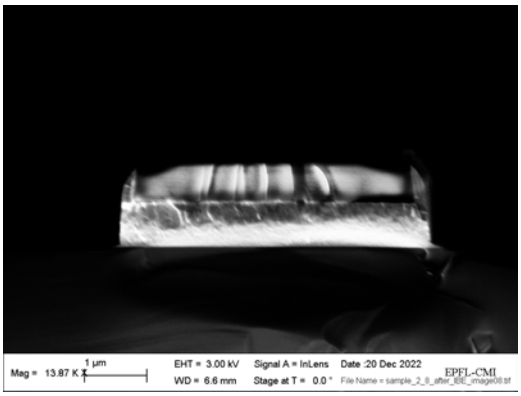
(e) Sample 2.8



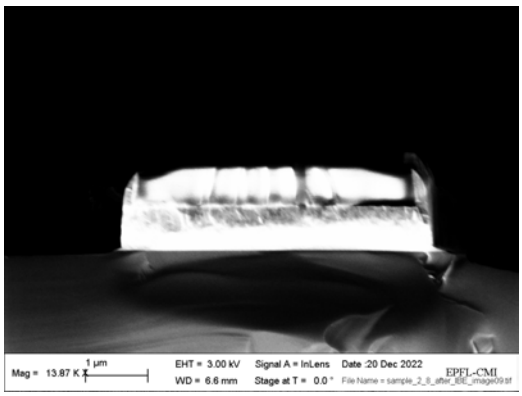
(f) Sample 2.8



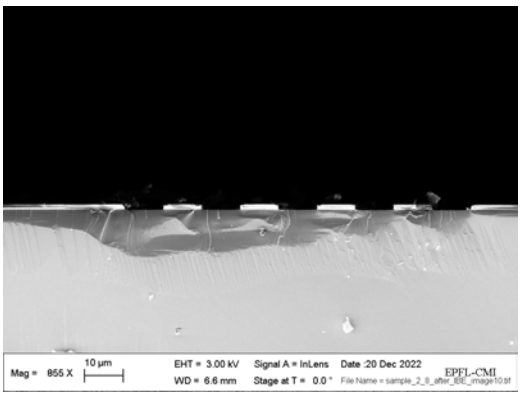
(g) Sample 2.8



(h) Sample 2.8



(a) Sample 2.8



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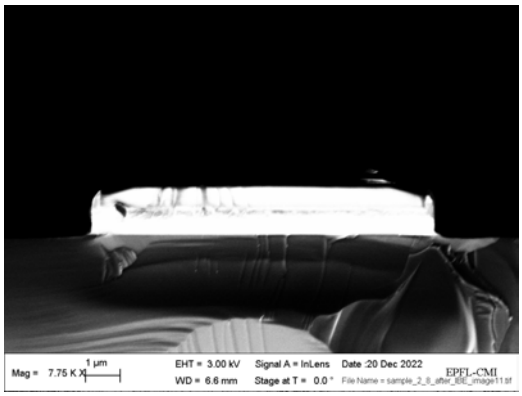
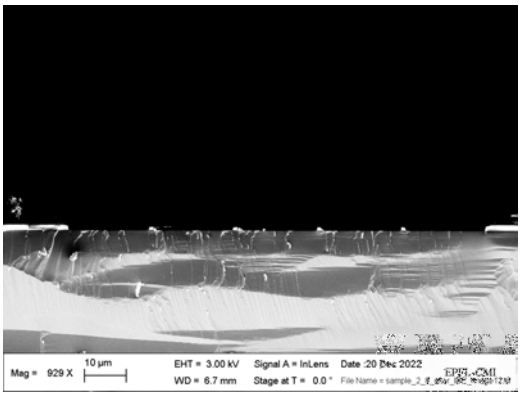
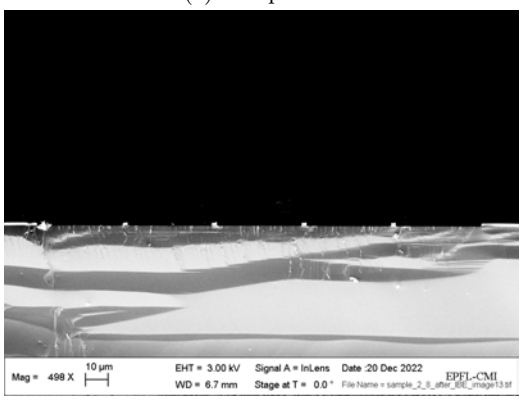


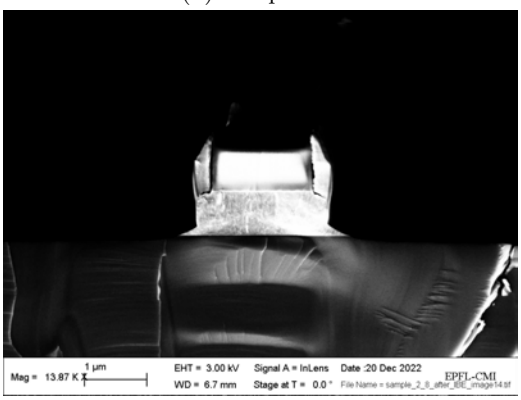
(c) Sample 2.8



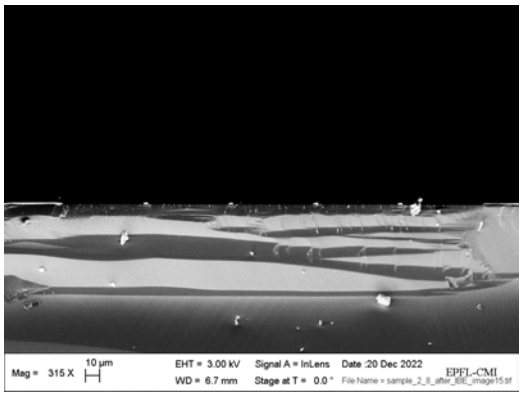
(d) Sample 2.8



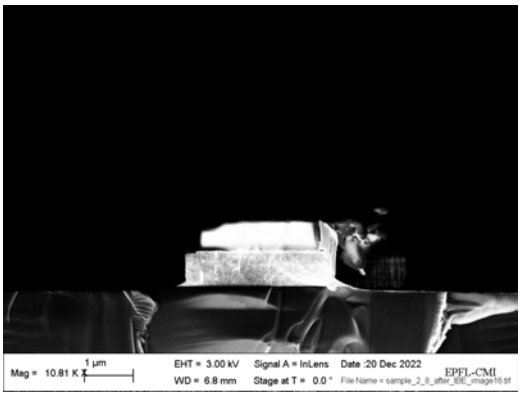
(e) Sample 2.8



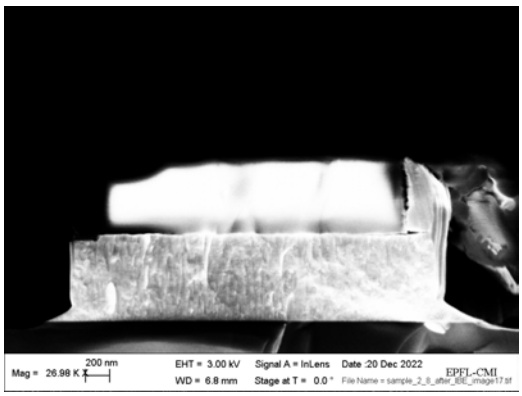
(f) Sample 2.8



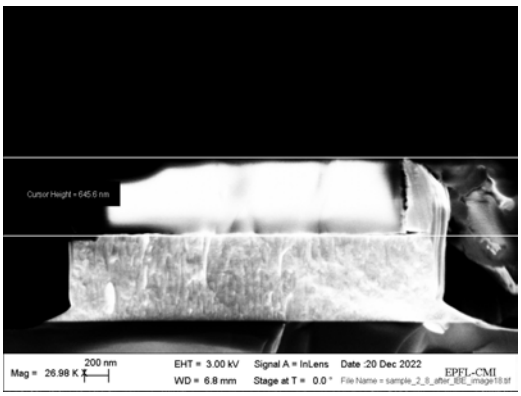
(g) Sample 2.8



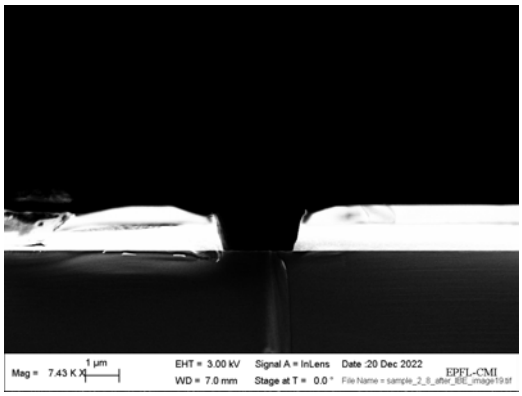
(h) Sample 2.8



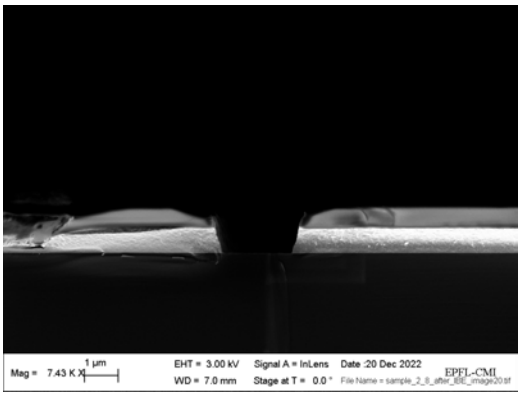
(a) Sample 2.8



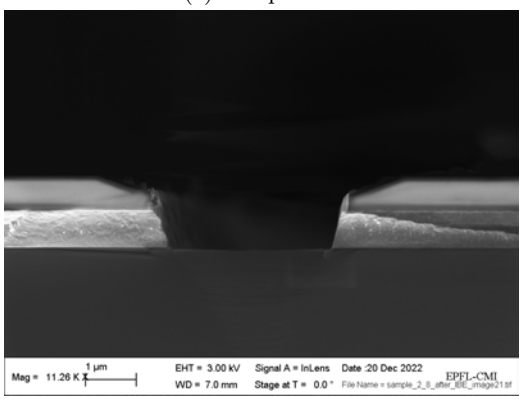
(b) Sample 2.8



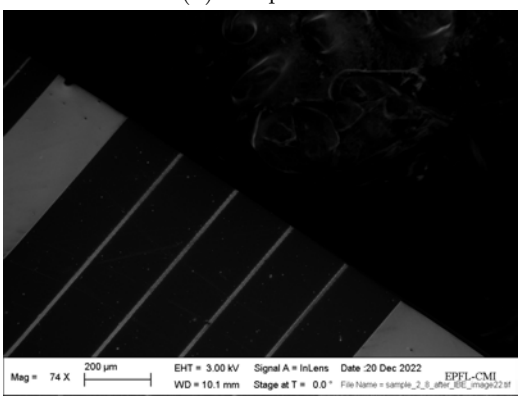
(c) Sample 2.8



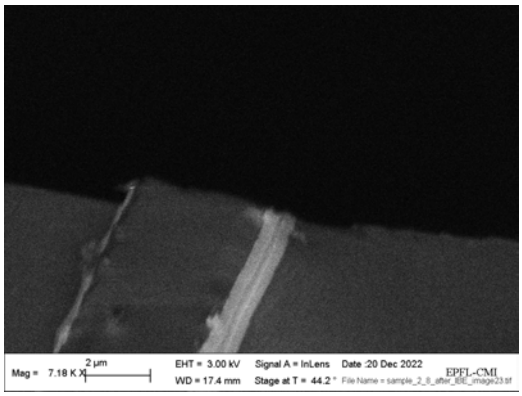
(d) Sample 2.8



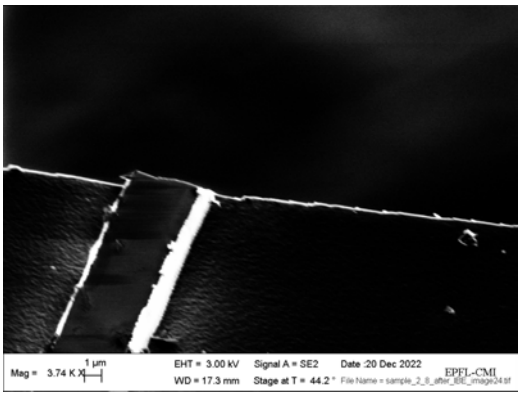
(e) Sample 2.8



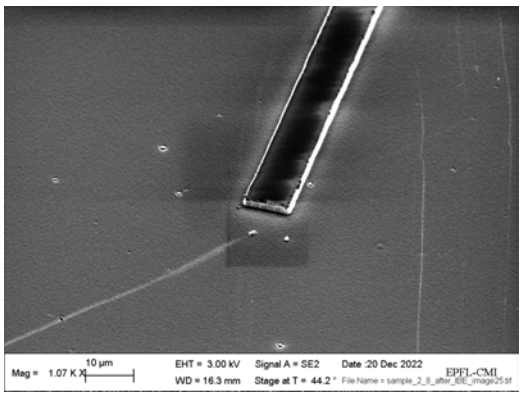
(f) Sample 2.8



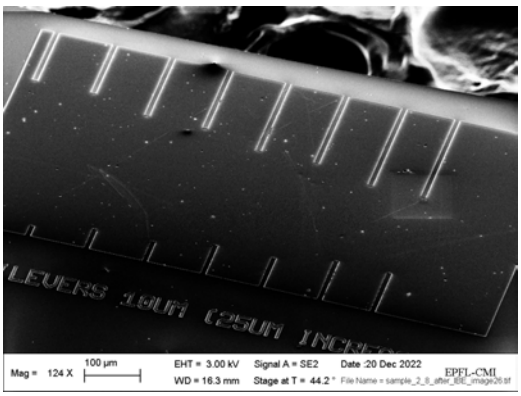
(g) Sample 2.8



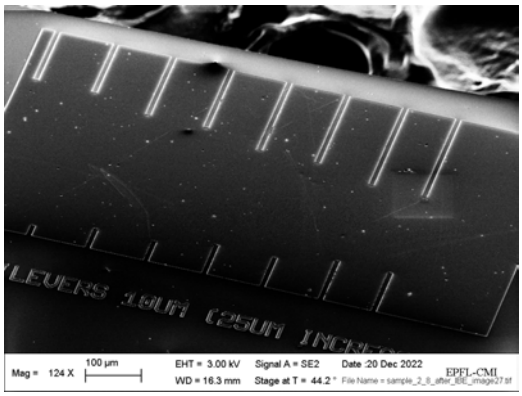
(h) Sample 2.8



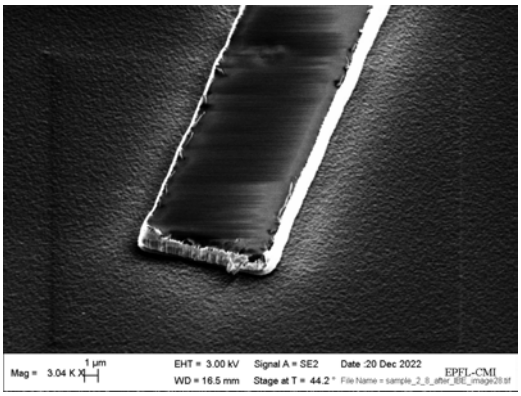
(a) Sample 2.8



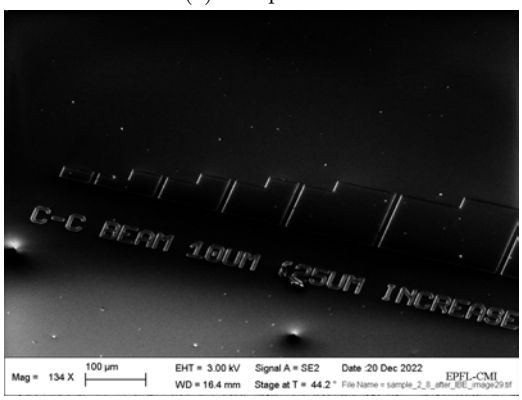
(b) Sample 2.8



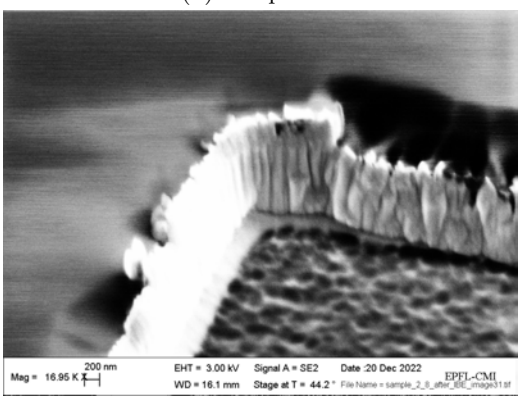
(c) Sample 2.8



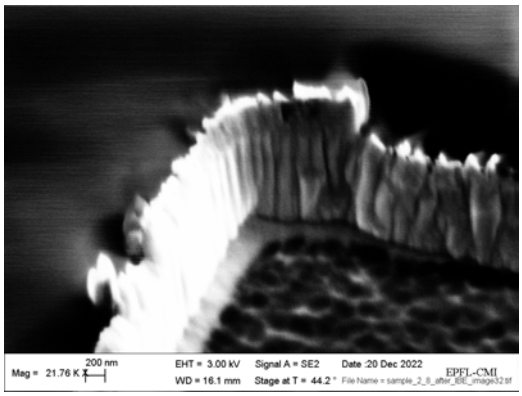
(d) Sample 2.8



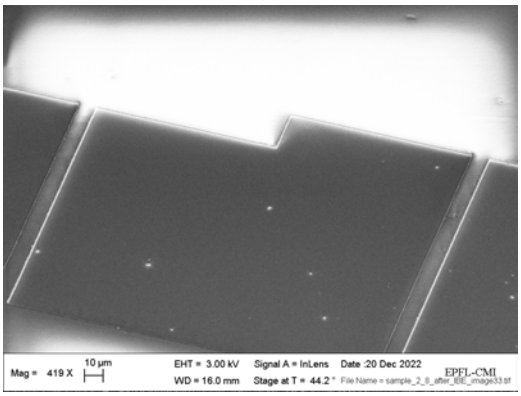
(e) Sample 2.8



(f) Sample 2.8

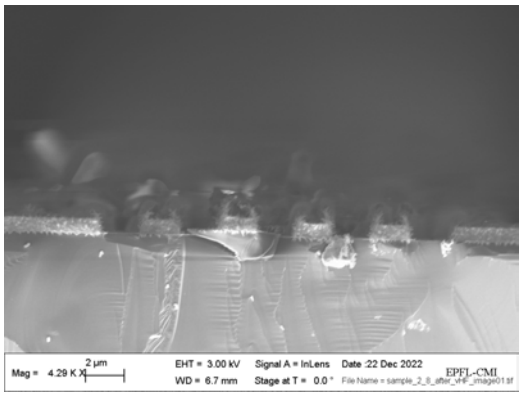


(g) Sample 2.8

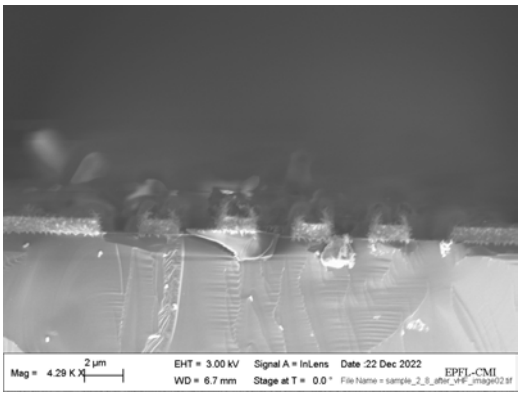


(h) Sample 2.8

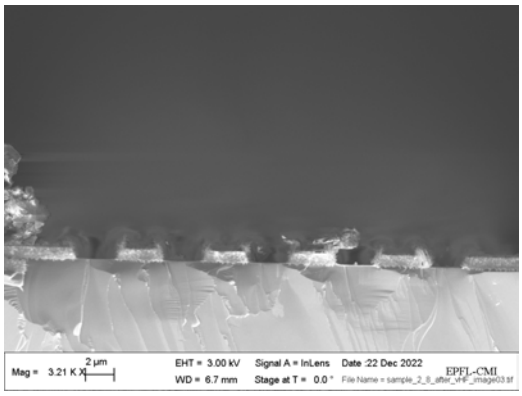
F.10.2 After vapour HF



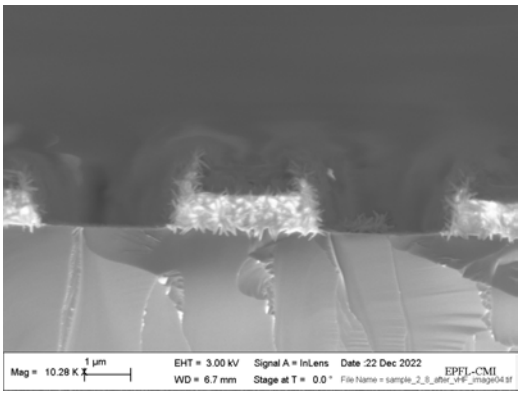
(a) Sample 2.8



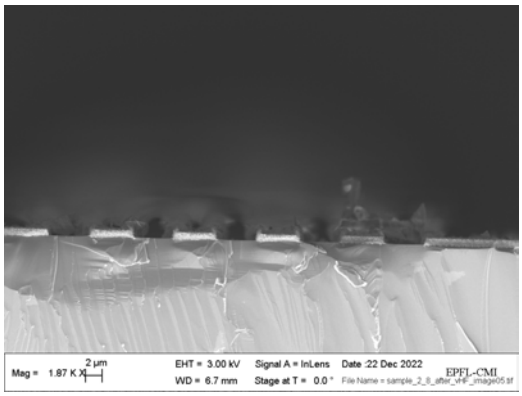
(b) Sample 2.8



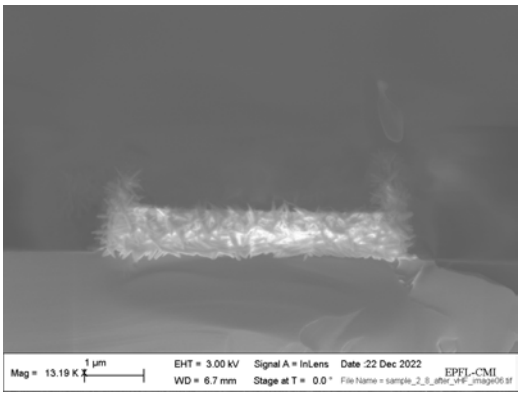
(c) Sample 2.8



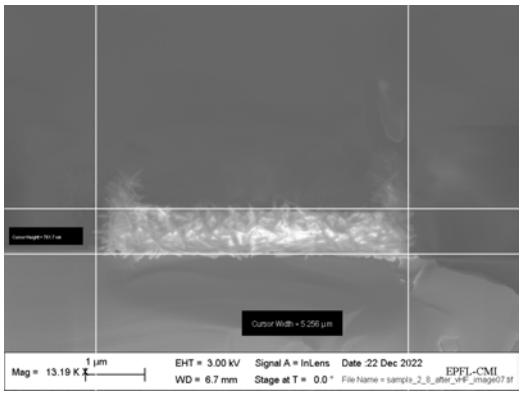
(d) Sample 2.8



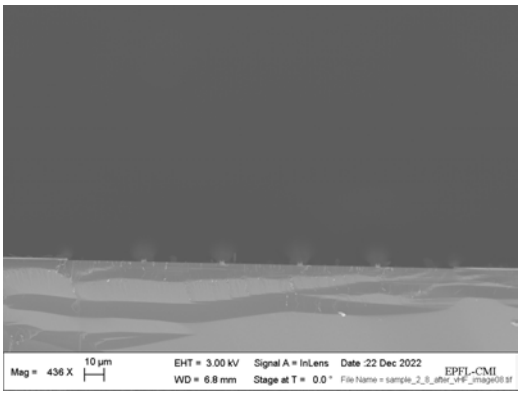
(e) Sample 2.8



(f) Sample 2.8



(g) Sample 2.8



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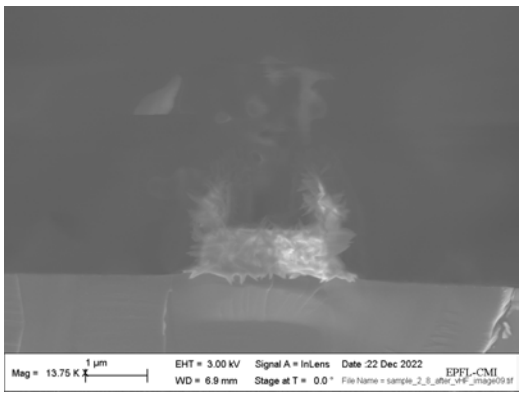

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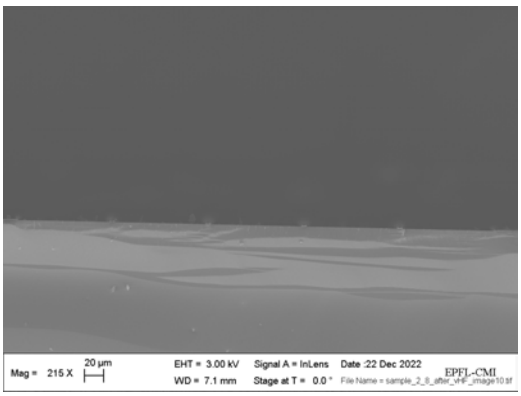

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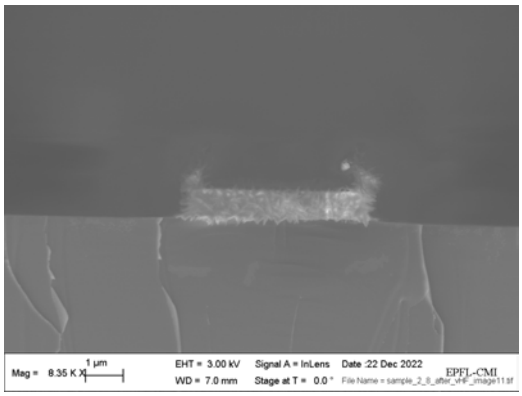

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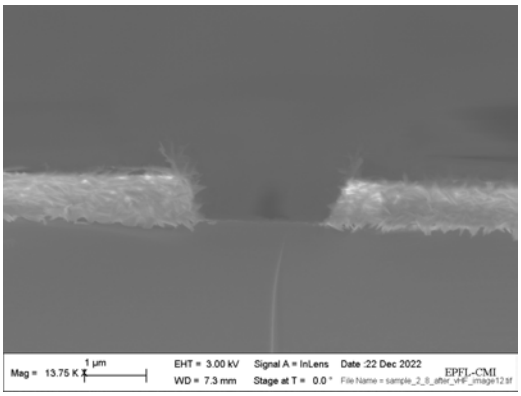

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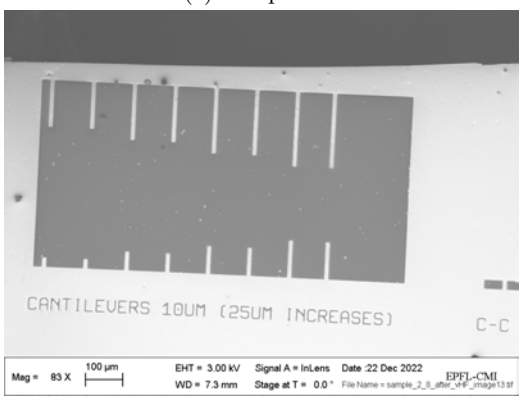

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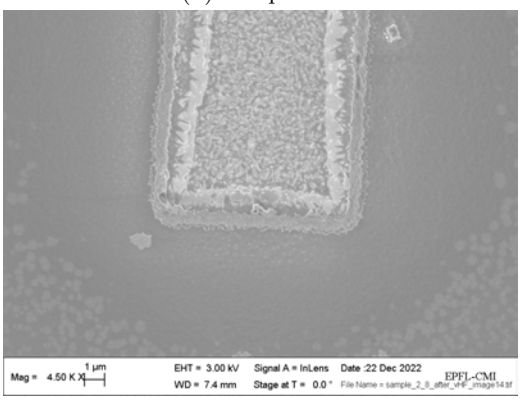

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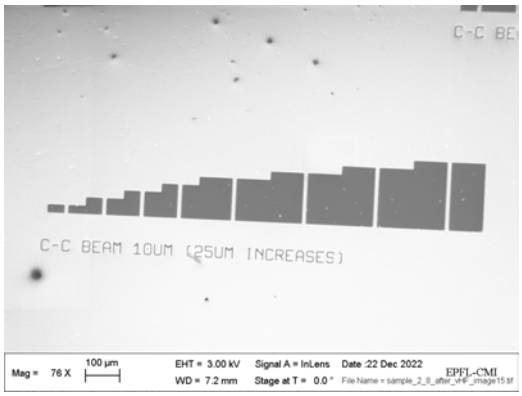

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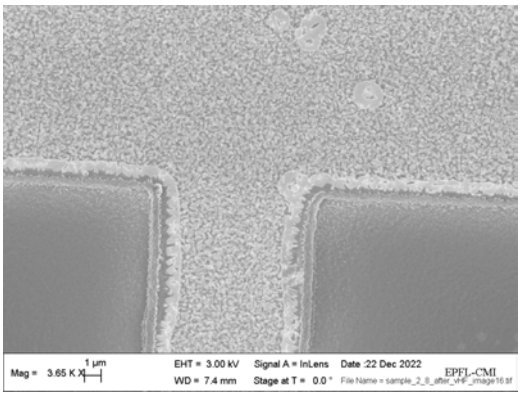

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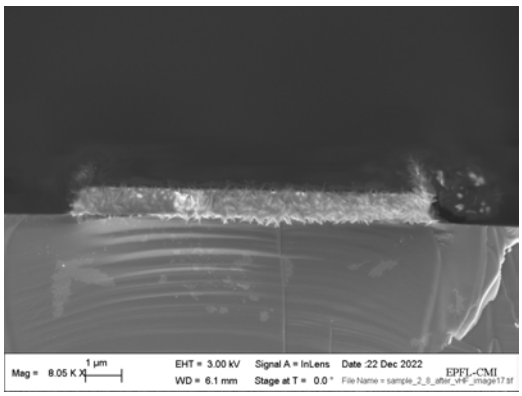
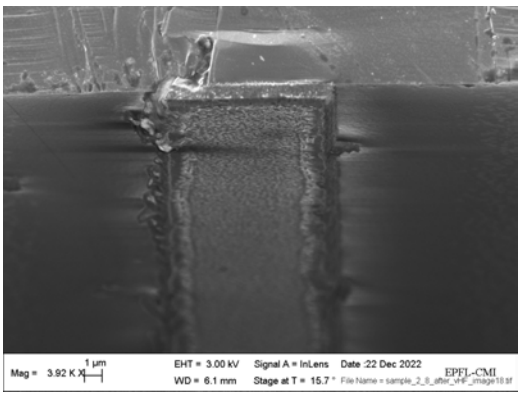


(a) Sample 2.8



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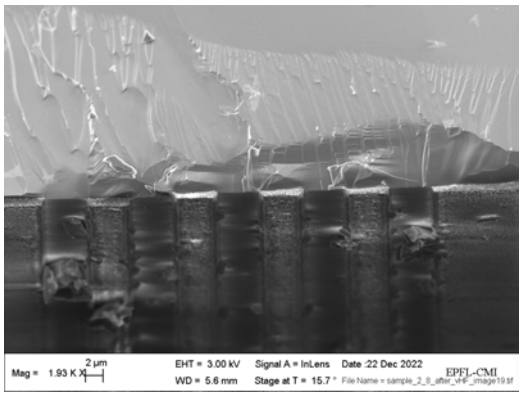

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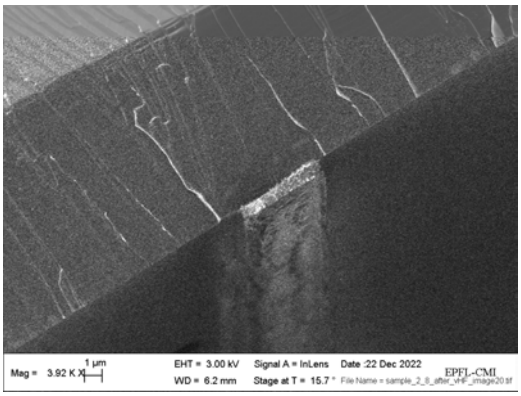

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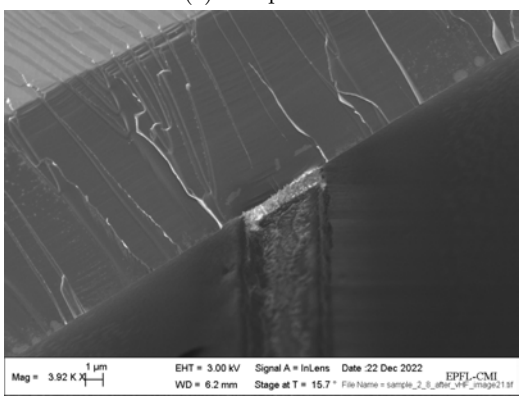

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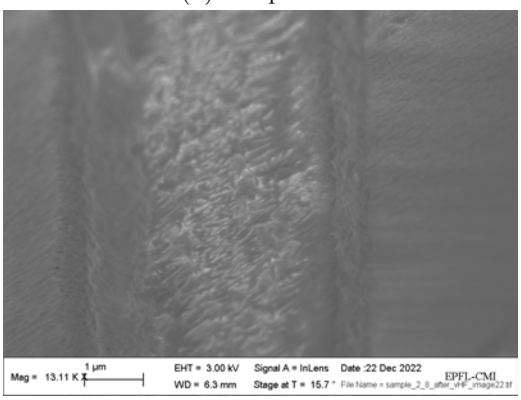

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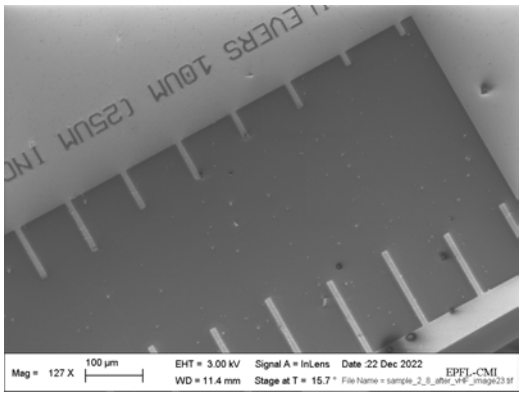

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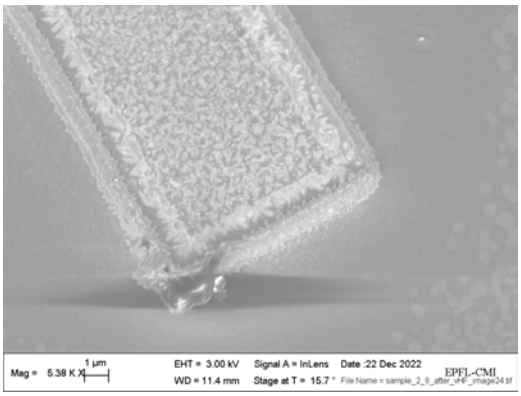

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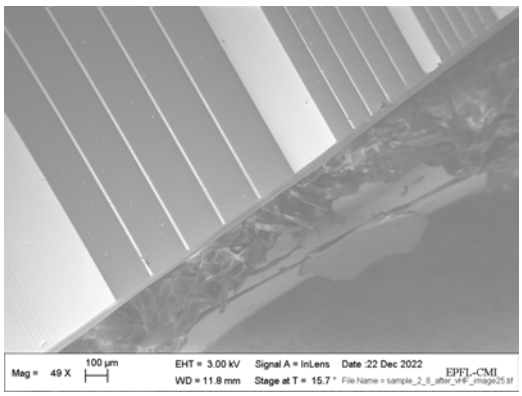

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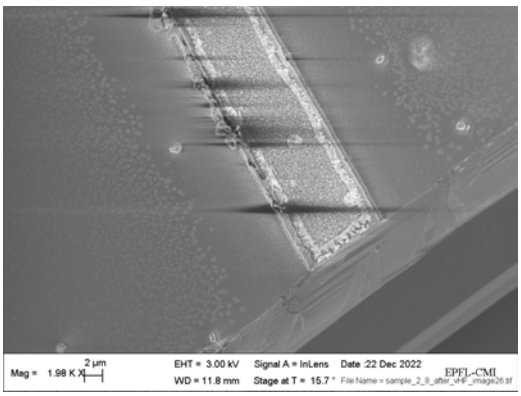

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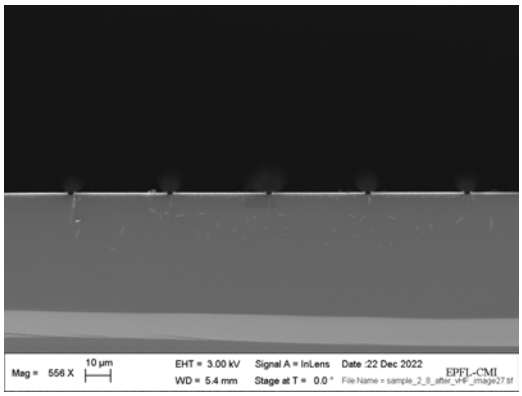

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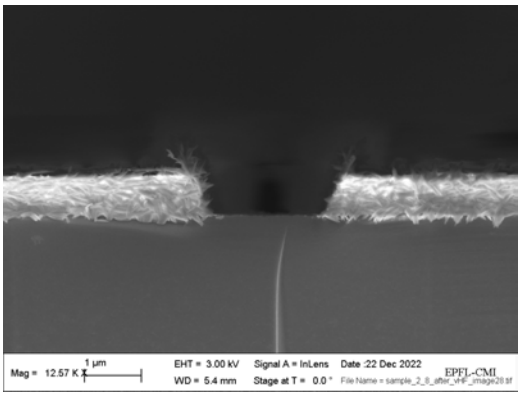

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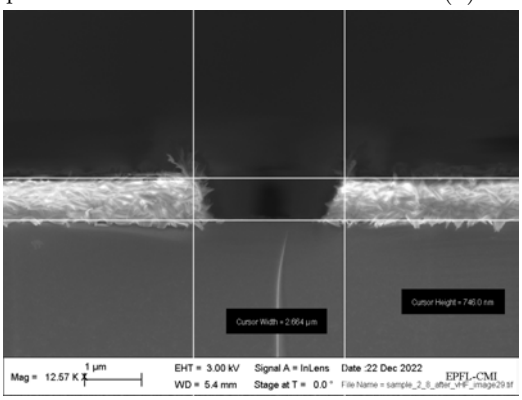
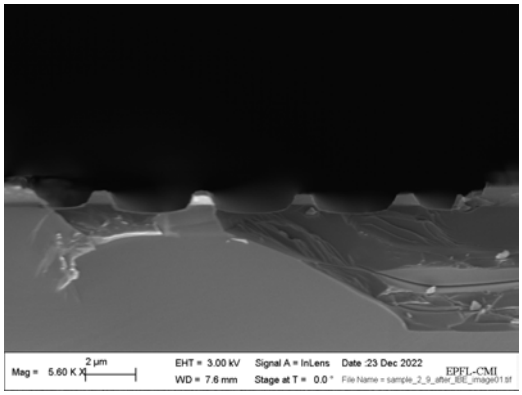


(e) Sample 2.8

F.11 Sample 2.9



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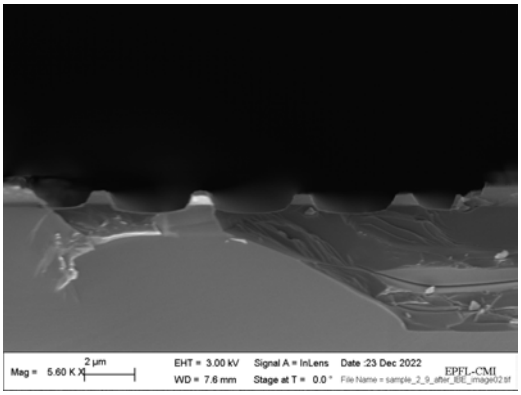

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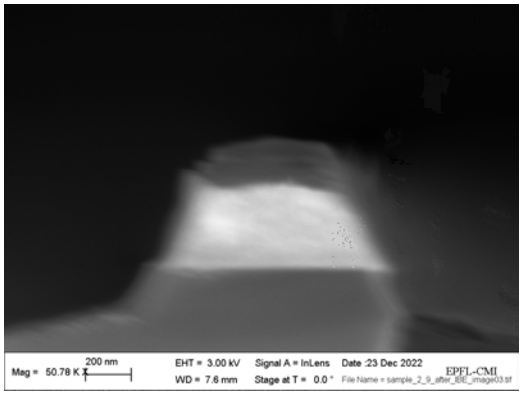

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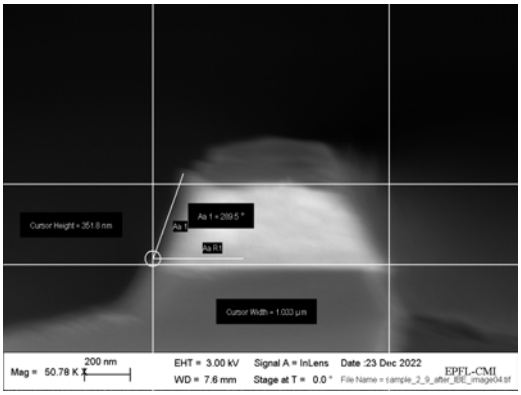

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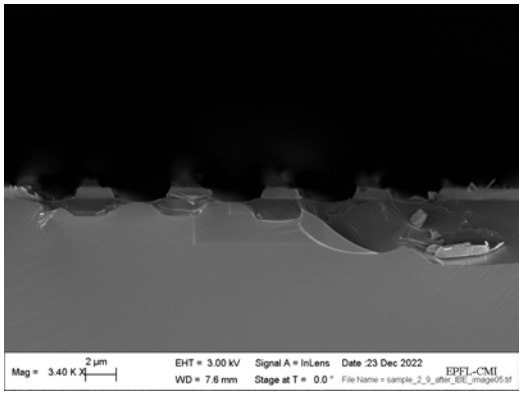

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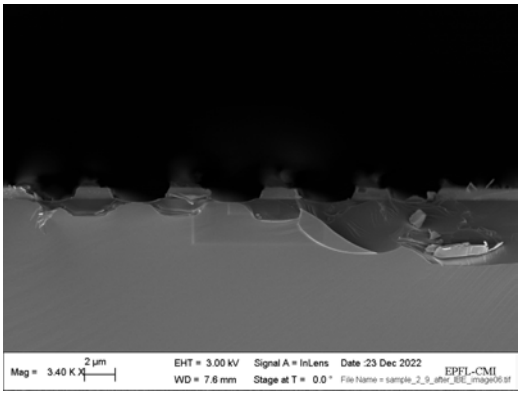

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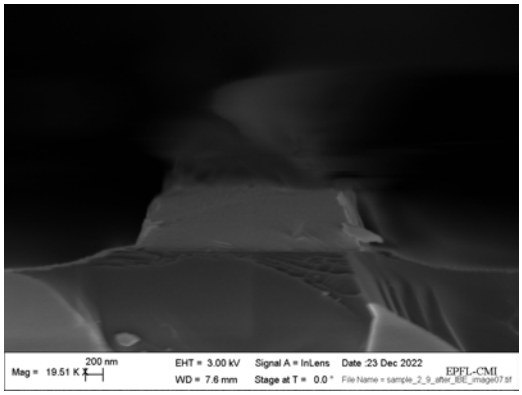

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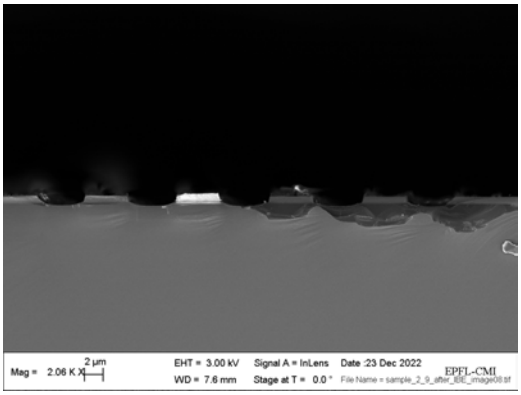

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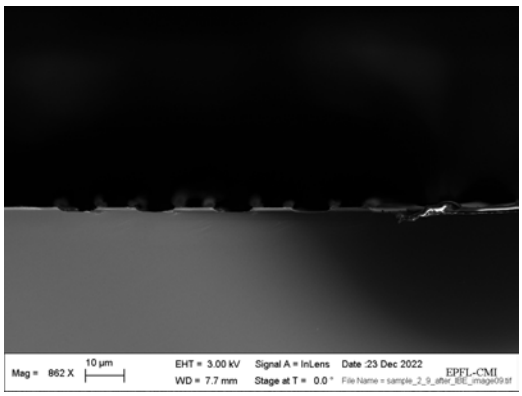

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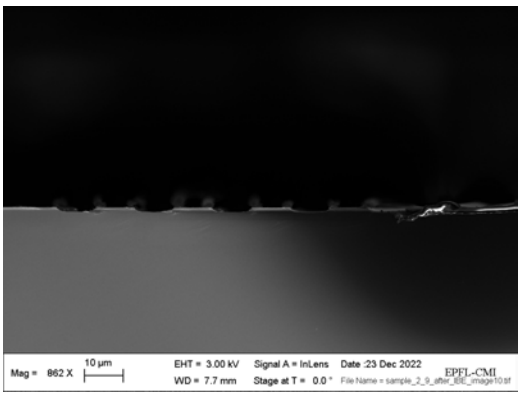

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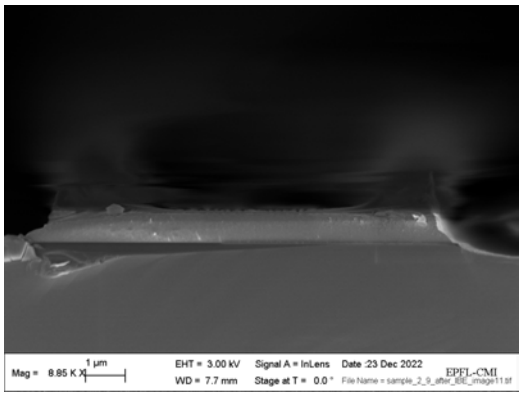

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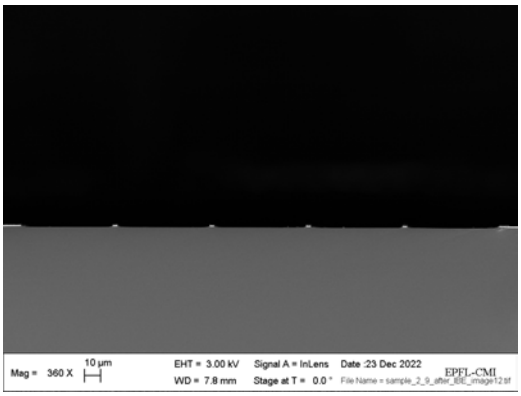

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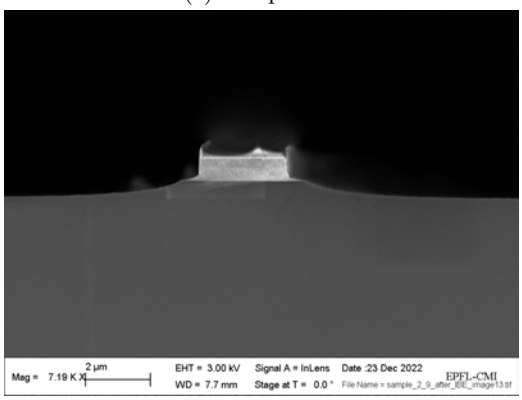

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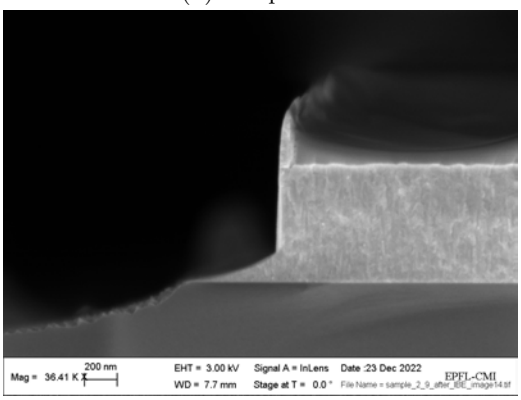

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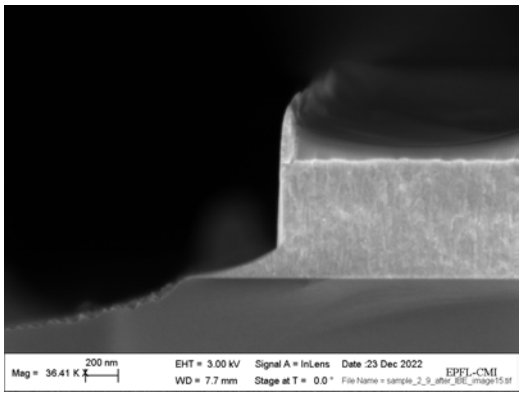

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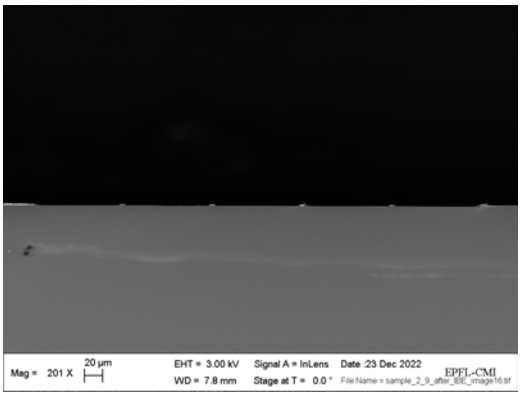

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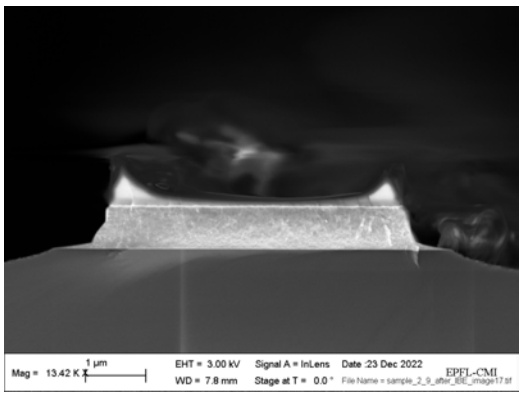

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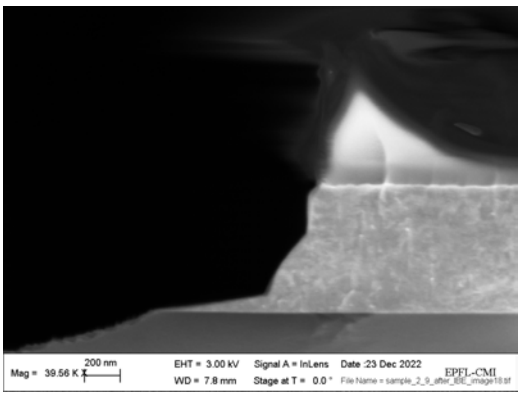

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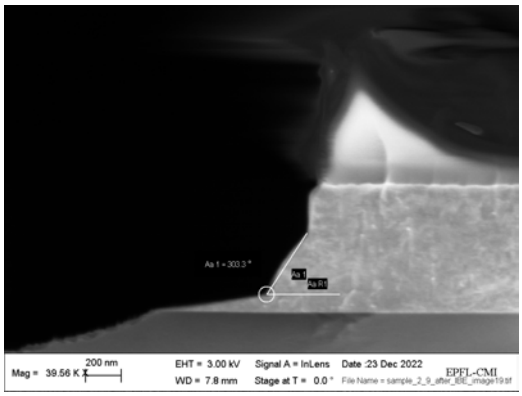

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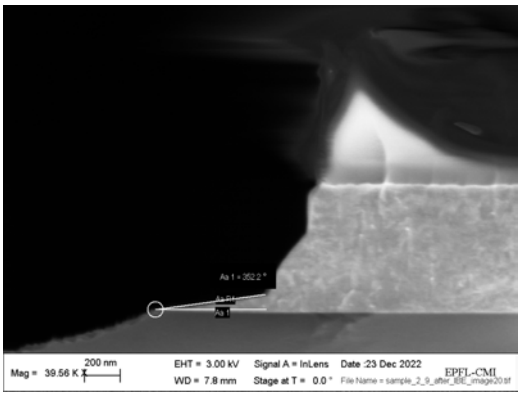

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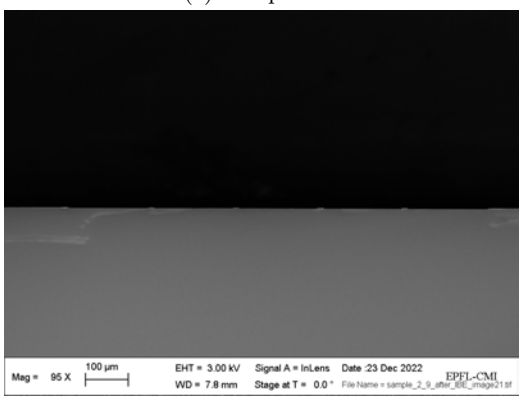

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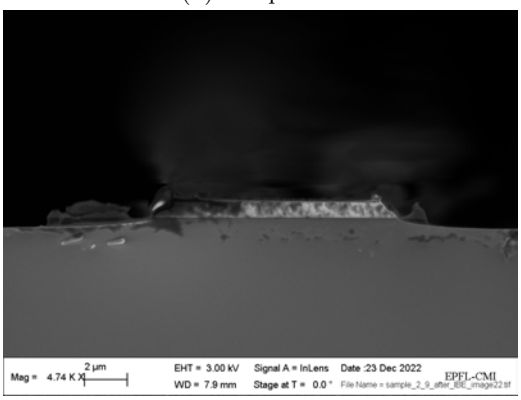

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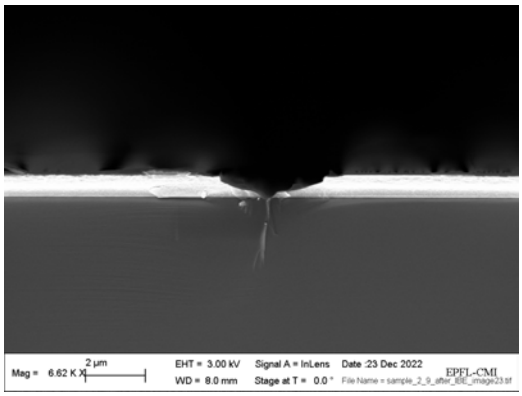

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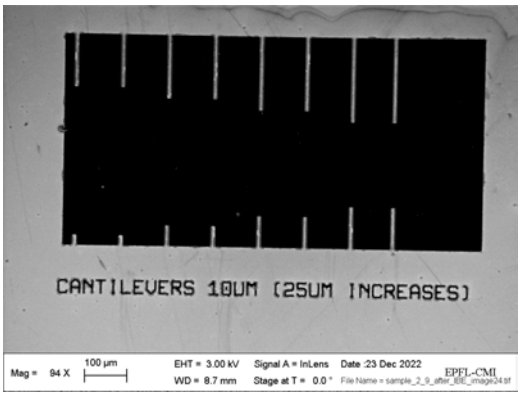

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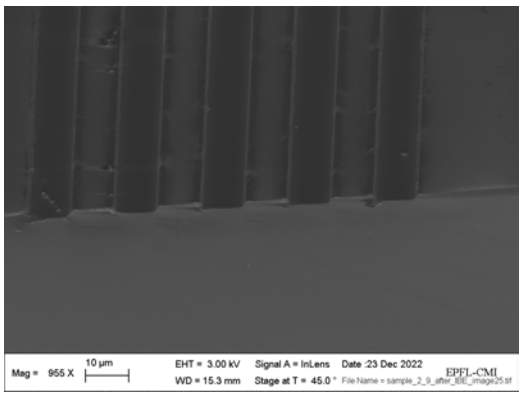

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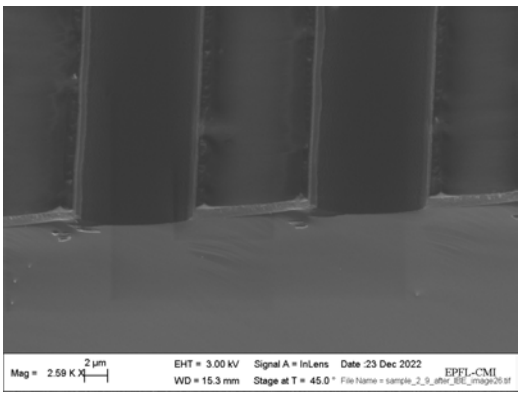

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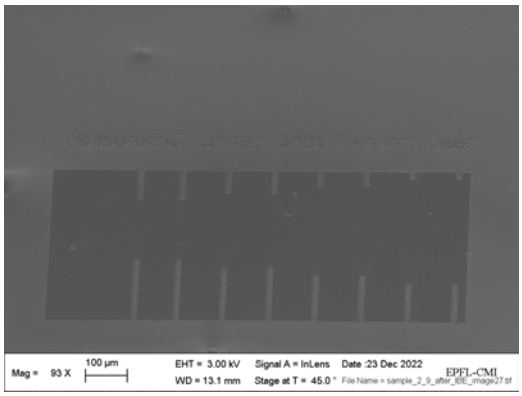

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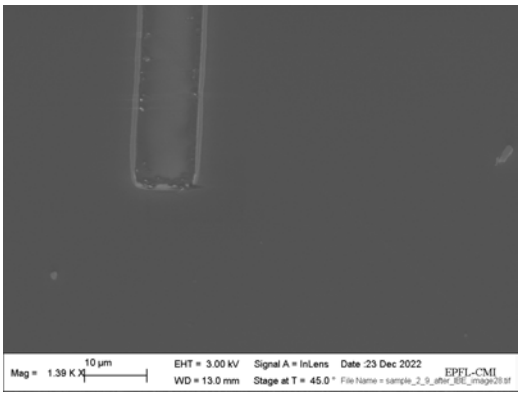

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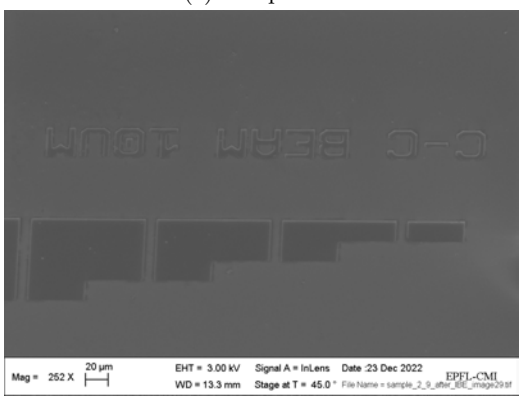

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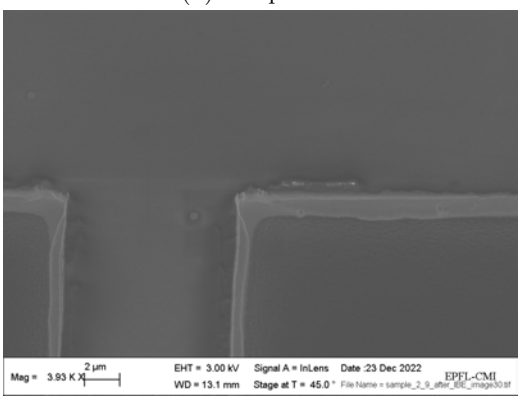

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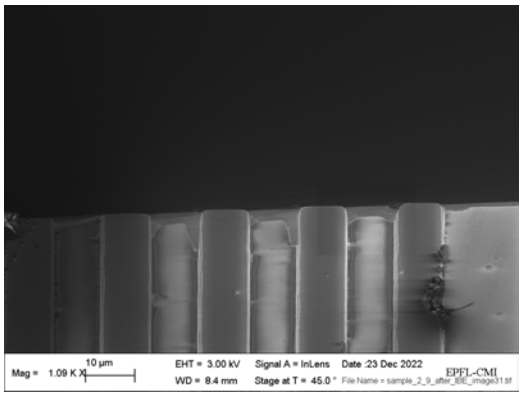

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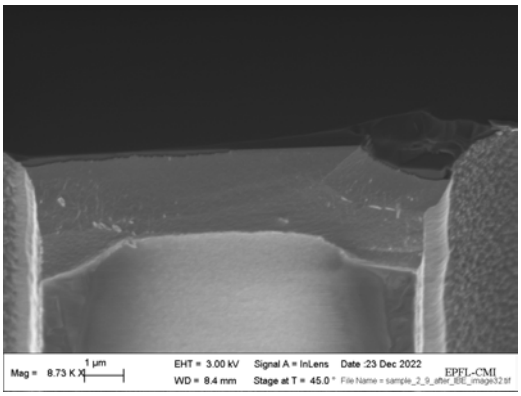

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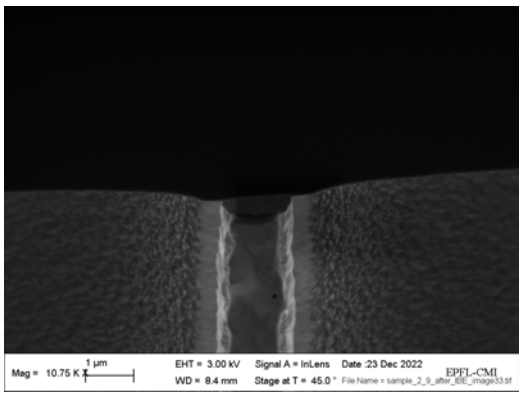

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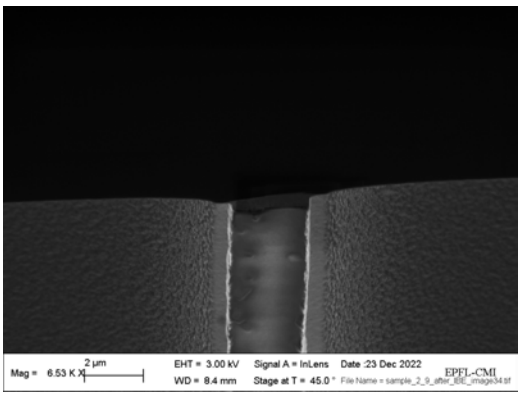

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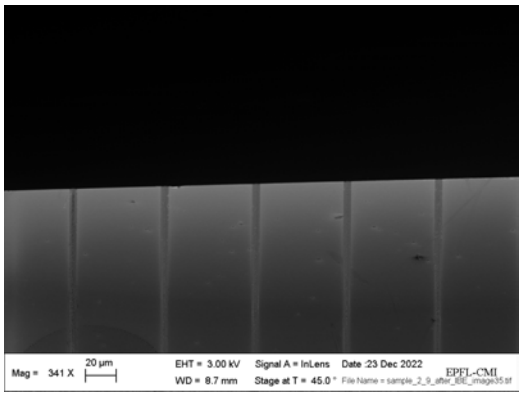
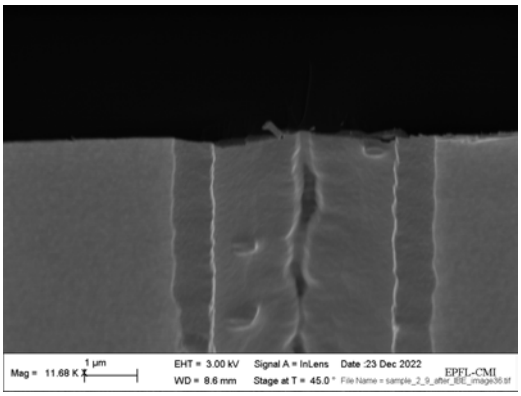
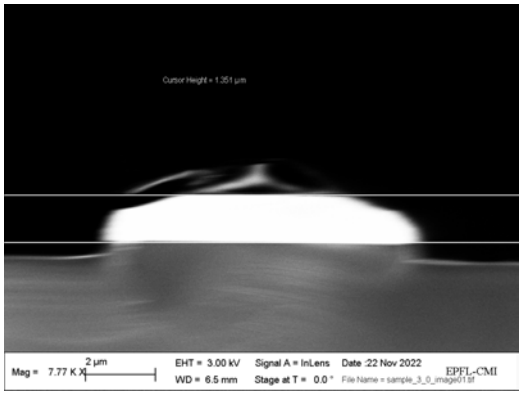


(c) Sample 2.9

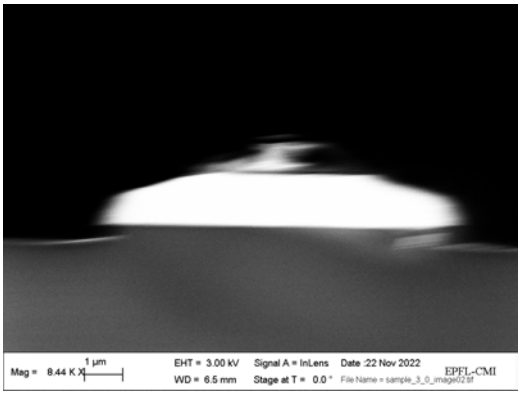


(d) Sample 2.9

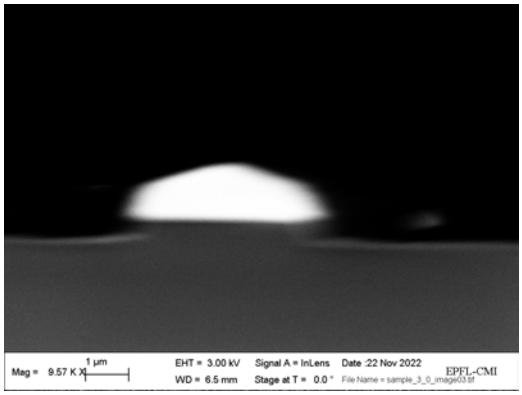
F.12 Sample 3.0



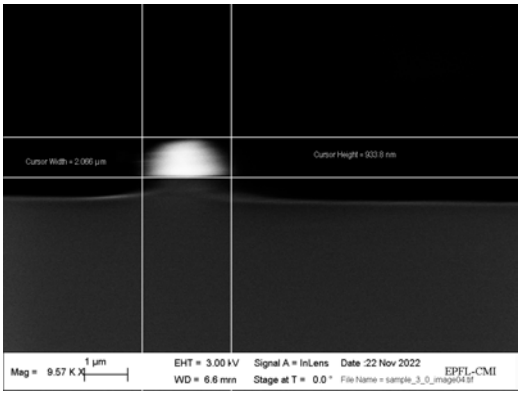
(a) Sample 3.0



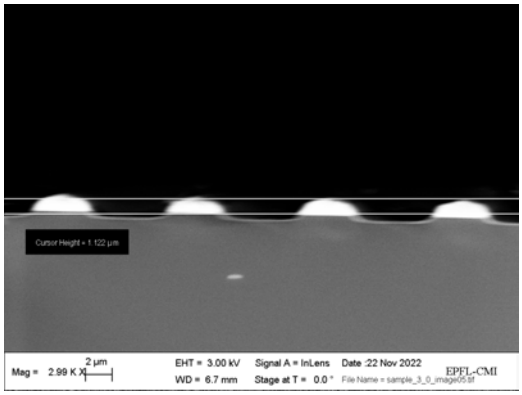
(b) Sample 3.0



(c) Sample 3.0



(d) Sample 3.0



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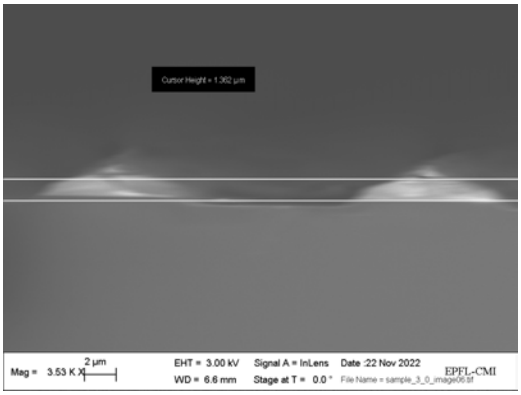
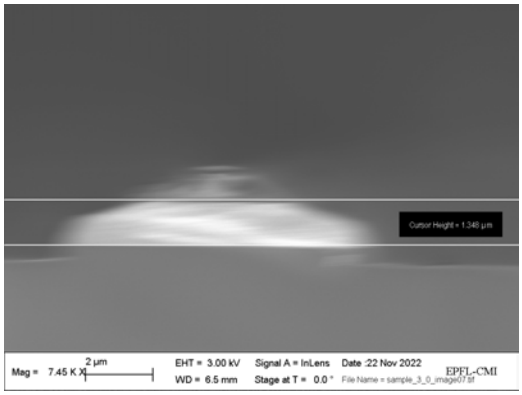
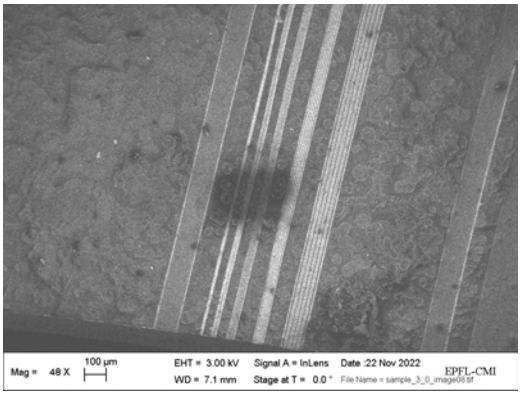


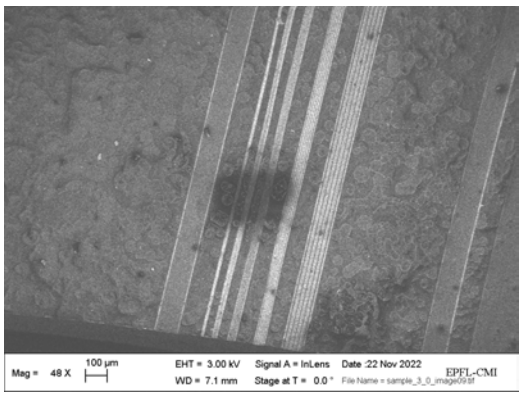
(f) Sample 3.0



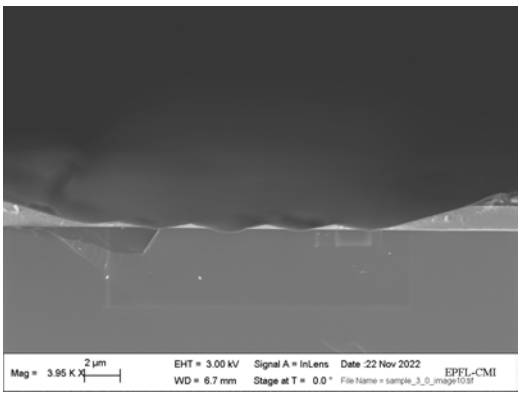
(g) Sample 3.0



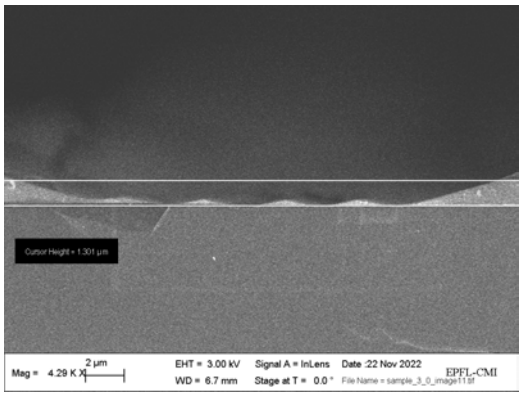
(h) Sample 3.0



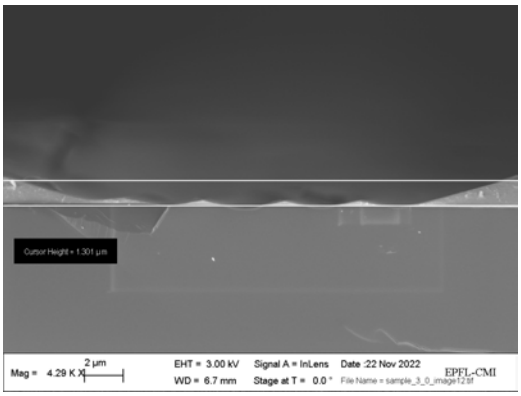
(a) Sample 3.0



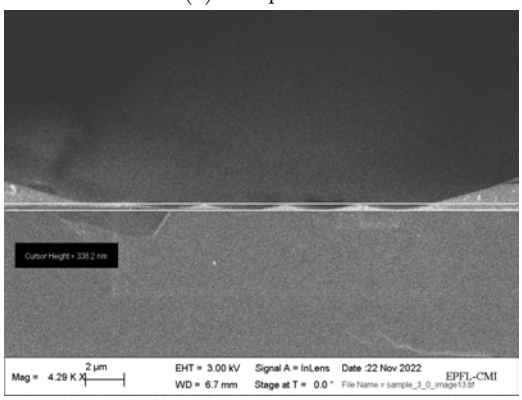
(b) Sample 3.0



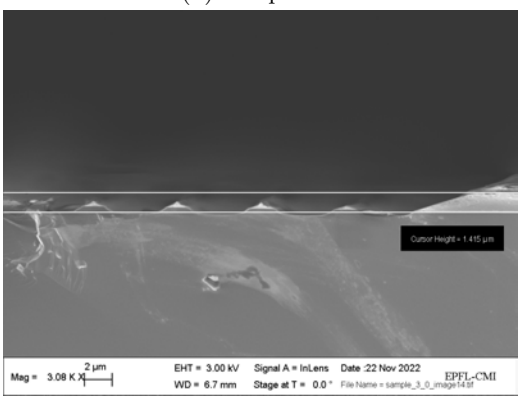
(c) Sample 3.0



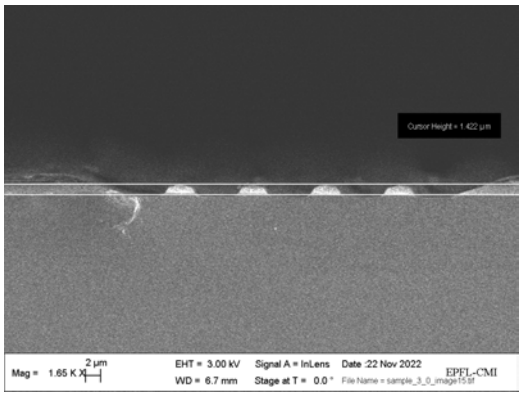
(d) Sample 3.0



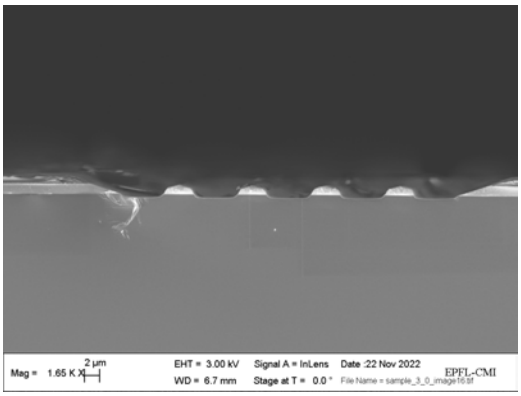
(e) Sample 3.0



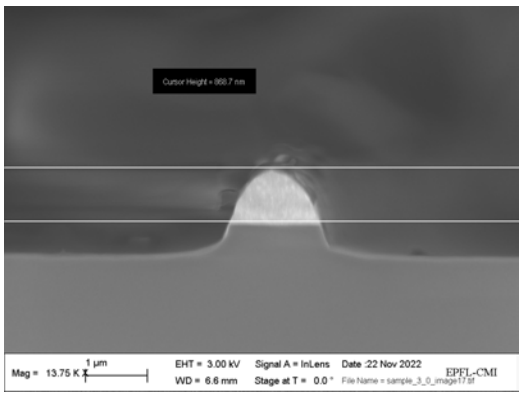
(f) Sample 3.0



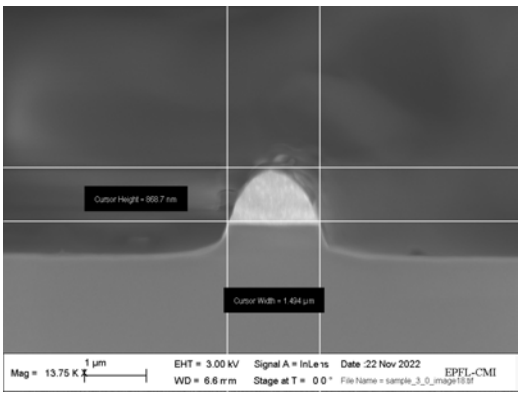
(g) Sample 3.0



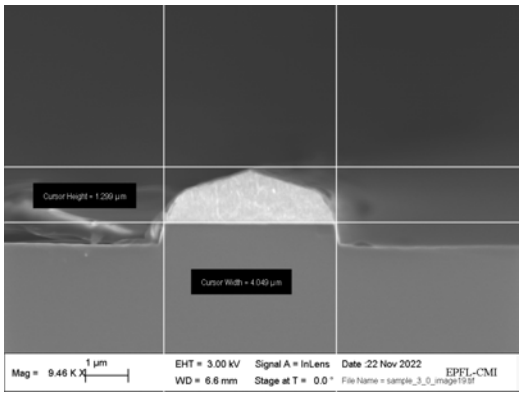
(h) Sample 3.0



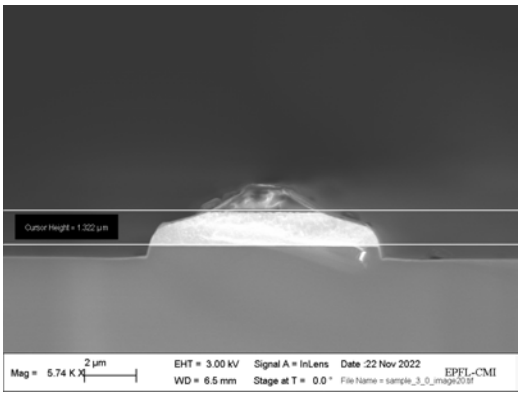
(a) Sample 3.0



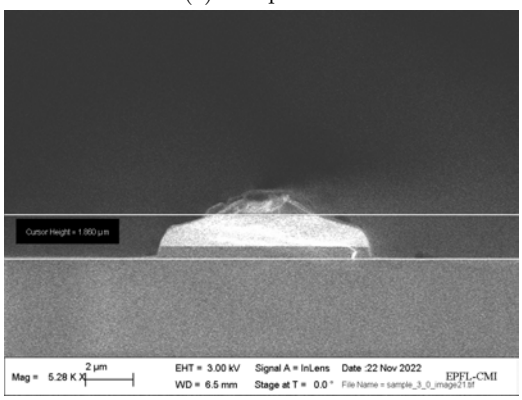
(b) Sample 3.0



(c) Sample 3.0



(d) Sample 3.0



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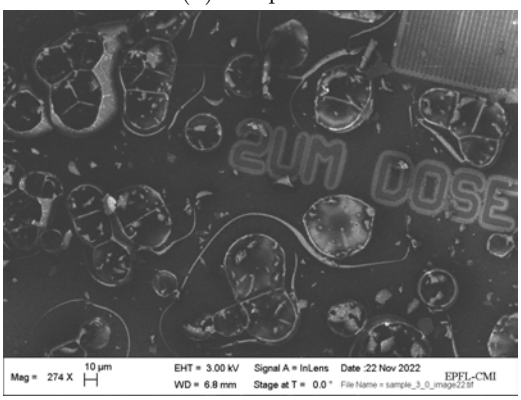
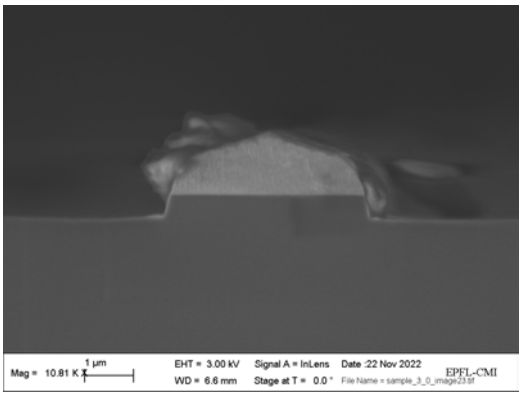


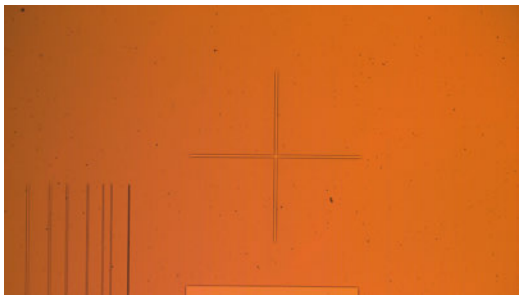
(f) Sample 3.0



(g) Sample 3.0

Optical Microscope Images

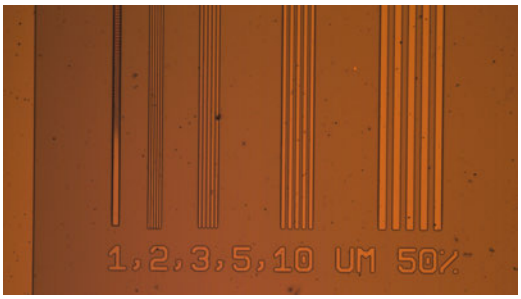
G.0.1 Sample 2.2



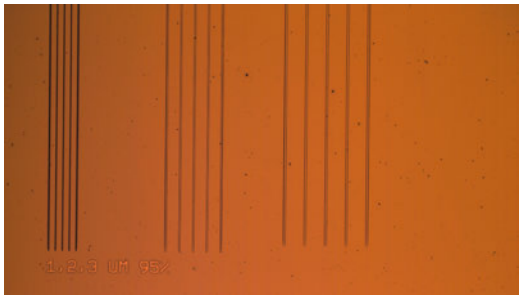
(a) Sample 2.2 before IBE

(b) Sample 2.2 before IBE

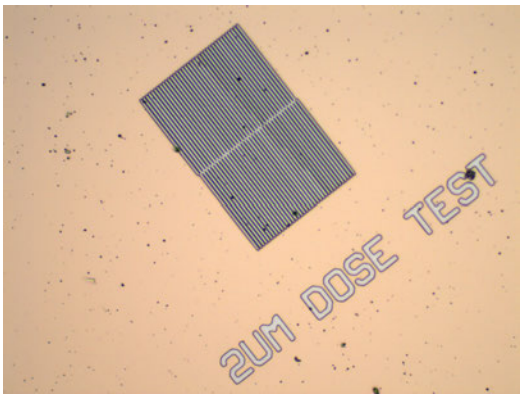
(c) Sample 2.2 before IBE



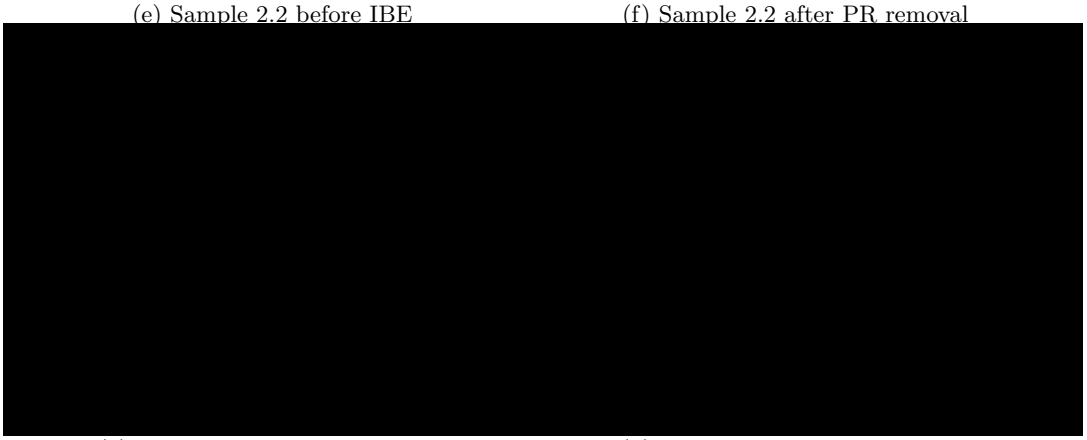
(d) Sample 2.2 before IBE



(e) Sample 2.2 before IBE



(f) Sample 2.2 after PR removal

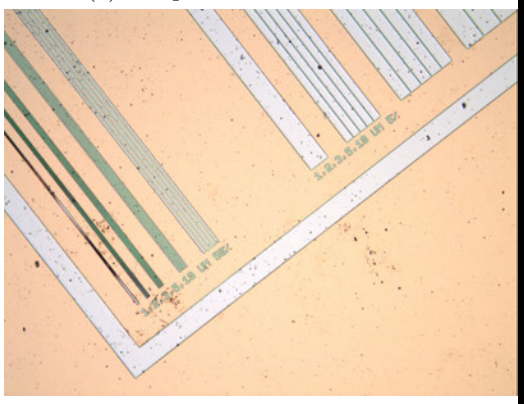


(g) Sample 2.2 after PR removal

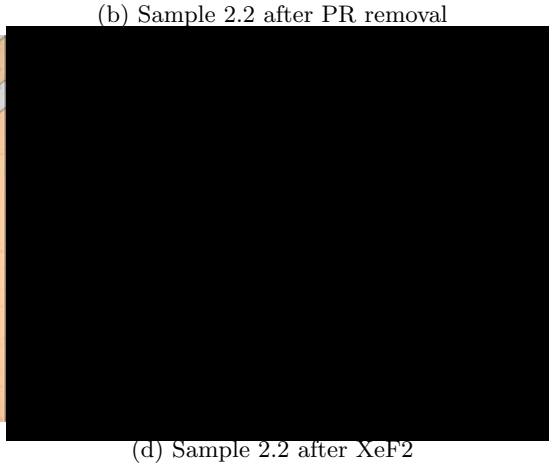
(h) Sample 2.2 after PR removal

(a) Sample 2.2 after PR removal

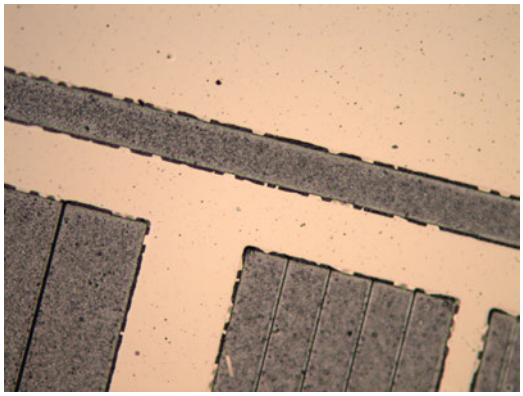
(b) Sample 2.2 after PR removal



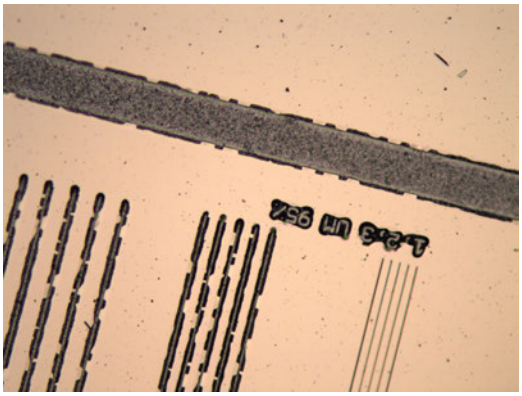
(c) Sample 2.2 after PR removal



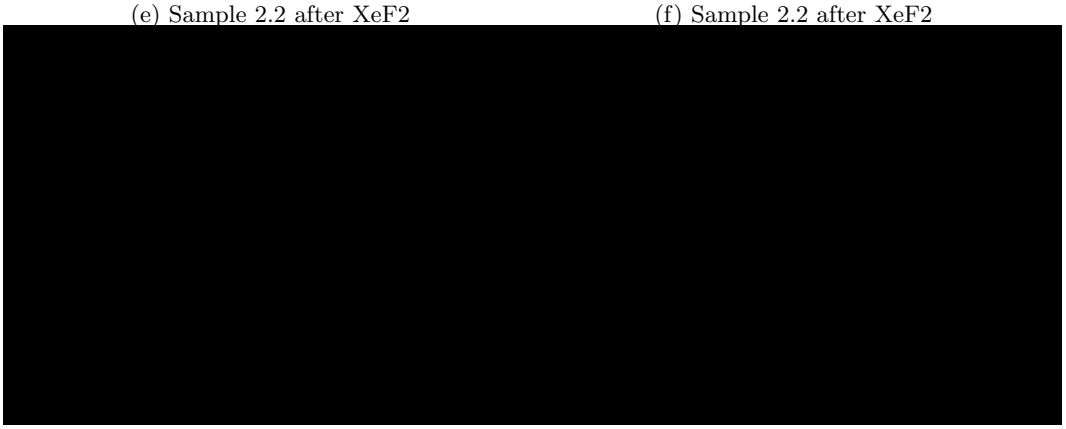
(d) Sample 2.2 after XeF2



(e) Sample 2.2 after XeF2



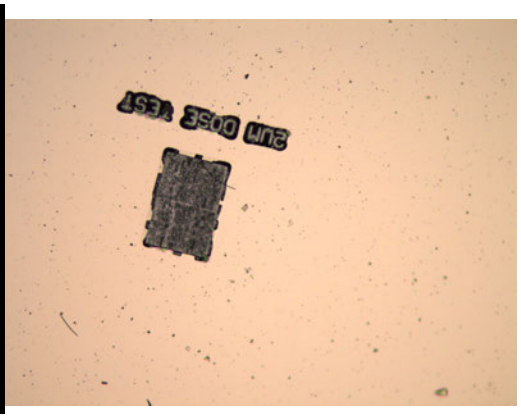
(f) Sample 2.2 after XeF2



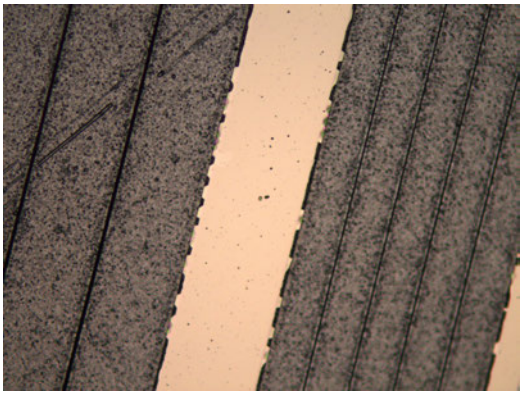
(g) Sample 2.2 after XeF2

(h) Sample 2.2 after XeF2

(a) Sample 2.2 after XeF2

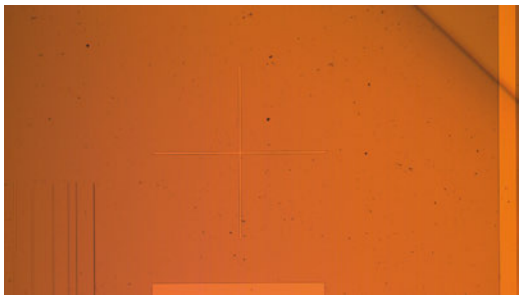


(b) Sample 2.2 after XeF2

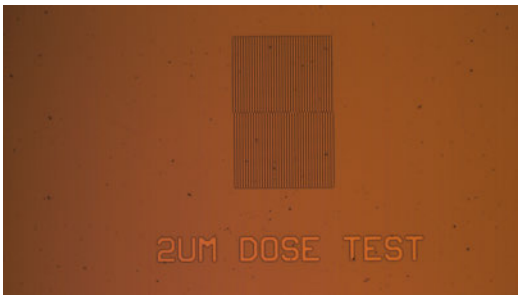


(c) Sample 2.2 after XeF2

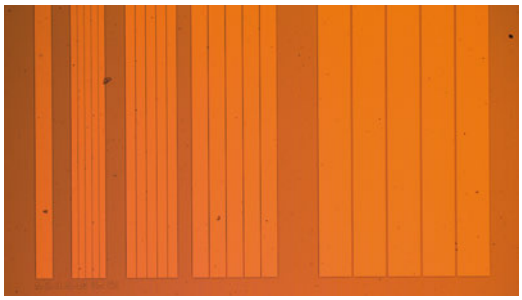
G.0.2 Sample 2.3



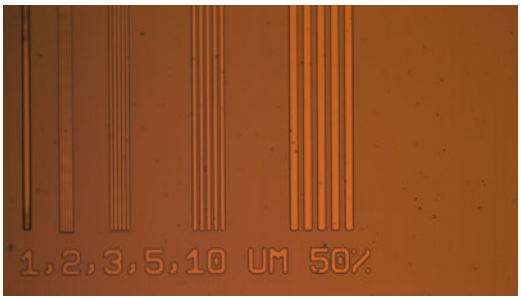
(a) Sample 2.3 before IBE



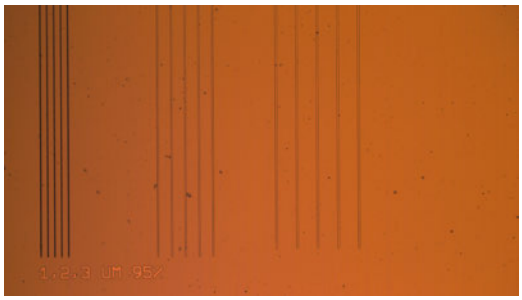
(b) Sample 2.3 before IBE



(c) Sample 2.3 before IBE

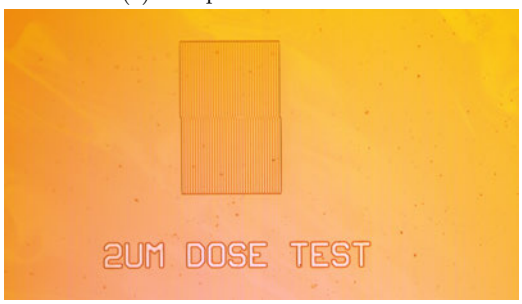


(d) Sample 2.3 before IBE

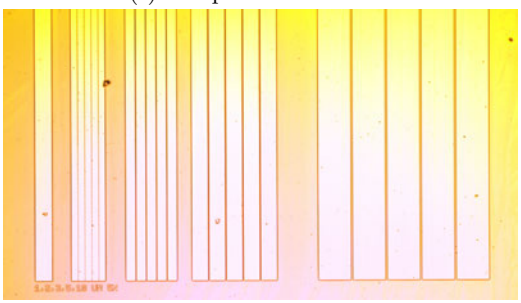


(e) Sample 2.3 before IBE

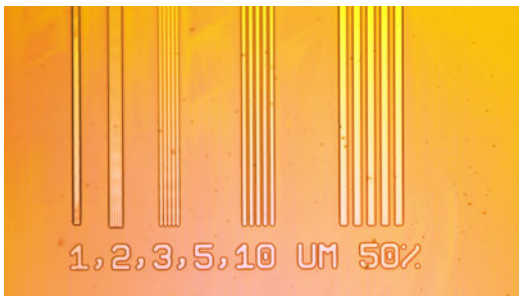
(f) Sample 2.3 after IBE



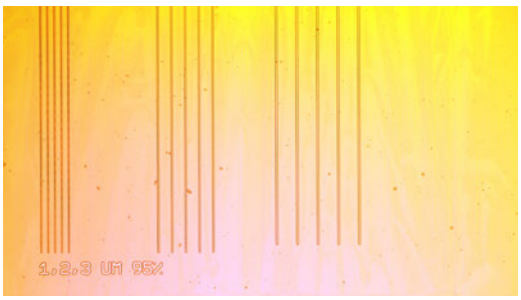
(g) Sample 2.3 after IBE



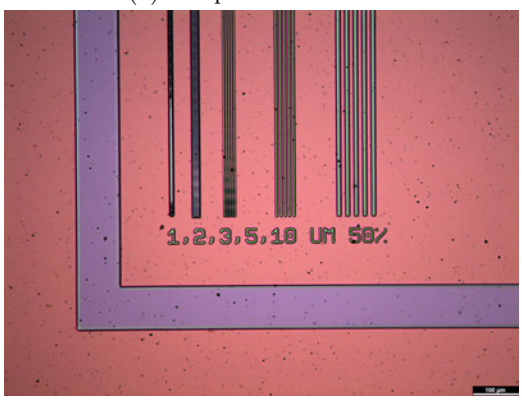
(h) Sample 2.3 after IBE



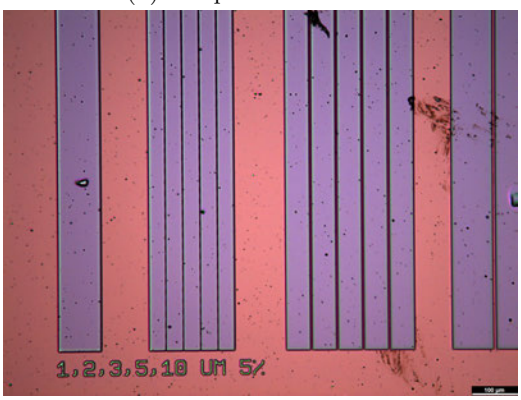
(a) Sample 2.3 after IBE



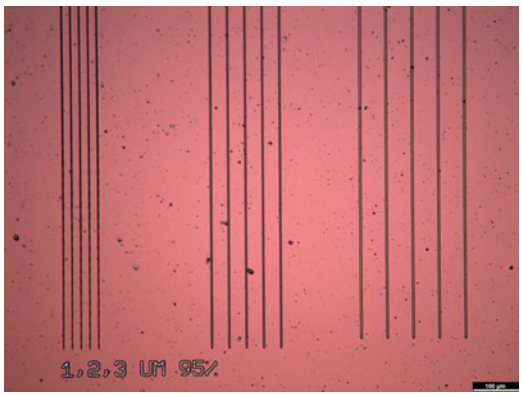
(b) Sample 2.3 after IBE



(c) Sample 2.3 after PR removal



(d) Sample 2.3 after PR removal



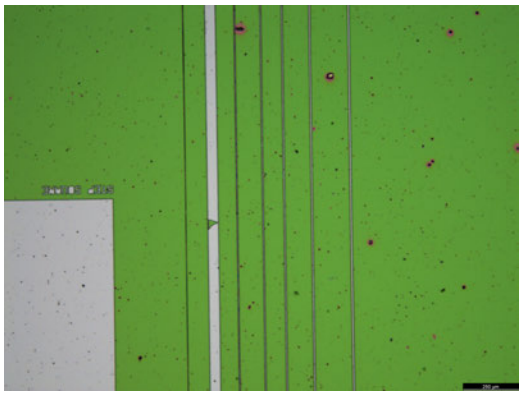
(e) Sample 2.3 after PR removal

(f) Sample 2.3 before SEM

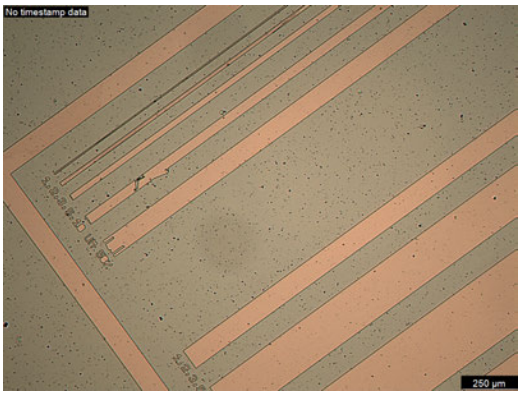
G.0.3 Sample 2.4

(a) Sample 2.4 after IBE

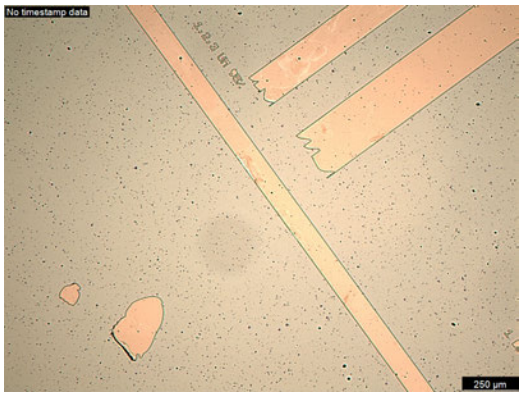
(b) Sample 2.4 after IBE



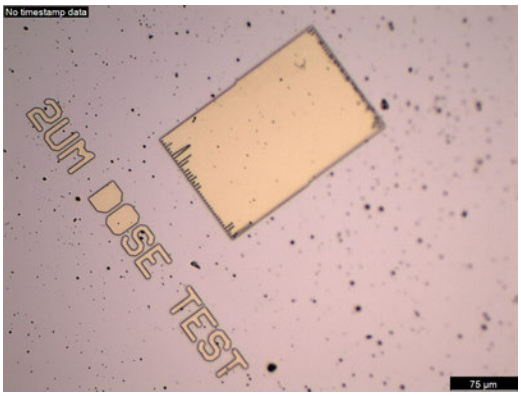
(c) Sample 2.4 after IBE



(d) Sample 2.4 after plasma

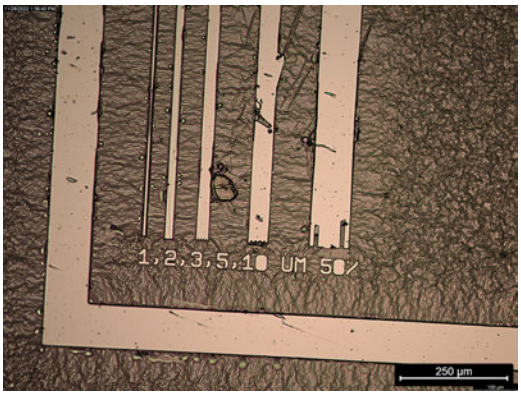


(e) Sample 2.4 after plasma

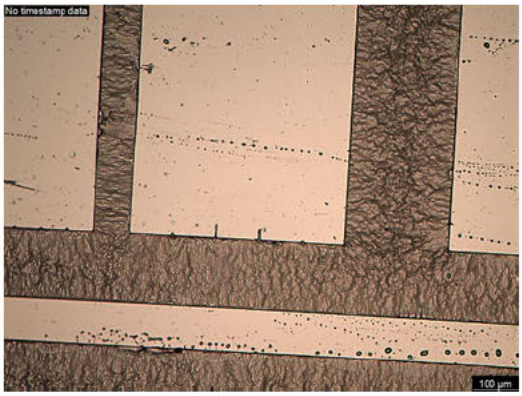


(f) Sample 2.4 after plasma

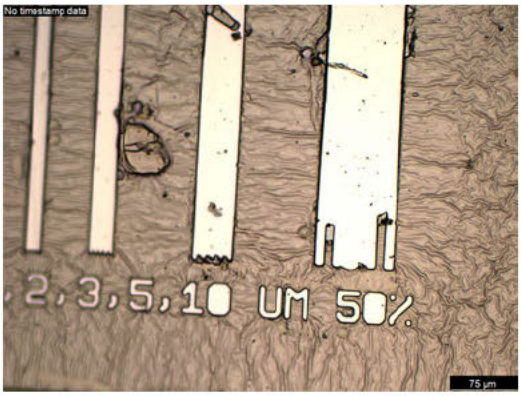
(g) Sample 2.4 after remover



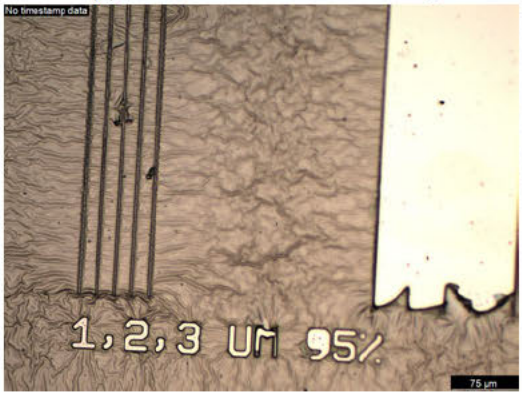
(h) Sample 2.4 after SiO etching



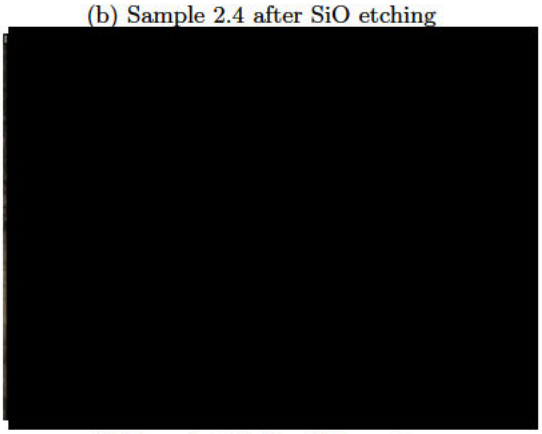
(a) Sample 2.4 after SiO etching



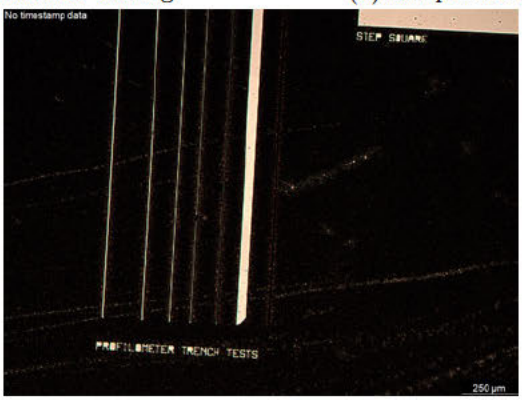
(b) Sample 2.4 after SiO etching



(c) Sample 2.4 after SiO etching

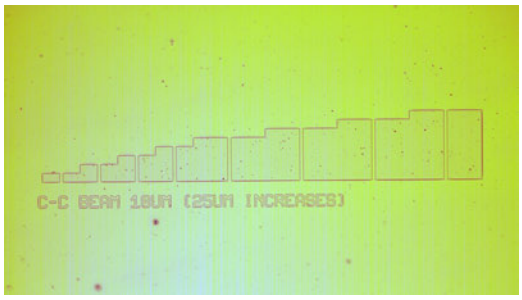


(d) Sample 2.4 after SiO etching

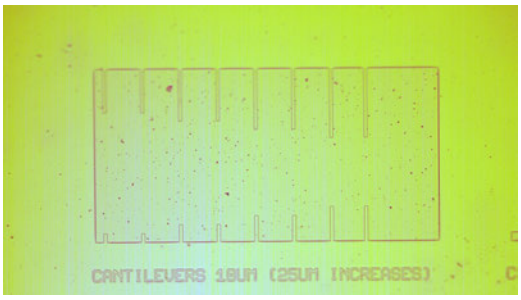


(e) Sample 2.4 after SiO etching

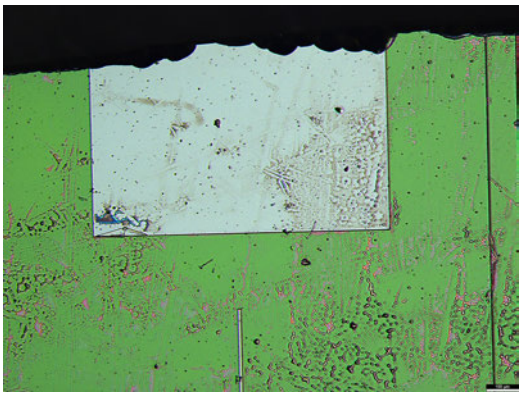
G.0.4 Sample 2.5



(a) Sample 2.5 after PL

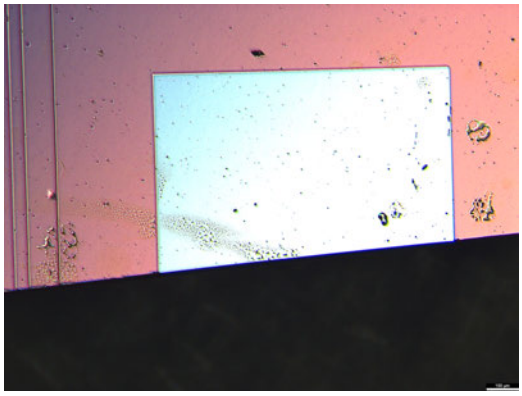


(b) Sample 2.5 after PL

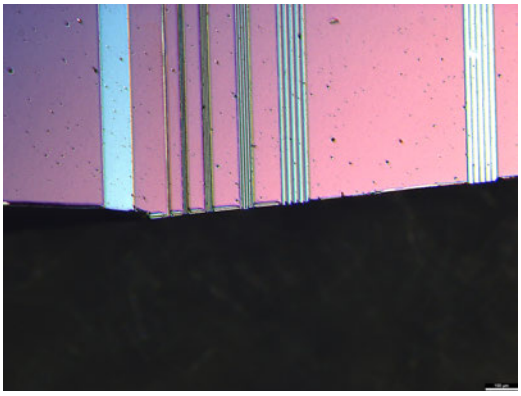


(c) Sample 2.5 after SEM

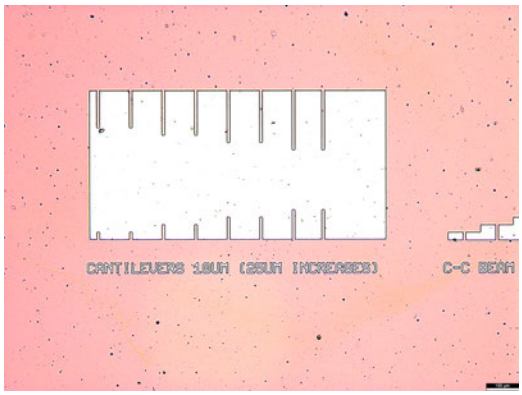
G.0.5 Sample 2.6



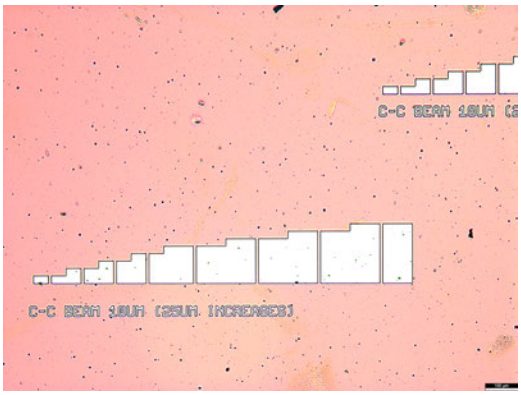
(a) Sample 2.6 after cleaving



(b) Sample 2.6 after cleaving

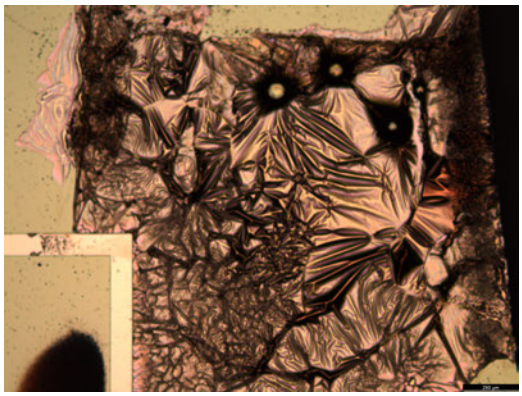


(c) Sample 2.6 after cleaving

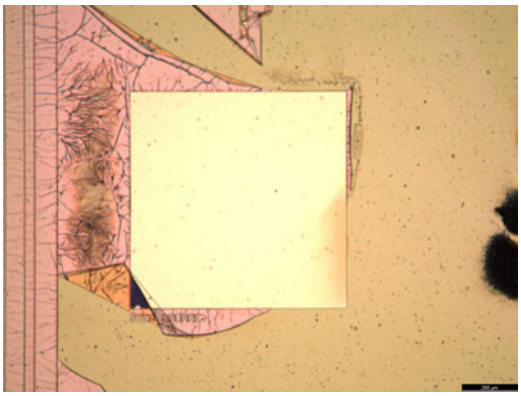


(d) Sample 2.6 after cleaving

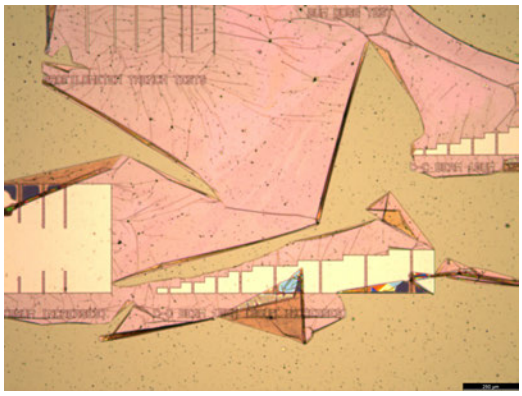
G.0.6 Sample 2.7



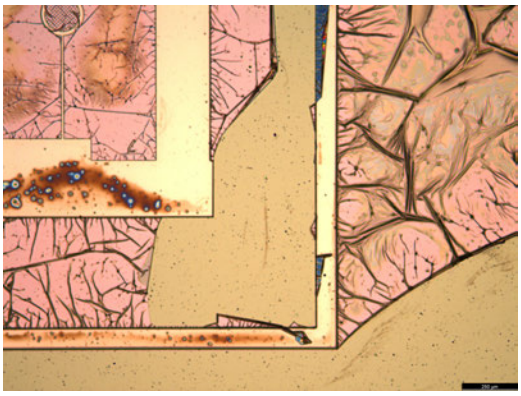
(a) Sample 2.7 before plasma



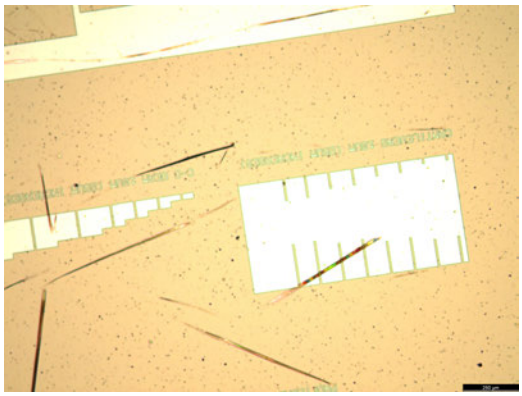
(b) Sample 2.7 before plasma



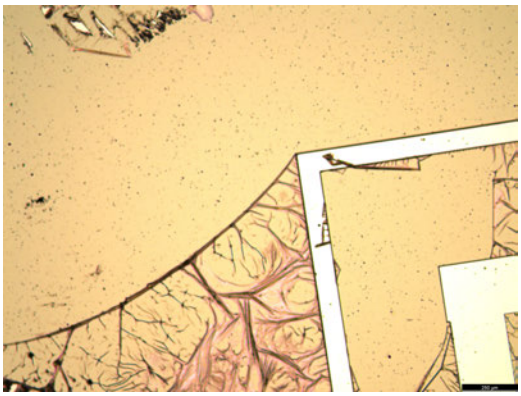
(c) Sample 2.7 before plasma



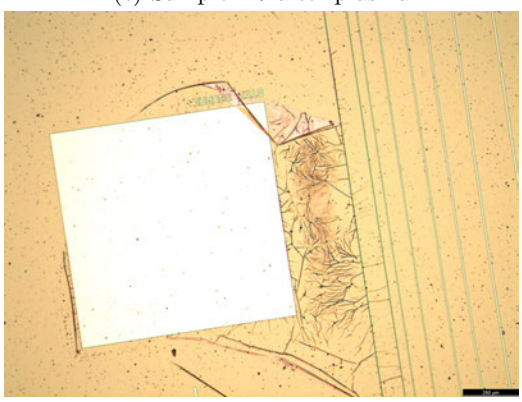
(d) Sample 2.7 before plasma



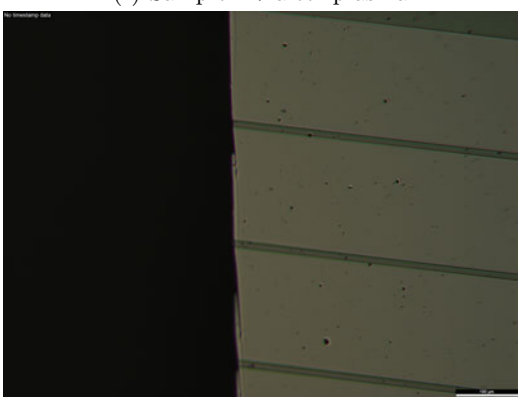
(e) Sample 2.7 after plasma



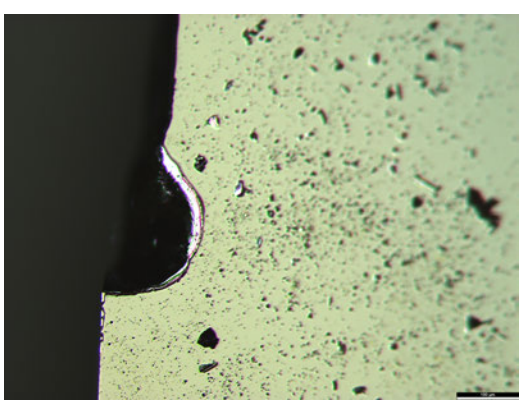
(f) Sample 2.7 after plasma



(g) Sample 2.7 after plasma



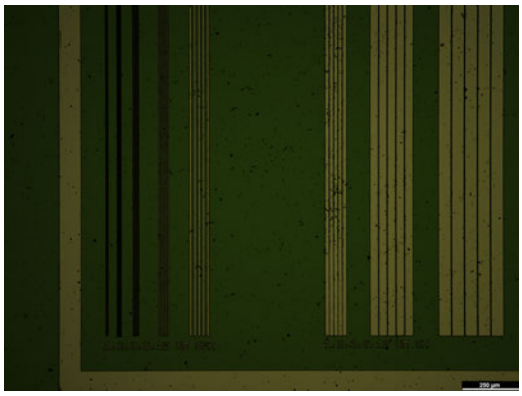
(h) Sample 2.7 after cleaving



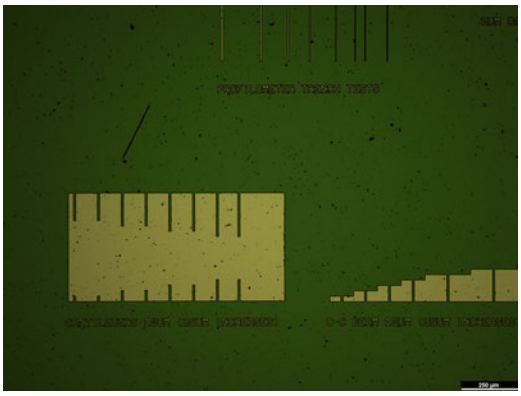
(a) Sample 2.7 after cleaving

(b) Sample 2.7 after cleaving

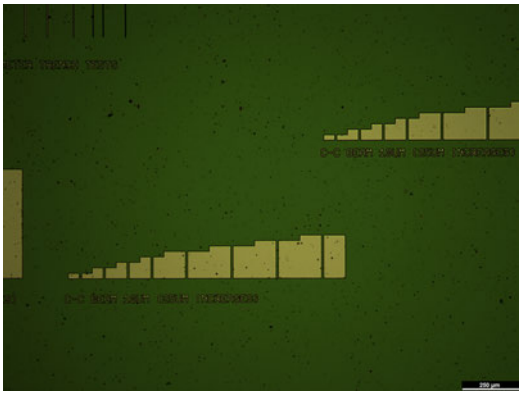
G.0.7 Sample 2.8



(a) Sample 2.8 after IBE

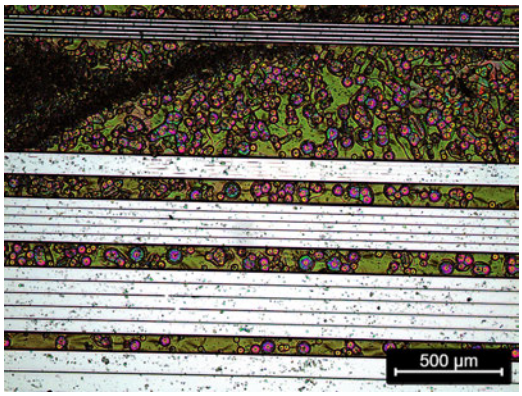


(b) Sample 2.8 after IBE

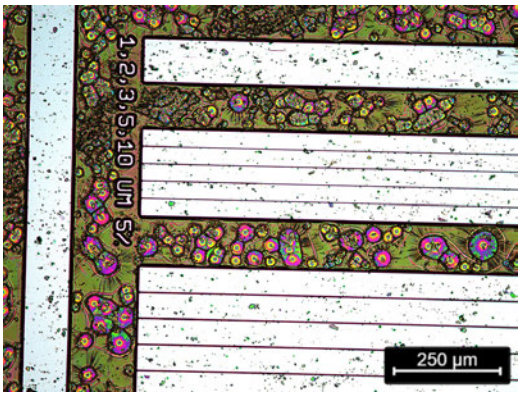


(c) Sample 2.8 after IBE

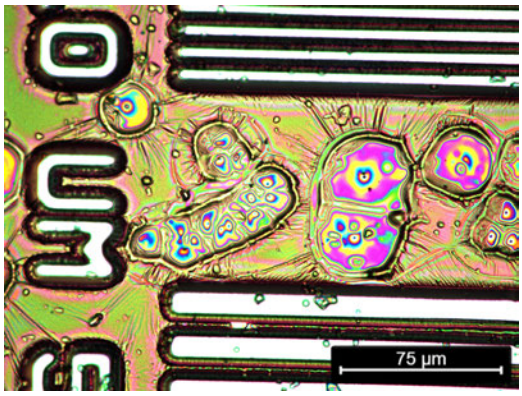
G.0.8 Sample 3.0



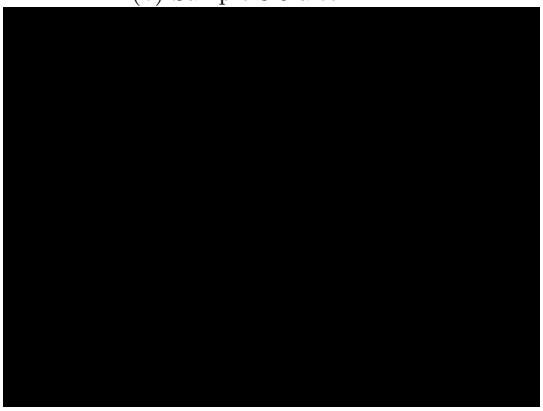
(a) Sample 3.0 after IBE



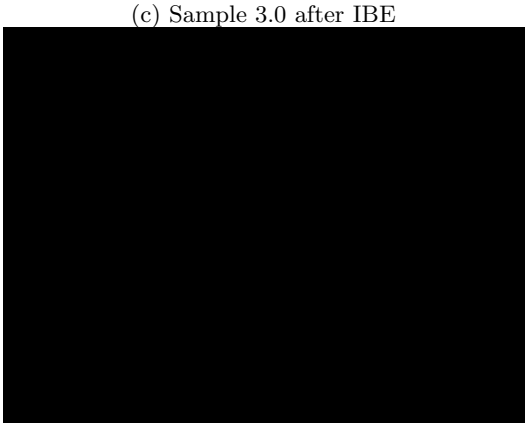
(b) Sample 3.0 after IBE



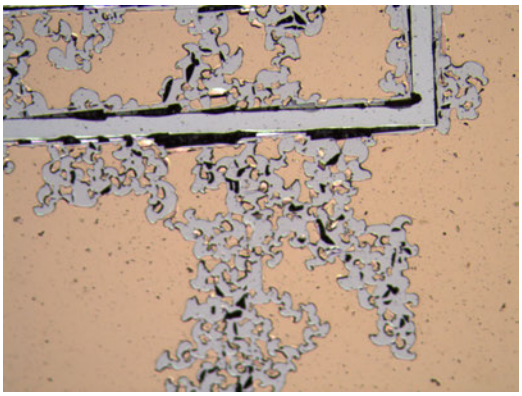
(c) Sample 3.0 after IBE



(d) Sample 3.0 after IBE



(e) Sample 3.0 after PR removal



(f) Sample 3.0 after PR removal

Profilometer Measurements

H.1 Sample 2.3

H.1.1 After PR Removal

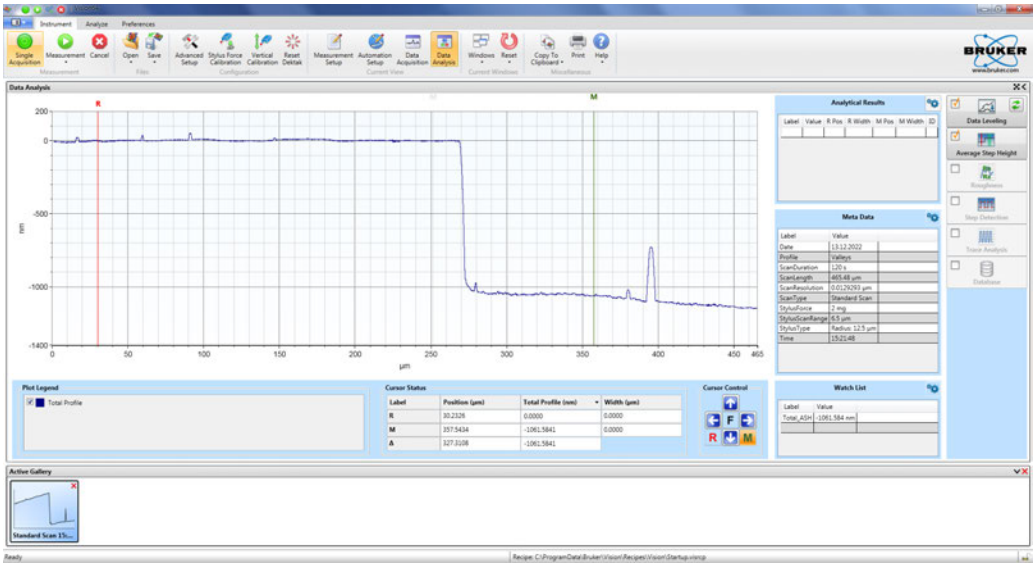


Figure H.1: DekTak Measurement of the step square on sample 2.3

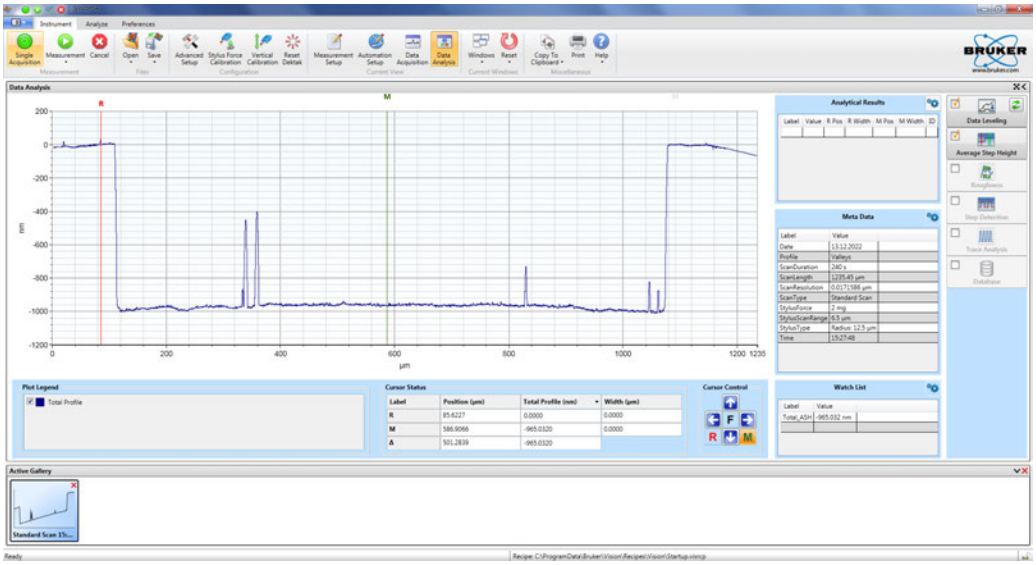


Figure H.2: DekTak Measurement of the step square on sample 2.3

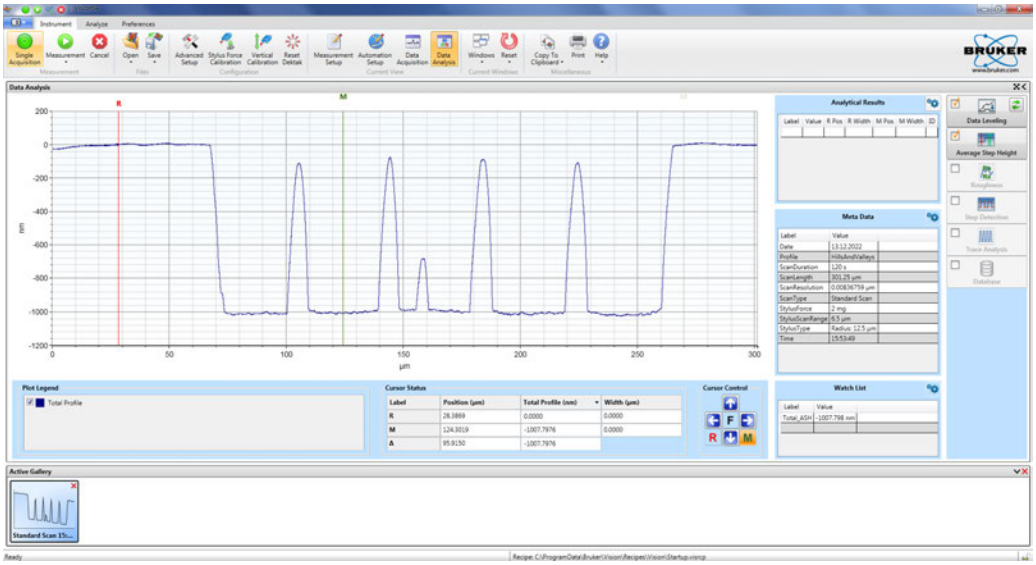


Figure H.3: DekTak Measurement of the $2\mu m$ walls on sample 2.3

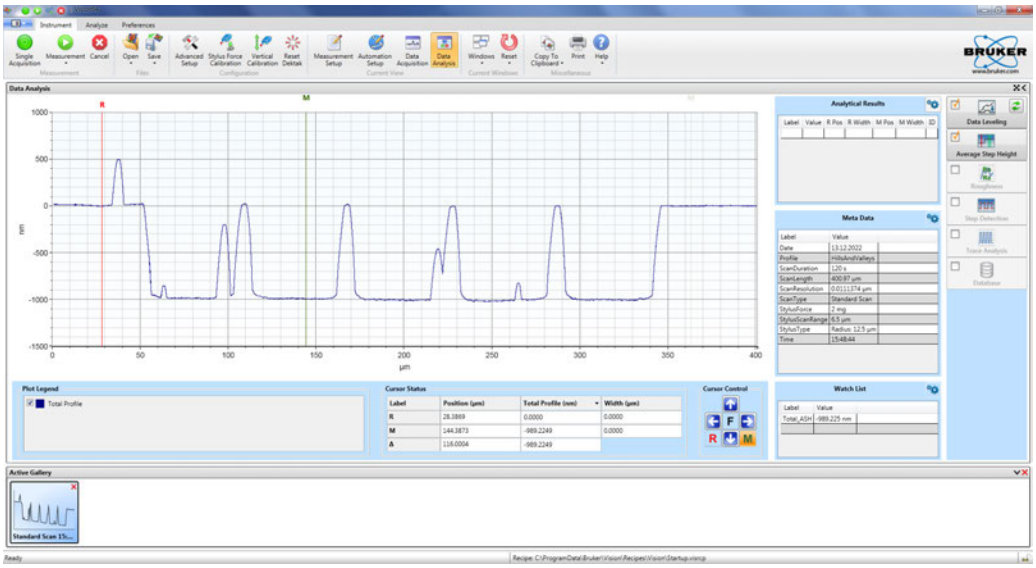


Figure H.4: DekTak Measurement of the $3\mu m$ walls on sample 2.3

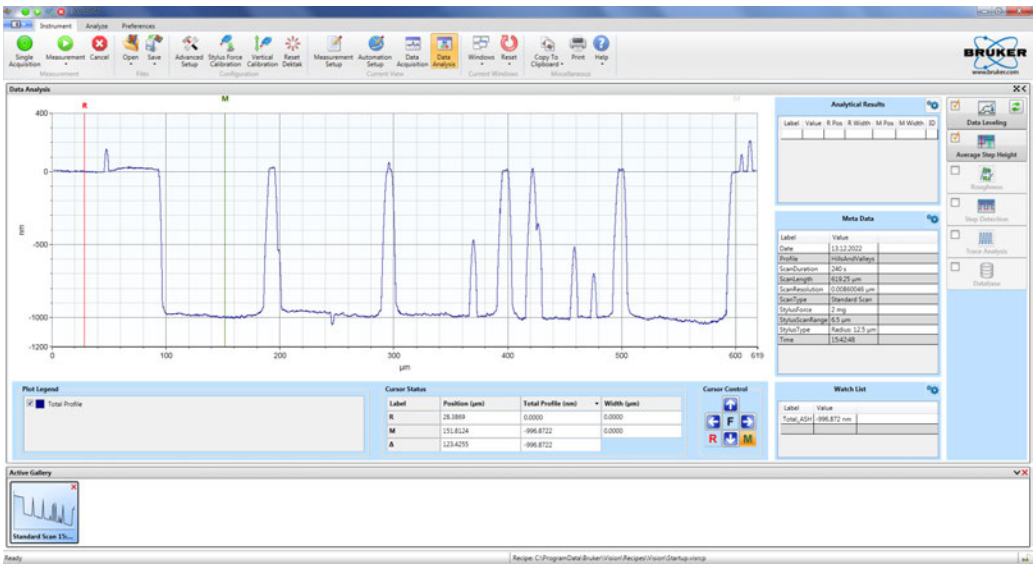


Figure H.5: DekTak Measurement of the $5\mu m$ walls on sample 2.3

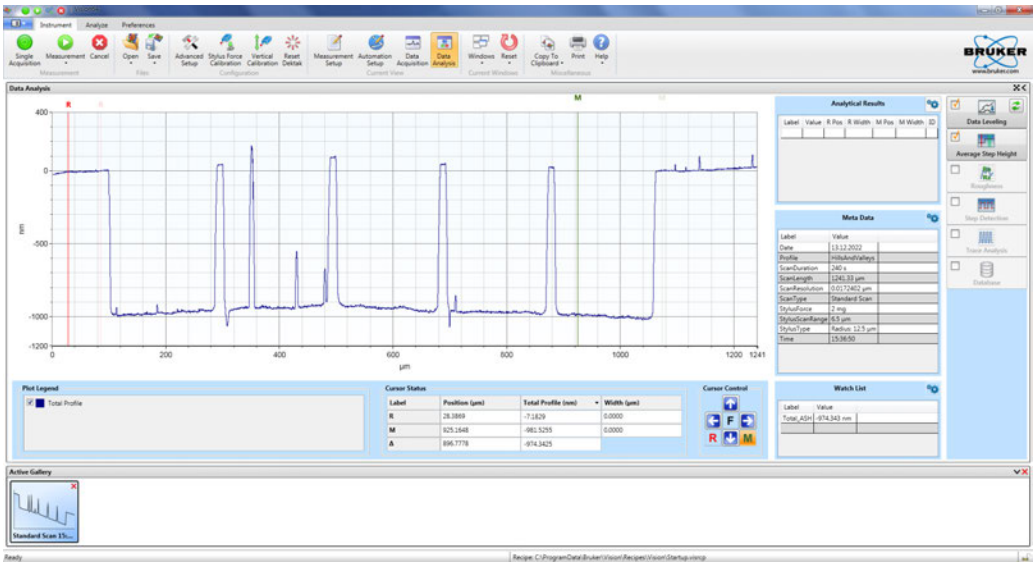


Figure H.6: DekTak Measurement of the $10\mu m$ walls on sample 2.3

H.2 Sample 2.4

H.2.1 After IBE and SEM

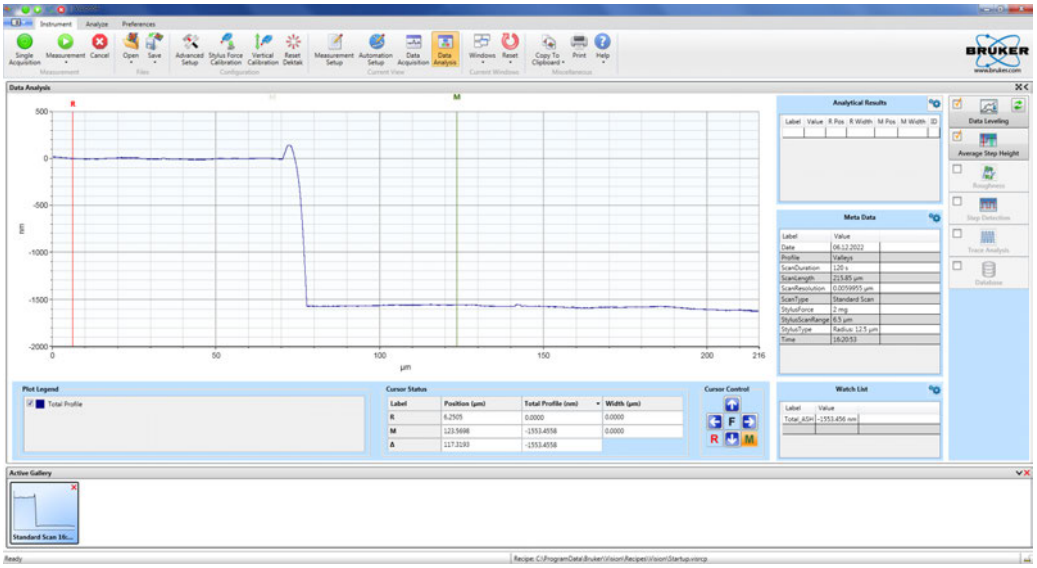


Figure H.7: DekTak Measurement of the step square on sample 2.4

H.3 Sample 2.5

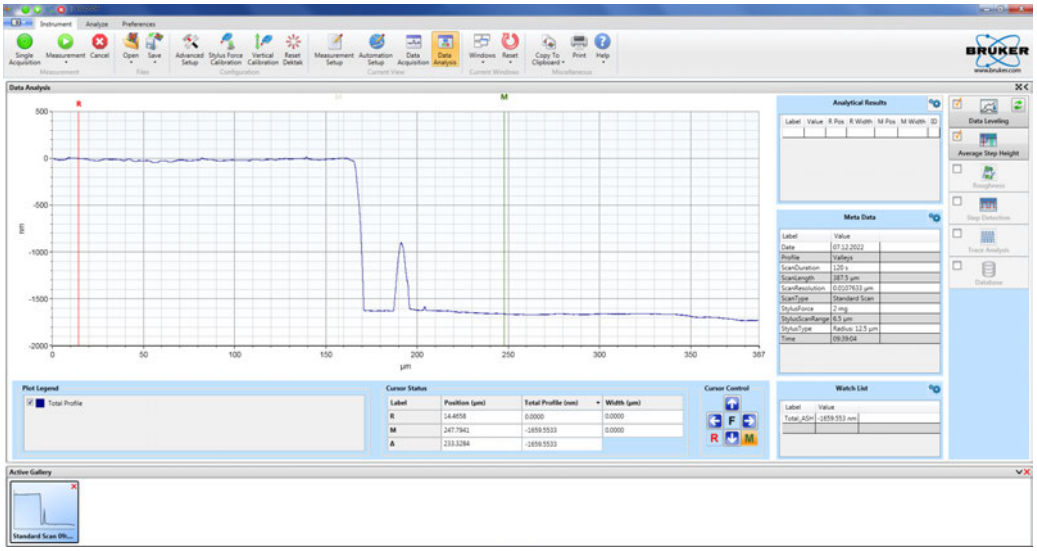


Figure H.8: DekTak Measurement of the step square on sample 2.5

Figure H.9: DekTak Measurement of the lines at 50% on sample 2.5

H.4 Sample 2.6

H.4.1 After SiO Etching

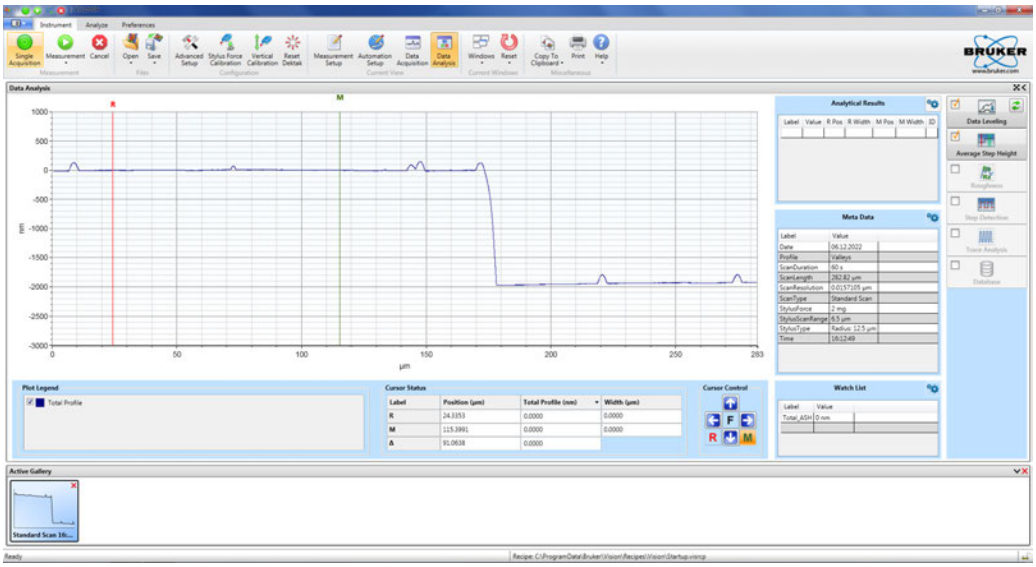


Figure H.10: DekTak Measurement of the stepsquare on sample 2.6 after etching the SiO hardmask

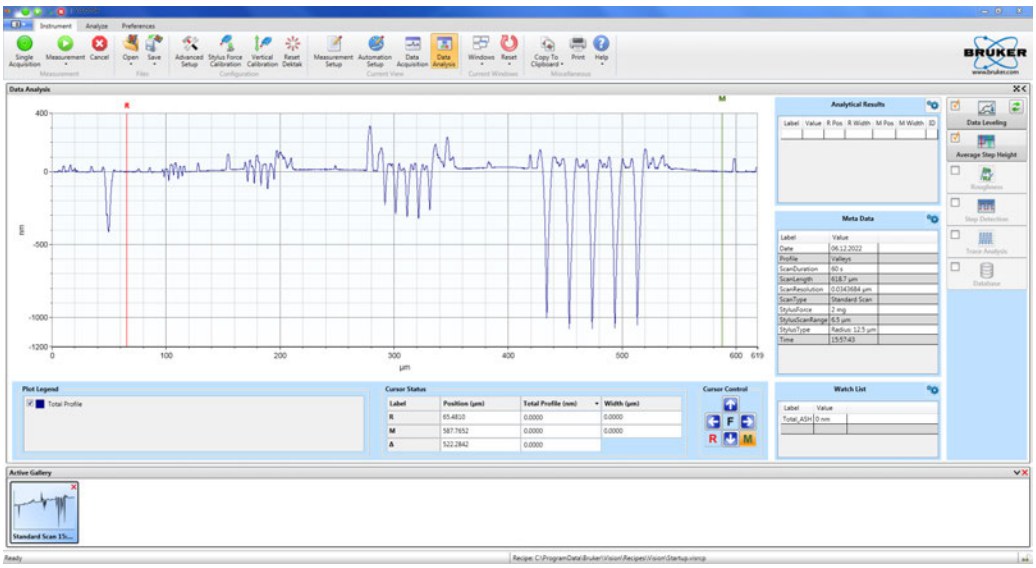


Figure H.11: DekTak Measurement of the lines at 50% on sample 2.6

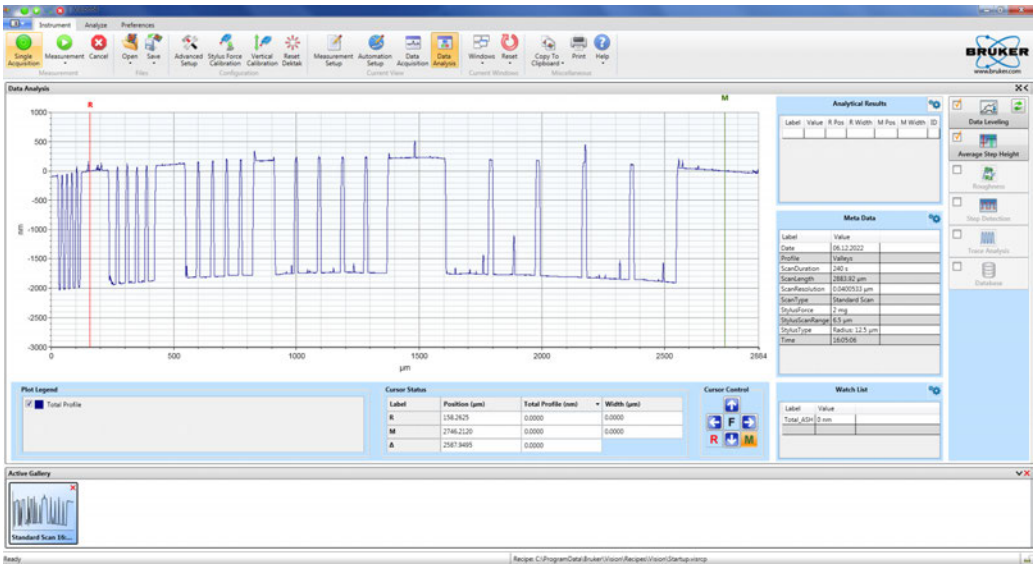


Figure H.12: DekTak Measurement of the lines at 95% on sample 2.6

H.4.2 After IBE

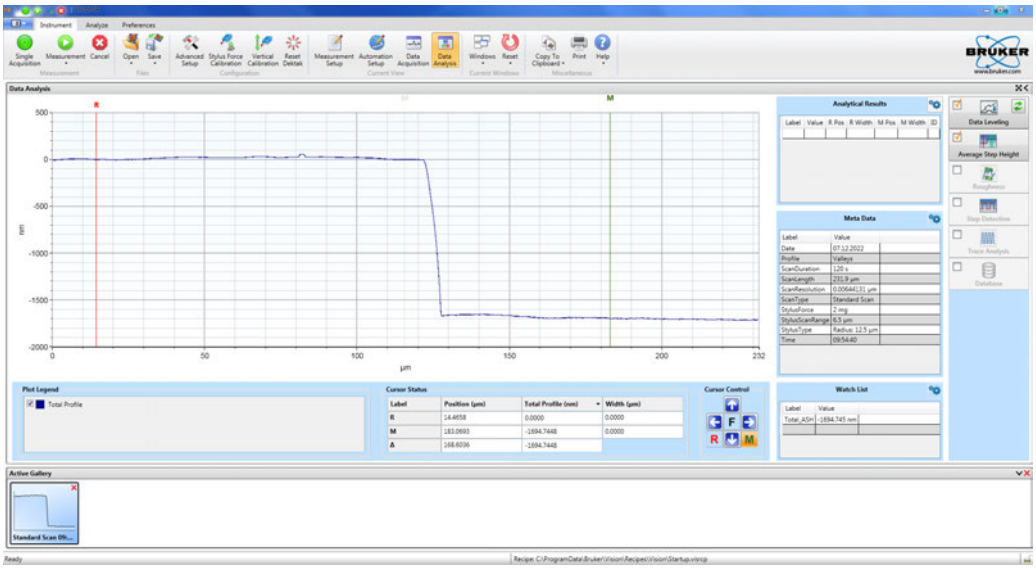


Figure H.13: DekTak Measurement of stepsquare on sample 2.6 after IBE

Machines Used

The following table lists all the fabrication machines used in CMi. The link to the detailed infos on CMi's website is provided for each machine.

Machine Name	Location	Website
ATMsse SB20	Zone 13	https://www.epfl.ch/research/facilities/cmi/equipment/photolithography/sse-sb20/
SPTS APS	Zone 2	https://www.epfl.ch/research/facilities/cmi/equipment/etching/spts-aps/
Heidelberg Instruments MLA150	Zone 16	https://www.epfl.ch/research/facilities/cmi/equipment/photolithography/mla-150/
Tepla 300	Zone 11	https://www.epfl.ch/research/facilities/cmi/equipment/etching/tepla-300/
Tepla GiGAbatch	Zone 2	https://www.epfl.ch/research/facilities/cmi/equipment/etching/
SPTS uEtch	Zone 2	https://www.epfl.ch/research/facilities/cmi/equipment/etching/spts-uetch/
SPTS Xactix X4	Zone 11	https://www.epfl.ch/research/facilities/cmi/equipment/etching/xef2-silicon-etching-system/
Veeco Nexus IBE350	Zone 11	https://www.epfl.ch/research/facilities/cmi/equipment/etching/veeco-nexus-ibe350/

Note: All URLs accessed 4.1.2023

Process Flows

On the next pages one can find the process flows used during this project in the form they have been validated by the CMi technical committee.

Lab : EPFL STI IGM NEMS
Operator Name : Fabian Bauer
Supervisor Name : Marco Liffredo
Phone : +41 79 938 45 06
Office : -
E-mail : fabian.bauer@epfl.ch

CMi EPFL Center of MicroNanoTechnology

☒ Semestral Project ☐ Master Project ☐ Thesis ☐ Other

Project Name

Description of the fabrication project

The goal of this project is to study the etching of a HfC (hafnium carbide) thin film using the Veeco Ion Beam Etcher. The parameter of interest at this time is: the etch rate.
This is the first step in determining the viability of a bigger manufacturing project.

Technologies used
Ion Beam Etching, Thin-Film Analyzer, Photolithography

Ebeam litho data - Photolitho masks - Laser direct write data			
Mask #	**Critical Dimension**	**Critical Alignment**	**Remarks**
1	> **1mm**	>1 mm	Mask to allow for only one quarter of the waver to be used per test.

Substrate Type
Silicon with HfC (hafnium carbide) thin film already on it. Different wafers have different thicknesses ranging from 100-2000nm.

Interconnections and packaging of final device

Thinning/grinding/polishing of the samples is required at some stage of the process.
☑ No ☐ Yes => confirm involved materials with CMi staff

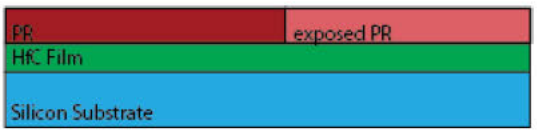
Dicing of the samples is required at some stage of the process.
☑ No ☐ Yes => confirm dicing layout with CMi staff

Wire-bonding of dies, with glob-top protection, is required at the end of the process.
☑ No ☐ Yes => confirm pads design (size, pitch) and involved materials with CMi staff

Lab : EPFL STI IGM NEMS
Operator Name : Fabian Bauer
Supervisor Name : Marco Liffredo
Phone : +41 79 938 45 06
Office : -
E-mail : fabian.bauer@epfl.ch

CMi EPFL Center of MicroNanoTechnology

Step-by-step process outline

Step	Process description	Cross-section after process
01	Measurement of HfC film thickness Instrument: FilMetrics F20-UV	
02	Photolitography Coating Machine: ACS200 Resist : ECI3027 Target Thickness: 2μm	
03	Photolitography Exposure Machine: Süss MicroTec MA6 gen3 Mask : #1	
04	Photolit Development Machine: ACS200 Developer: MF CD 26	
05	Measurement of PR film thickness Instrument: FilMetrics F20-UV	
06	Ion Beam Etching Machine: Veeco Nexus IBE350 Power: Medium	
07	Measurement of HfC and PR film thickness Instrument: FilMetrics F20-UV	

08	Photolit Stripp Off O2 plasma 1min in Tepla + remover 1165 in UFT wet bench Z2	
09	Measurement of step size Instrument: Bruker Dektak XT	

Lab : EPFL STI IGM NEMS
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Supervisor Name : Marco Liffredo

Phone : +41 79 938 45 06
Office : -
E-mail : fabian.bauer@epfl.ch

CMi EPFL Center of MicroNanoTechnology

☒ Semestral Project ☐ Master Project ☐ Thesis ☐ Other

Project Name

Description of the fabrication project

The goal of this project is to study the etching of a HfC (hafnium carbide) thin film using the Veeco Ion Beam Etcher. The parameter of interest at this time is: the etch rate.
This is the first step in determining the viability of a bigger manufacturing project.

Technologies used
Ion Beam Etching, Thin-Film Analyzer, Photolithography

Ebeam litho data - Photolitho masks - Laser direct write data			
Mask #	**Critical Dimension**	**Critical Alignment**	**Remarks**
1	> **1mm**	>1 mm	Test mask

Substrate Type
Chips and Wafers of Silicon with HfC (hafnium carbide) thin film already on it. Different wafers/chips have different thicknesses ranging from 100-2000nm.

Interconnections and packaging of final device

Thinning/grinding/polishing of the samples is required at some stage of the process.
☑ No ☐ Yes => confirm involved materials with CMi staff

Dicing of the samples is required at some stage of the process.
☒ Yes => confirm dicing layout with CMi staff

Wire-bonding of dies, with glob-top protection, is required at the end of the process.
☑ No ☐ Yes => confirm pads design (size, pitch) and involved materials with CMi staff

Lab : EPFL STI IGM NEMS
Operator Name : Fabian Bauer
Supervisor Name : Marco Liffredo
Phone : +41 79 938 45 06
Office : -
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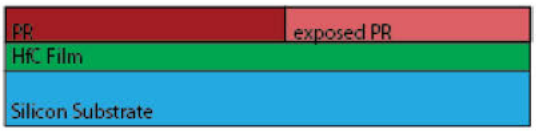
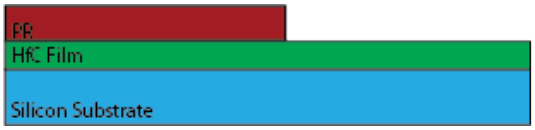
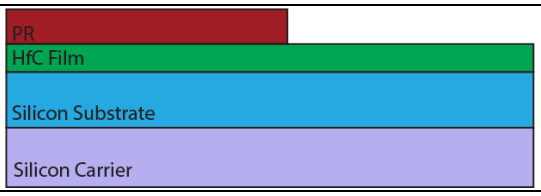
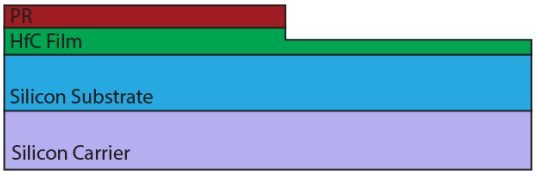
CMi EPFL Center of MicroNanoTechnology

Step-by-step process outline

Step	Process description	Cross-section after process
-03	Photolitography Coating (as protection during dicing) Machine ATMsse SB20 Resist : ECI3027 Target Thickness: 2μm	
-02	Dicing of half wafer into chips as CMi service (20mmx20mm chips from half wafer)	
-01	Remove PR remover 1165 in UFT wet bench Z2	
00	Inspection of surface texture Instrument: Zeiss MERLIN	
01	Measurement of HfC film thickness Instrument: FilMetrics F20-UV	
02	Photolitography Coating Machine ATMsse SB20 Resist : ECI3027 Target Thickness: 2μm	
03	Photolitography Exposure Machine: MLA150 Mask : #1	
04	Photolit Development Wet bench Zone 13 Developer: MF CD 26	

05	Measurement of PR film thickness Instrument: FilMetrics F20-UV	
06	Mounting of Chip to 100mm Carrier Wafer using Quick Stick Machine: Z6 – RC8 THP-Manual Coater	
07	Ion Beam Etching Machine: Veeco Nexus IBE350 Power: Medium Add Kapton tape for extra fixation	
08	Revome Chip from Carrier Machine: Z6 – RC8 THP-Manual Coater	
09	Measurement of HfC and PR film thickness Instrument: FilMetrics F20-UV	
10	Photolit Stripp Off O2 plasma 1min in Tepla + remover 1165 in UFT wet bench Z2	
11	Measurement of step size Instrument: Bruker Dektak XT	

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☒ Semestral Project ☐ Master Project ☐ Thesis ☐ Other

Project Name

Description of the fabrication project

The goal of this project is to study the etching of a HfC (hafnium carbide) thin film using the Veeco Ion Beam Etcher. The parameter of interest at this time is: the etch rate.
This is the first step in determining the viability of a bigger manufacturing project.

Technologies used
Ion Beam Etching, Thin-Film Analyzer, Photolithography

Ebeam litho data - Photolitho masks - Laser direct write data			
Mask #	**Critical Dimension**	**Critical Alignment**	**Remarks**
1	2 um	>1 mm	

Substrate Type
Chips of Silicon with HfC (hafnium carbide) thin film already on it. Different wafers/chips have different thicknesses ranging from 100-2000nm.

Interconnections and packaging of final device

Thinning/grinding/polishing of the samples is required at some stage of the process.
☑ No ☐ Yes => confirm involved materials with CMi staff

Dicing of the samples is required at some stage of the process.
☐ Yes => confirm dicing layout with CMi staff

Wire-bonding of dies, with glob-top protection, is required at the end of the process.
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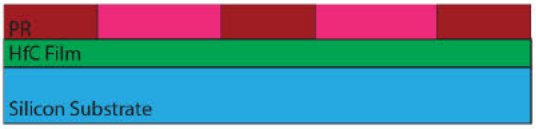
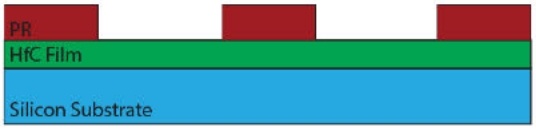
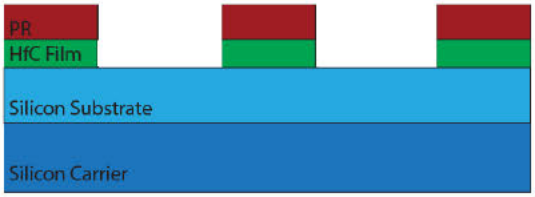
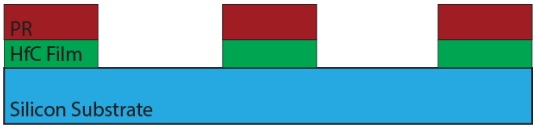
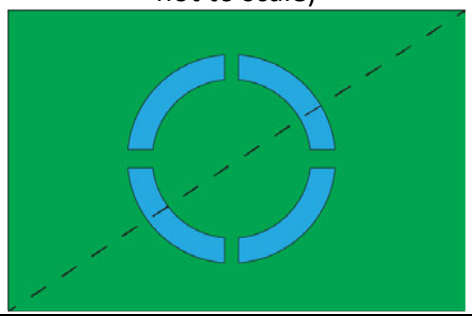
Step-by-step process outline

Step	Process description	Cross-section after process
00	Inspection of surface texture Instrument: Zeiss MERLIN	
01	Measurement of HfC film thickness Instrument: FilMetrics F20-UV	
02	Photolitography Coating Machine ATMsse SB20 Resist : ECI3027 Target Thickness: 2μm	
03	Photolitography Exposure Machine: MLA150 Mask : #1	
04	Photolit Development Wet bench Zone 13 Developer: MF CD 26	
05	Mounting of Chip to 100mm Carrier Wafer using Quick Stick Machine: Z6 – RC8 THP-Manual Coater	
06	Ion Beam Etching Machine: Veeco Nexus IBE350 Power: Medium Add Kapton tape for extra fixation	

07	Revome Chip from Carrier Machine: Z6 – RC8 THP-Manual Coater	
08	Photolit Stripp Off O2 plasma 1min in Tepla + remover 1165 in UFT wet bench Z2	
09	Release of HfC device: Etching of Si Machine: SPTS Xactix X4	 Top view (dashed line indicating cross section, not to scale) 
10	Inspection Machine: SEM Leo	

Lab : EPFL STI IGM NEMS
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Ebeam litho data - Photolitho masks - Laser direct write data			
Mask #	**Critical Dimension**	**Critical Alignment**	**Remarks**
1	2um	No alignment	-

Substrate Type
Part of a Si wafer with HfC thin film already on it: ca.50mm x50mm

Interconnections and packaging of final device

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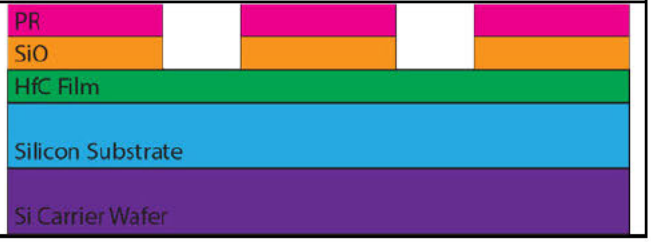
01	**Deposition of SiO** Machine: Pfeiffer SPIDER 600	
02	**Lithography: PR Coating** Machine: ATMsse SB20 Resist: AZ ECI 3027	
03	**Lithography: PR Exposure** Machine: Heidelberg Instruments MLA 150	
04	**Lithography: PR Development** Wetbench: Zone 13 Base Developer: AZ 726 MIF	
05	**Mounting on Carrier** Machine: Hotplate of RC8 THP-Manual Coater Material: QS135 Comment: Add Kapton tape for extra fixation	
06	**Etching SiO** Machine: SPTS APS	

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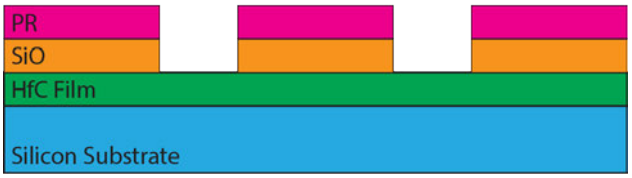
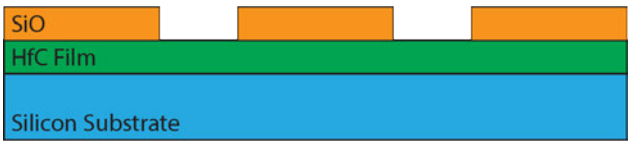
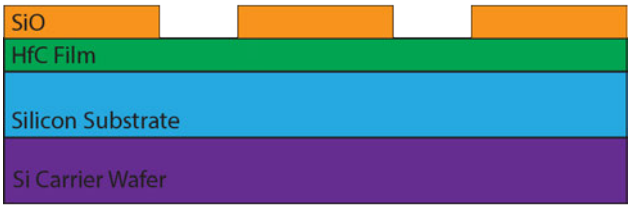
07	**Removal from Carrier** Machine: Hotplate of RC8 THP-Manual Coater	
08	**Lithography: PR Removal 1** Machine: Tepla 300	
09	**Lithography: PR Removal 2** Wetbench: UFT Remover: 1165	
10	**Mounting on Carrier** Machine: Hotplate of RC8 THP-Manual Coater Material: QS135 Comment: Add Kapton tape for extra fixation	
11	**Etching HfC** Machine: Veeco Nexus IBE 350	
12	**Removal from Carrier** Machine: Hotplate of RC8 THP-Manual Coater	

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Ebeam litho data - Photolitho masks - Laser direct write data			
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1	2um	No alignment	-
Substrate Type			
Part of a Si wafer with HfC thin film already on it: ca.50mm x50mm			

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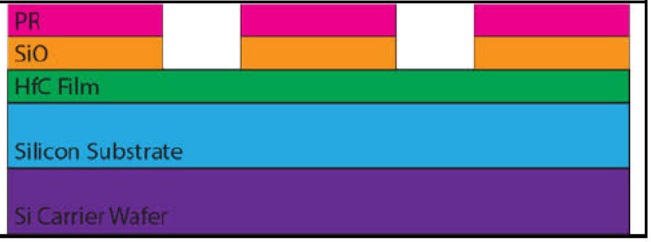
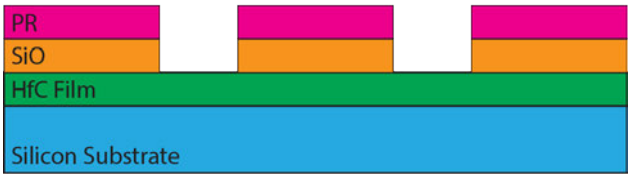
06	**Etching SiO** Machine: SPTS APS	

07	**Removal from Carrier** Machine: Hotplate of RC8 THP-Manual Coater	
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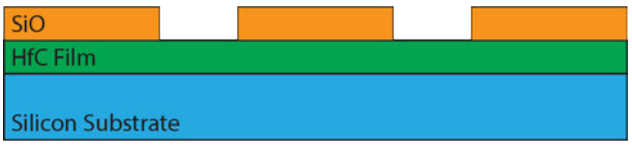
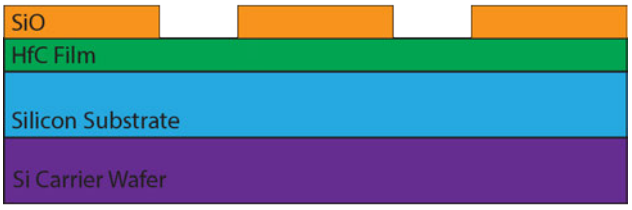
09	**Lithography: PR Removal 2** Wetbench: UFT Remover: 1165	
10	**Mounting on Carrier** Machine: Hotplate of RC8 THP-Manual Coater Material: QS135 Comment: Add Kapton tape for extra fixation	
11	**Etching HfC** Machine: Veeco Nexus IBE 350	
12	**Removal from Carrier** Machine: Hotplate of RC8 THP-Manual Coater	
13	**Etching of SiO Mask** Machine: SPTS uEtch Note: Try on a dummy first (piece with just HfC film and measure thickness before and after with FilMetrics)	

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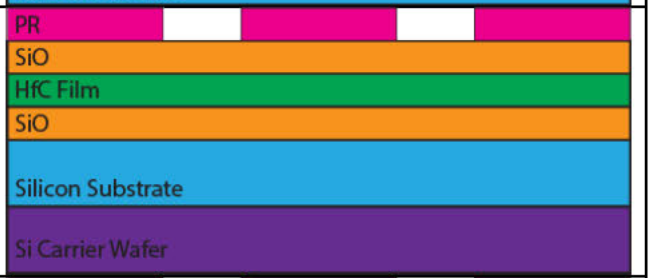
05	**Mounting on Carrier** Machine: Hotplate of RC8 THP-Manual Coater Material: QS135 Comment: Add Kapton tape for extra fixation	
06	**Etching SiO** Machine: SPTS APS	

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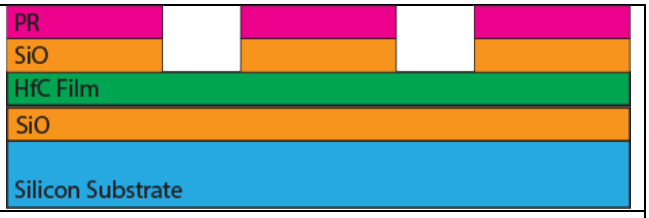
07	**Removal from Carrier** Machine: Hotplate of RC8 THP-Manual Coater	
08	**Lithography: PR Removal 1** Machine: Tepla 300 1' O2 Plasma	
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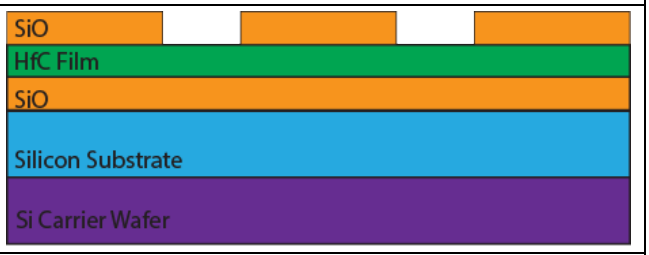
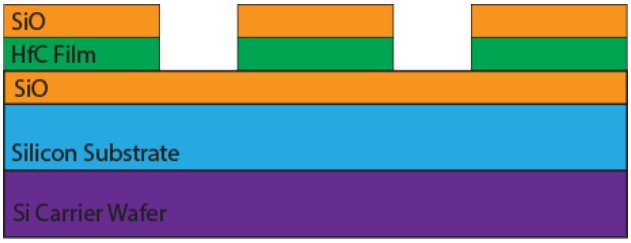
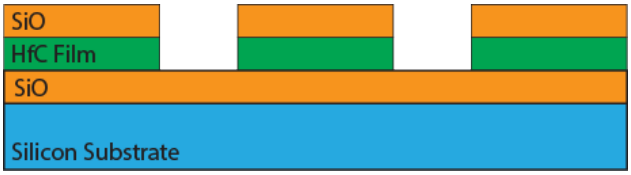
10	**Mounting on Carrier** Machine: Hotplate of RC8 THP-Manual Coater Material: QS135 Comment: Add Kapton tape for extra fixation	
11	**Etching HfC** Machine: Veeco Nexus IBE 350	
12	**Removal from Carrier** Machine: Hotplate of RC8 THP-Manual Coater	
13	**Release** Machine: SPTS uEtch Note: Try on a dummy first (piece with just HfC film and measure thickness before and after with FilMetrics)	

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Part of a Si wafer with SiO rlease layer and HfC thin film already on it: ca.50mm x50mm

Interconnections and packaging of final device

Thinning/grinding/polishing of the samples is required at some stage of the process.
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11	**Etching HfC** Machine: Veeco Nexus IBE 350	
12	**Removal from Carrier** Machine: Hotplate of RC8 THP-Manual Coater	

	Lithography: PR Removal 1 Machine: Tepla 300 1' O2 Plasma	
	Lithography: PR Removal 2 Wetbench: UFT Remover: 1165	
13	**Release** Machine: SPTS uEtch Note: Try on a dummy first (piece with just HfC film and measure thickness before and after with FilMetrics)	

Scan Lab Notebook

DYNclean

lint-free notebook A4

packed in cleanroom

antistatic

company EPFL

division ANEMS

name Fabian Bauer

date 13.10.2022

13.10.2022

Coating of Si Test Wafers with PR:

Surface preparation: Vapor HMDS
Standard recipe

Spincoating:
Machine: SSE SB-20
Recipe: STD_6000
Resist: AZ ECI 3027 Lot: DEAA363196-TVE6
Pool size: ≈ 5cm Exp: 12/2022

Softbake:
Temperature: 90°C
Time: 90s = 1' 30"

Exact steps:

1) Remove wafer from cassette and place on hotplate (using tweezers)
2) Close lid and select HMDS standard
3) Start heating up right hotplate to 90°C check with thermocouple
4) Wait for HMDS cycle to finish and remove wafer
~~5) Remove wafer and place on hotplate for 1'30"~~
~~6) Rem~~
5) Put on gloves and get PR ready ← check exp. date
6) select chuck
7) Insert chuck
8) Place wafer + activate vaccum
9) Close lid & spin STD-6000
10) Remove wafer & place on hot plate for 1' 30"
11) Place wafer in lightproof box

Observation: There seem to be a few particles on the suface that are visible after spincoating. Wafer new, first time removed from carrier. Handled only with my tweezers. Put down only on paper and machines.

Where could the particles come from?

Remark: Plan cleaning of equipments as well as use.

13.10.2022

13.10.2022

Training MLA 150 on Si Test Wafer

Materials: • 1 Wafer 100mm Si (Test Grade) coated with ECI 3027 PR approx. 2µm thickness (according to spincurve)

Machine: MLA 150 in CMi

Trainer: DORSAZ Julien

13.10.2022 F. Bauer

Training Development PR

Trainer: DORSAZ Julien
Materials: Wafer exposed in previous step on MLA 150
Zone: 13
Developer: AZ 726 MIF
Important: Gloves!
PEB: 90"
Development time: 60"

F. Bauer 13.10.2022

14.10.2022

Training: Resist removal
Materials: Wafer # 5 prediced by CMi
Zone: 2
Important: Put the right gloves at the right time (white, orange, big)

14.10.2022
F. Sauer

17.10.2022

Training Tepla

Materials: 1 chip from wafer #5 (the one with the #)
Zone: 11
Goal: remove remaining PR from sample. and verify under optical microscope.

Done: 1' + 2' for 3' total

Observation: Looks the same under the microscope...

17.10.2022
F. Sauer

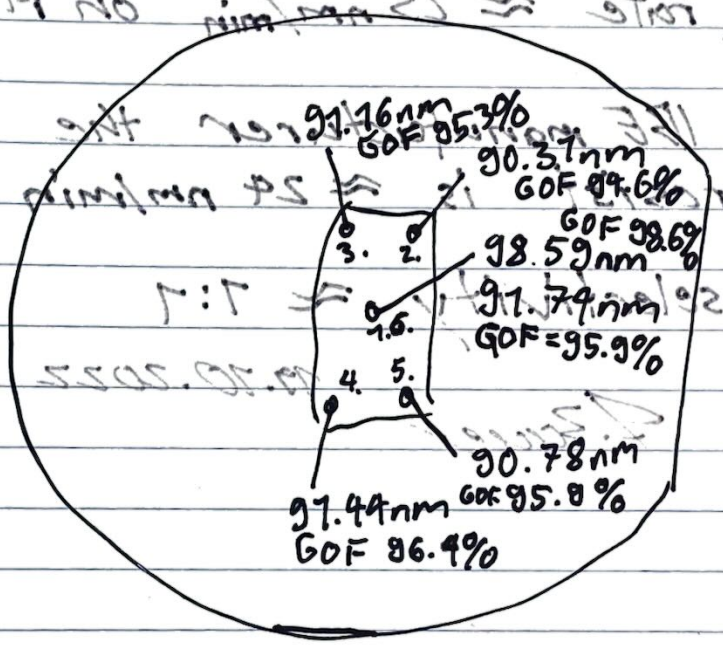
18.10.2022

Quickstiched Part of the 100nm wafer onto a carrier test wafer. Reading for IBE etch rate test.

Measured thichness of "100nm" wafer HfC film layer.

1. Baseline Procedure
2. Measurements using Recipe HfC on Si
   nominal 100nm ± 50%
   Fixed range 400nm +/- 1000nm

I changed this after the measurements to see how sensitive it is to it
=> about ± 70nm depending on the starting wavelength.



18.10.2022
F. Sauer

suspect false settings on IBE

19.10.2022	after 1st IBE	after 2nd IBE
1. 90.93	GOF 87%	65.32 GOF 98%
2. 90.85	GOF 82%	62.97 GOF 99%
3. 90.98	GOF 82%	64.34 GOF 99%
4. 90.80	GOF 87%	65.98 GOF 98%
5. 90.3	GOF 87%	66.48 GOF 99%
6. 90.3	GOF 88%	63.22 GOF 99%

90 - 65 → 25 ± 5

19.10.2022

100nm piece 1' at med power ÷70° in IBE.

Measured on Filmetrics → no change

▽ IBE Recipe verify which ▽
○ step you are on do not ○
modify "start" step.

Tried another 1' then measured again → this time it worked.

Estimated etch rate ≈ 25 nm/min on MEDIUM

According table of IBE manufacturer the rate for photoresist is ≈ 24 nm/min

⇒ selectivity ≈ 1:1

19.10.2022

T. Zacek

27.10.2022

cleaved a piece of ca. 1 cm² from the 600 nm wafer for use in a PL test

27.10.2022

F. Bauer

29.10.2022

Coating of 600 nm piece:

10 min dehydrate

coat using "CHIP_6000" EZI 3027

Softbake 90° 1'30" ← may have been closer to 2'

PR: Lot: DEAA363196-7DE6

Exp: 12/2022

Exposure: Dose: 275 $\frac{mJ}{cm^2}$

Defoc: -1

see job: fbauer_20221029

Development: PEB 100°C 90"

Developer: AZ 726 MIF

Development time: 60"

Rinse H_2O 1min

Still residues → +30" dev. time

+1' H_2O

after that no residues visible under microscope

Quickstick: Mounted the chip onto a Si Test wafer to prepare for IBE.
@ 110°C QS 135
24.10.2022
F. Sauer

IBE on 600nm piece:

Power: Med:

Duration: $\frac{600nm}{25 nm/min} \Rightarrow 24 min$

Worst case: $\frac{600nm}{20 \frac{nm}{min}} \Rightarrow 30 min$

Best-case: $\frac{600nm}{30 \frac{nm}{min}} \Rightarrow 20 min$

PR 2µm Film 600nm

$\Rightarrow t_{PR} \approx 3.3\, t_{film}$

Etch rate PR $\approx 25 \frac{nm}{min} \Rightarrow$ max etch time $= \frac{2000}{25} = 80 min$

Etch rate Si $37 \frac{nm}{min}$ worst case over etching = $10 min \cdot 37 \frac{nm}{min} \Rightarrow 370 nm$

"See if EPD works!" → does not

I did 25!

Then removed chip from carrier again @ 170°C on hotplate and cleaned in acetone → remove as much residues from quickstick and Kapton tape

1' in Tepla oxygen plasma

PR removal in wetbench zone 2

Inspection under optical microscope:
Looks good but some contamination visible. → inspect under SEM.

F. Zacharias 24.10.2022

25.10.2022

Tried to inspect under SEM, but SEM out of order → sample stuck in load dock → waited for technician to get it out.

25.10.2022

Next steps:
- retry SEM (also measure dimensions)
- Etch testing pattern and look at crosssection

27.10.2022

Cleaved off a piece of the 600nm wafer to use for cross section line etch test.

F. Zauer 27.10.2022

37.10.2022 Coating of 600nm piece for line test

- 10' dehydrate @ 135°C
- Coat using Clio 6000RPM with EGI 3027
- Softbake @ 90°C for 90"

Total time ca. 30'

Exposure: Dose: 275 $\frac{mJ}{cm^2}$

Defoc: -1

Development: PEB: 100°C 90" to top of AZ 726 MIF

Development time 1'20"

H_2O rinse

F. Zauer 37.10.2022

* Here a Quicketch Step is Missing 19.11.2022 F. Zauer

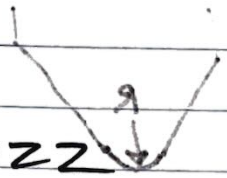
27.10.2022

IBE Etching line test:

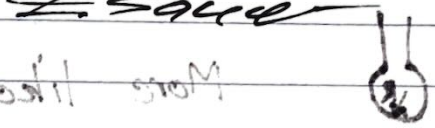
25! at med power and 70°
Flow cool: 25

PR removal:

2x 5' Remover
2x H_2O Rinse



2.11.2022



Tauber

27.10.2022

Good inspect. line test as well as devices under SEM
In particular look at sidewall profile and trench width.

Method: cleaved the line sample perpendicular to the lines using "diamond pen" and "cleaving pliers"

Observations: lines @ x 57.939
y 46.368

Hard to find lines!

2.11.2022

F. Tauber

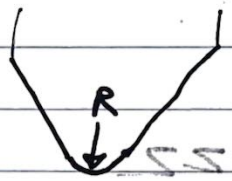
8.11.2022

Cleaved of 3 pieces from the 600nm wafer.
↳ for XeF_2 etch, ↳ for lines IBE −40°
rest for Marco → oxyde ↳ for lines Nan Recipe

Training Stylus Profilometer:
Zone 4
Dek Tak XT
Sample: "↳ device" 2.0 x5

Questions: stylus Tip size

Not like this ⇒ 

More like this 

Remarks add big (ca. 1mm x 1mm) square to lines. advanced layout to be able to test with profilometer.

Step size in "device" 800nm

Training XeF_2 etching:

Sample "↳ device" 2.0

Canceled due to broken equipment!

F. Sauer 8.11.2022

09.11.2022

Inspection of "H-device" sample under SEM, LEO

→ Error in equipment, not usable. → Called Mikhail told me to press "ignore"

14.11.2022

Coating samples 2.2 and 2.3

2 samples at the same time → 1. surface dehydration 10' @ 135°C

2. Coat using Chip 6000RPM with ECI 3027 Exp: 12/2022

1 after the other → 3. Soft bake @ 90°C for 90"

Exposure of samples : 2.2 and 2.3

Dose 275 mJ/cm2

Defoc -1

Development samples 2.2 and 2.3

PEB: 100°C 90"

AZ726 MIF

Development time 1'20"

H_2O rinse

Inspection under optical microscope

Mounting on test wafers using QS735

15.11.2022

IBE Advanced lines: EPD shows marked change @ t = 22'

Both first have a start step

Sample 2.2:
Recipe: MED Power t = 30' -70°

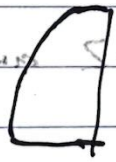
total 30'

Sample 2.3
Recipe:
-70° t = 15'
-40° t = 7,5'
-70° t = 7,5'

1' of cooling after each step

Inspection before etch:

2.2:  : Okay; 1µm lines not fully formed

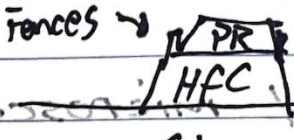
2.3  : Okay; also 1µm lines only partially formed

Inspection after etch: Optical: similar but rough surface, profile visible.

Inspection after etch SEM:

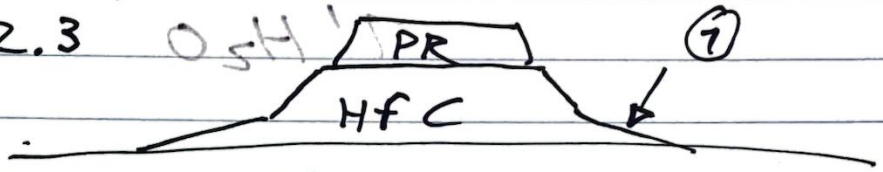
Sample 2.2: looks good

15.11.2022



Inspection after IBE with SEM

Sample: 2.3



Observation: Eliminated fences but created new problem as there are now ① this things.

Preparation: Cleaved wafer from wafer

Photolith:

Coating: 1. Dehydration: 10' @ 135°C

Samples 3.0 and 2.9 with SiO_2 layer

Chip 6000

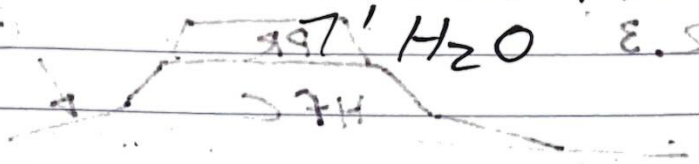
3. Softbake @ 90°C

Exposure: Dose 275 $\frac{mJ}{cm^2}$

Defoc -1

Design: advanced lines

Development PEB 100°C 90"
AZ 726 MIF 1'
1' H_2O



Inspection Optical ⇒ looks OK

27.11.2022

22.11.2022 Mounted Chip on carrier using GE 135 @ 110°C

IBE of Sample 3.0

estimated 1500 nm of HfC → $\frac{1500}{25} = 60'$

it is important to overetch in order to be able to release afterwards

⇒ add 20% buffer

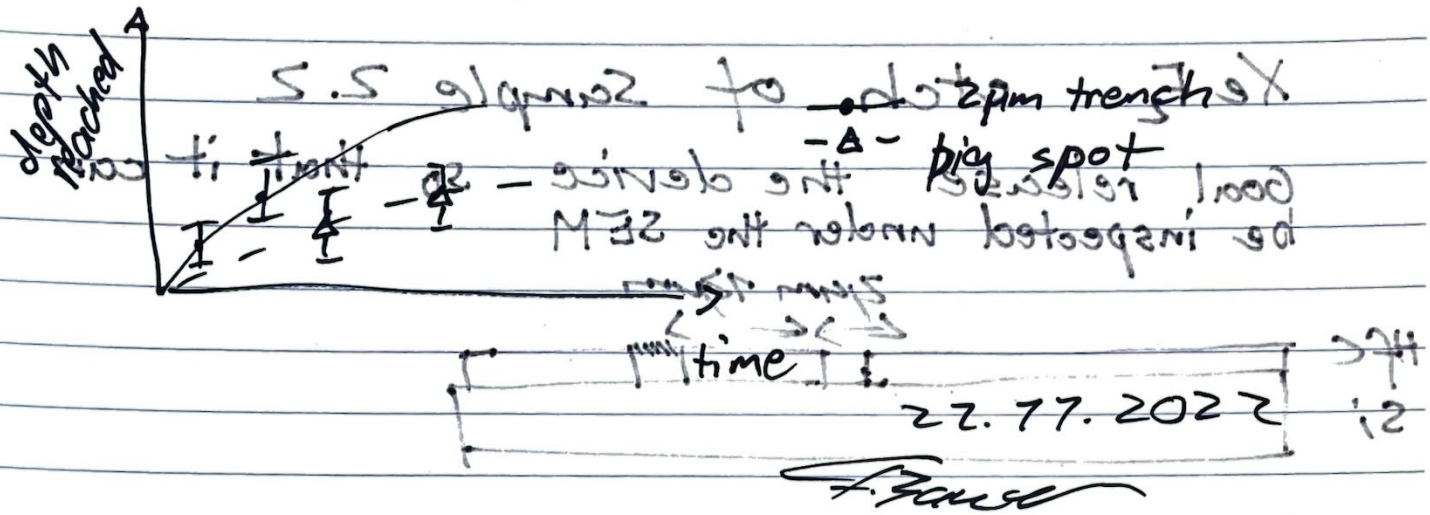
$60' \cdot 1,2 = 72$ min

I will etch at -70° as this proved to give the best results so far.

After 40' Clear drop off on EPD chart

⇒ decided to stop at 50'

For report create graph



23.07.2022

Samples removed

2.2 and 3.0

7' Oxygen Plasma in Tepla

(smallest piece of 3.0 may have been upside down on the plate)

Remover : bigger pieces are 2.2

@ 70°C in Remover

Water Bath

Optical Inspection:

2.2: looks okay only a few remaining particles

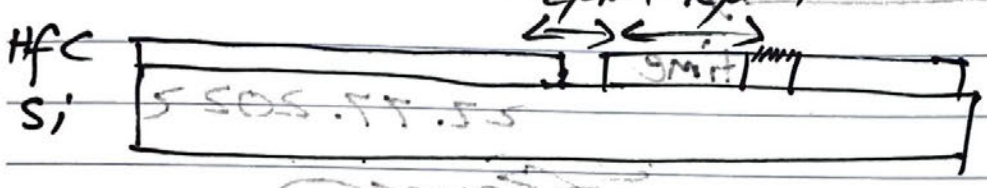
3.0: looks bad → delamination!

Training

SPTS Xea Xactix X4

XeF_2 etch of sample 2.2

Goal release the device so that it can be inspected under the SEM



2 chips ca. 7.5 x 7.5

74 cycle 7.7 expansion chamber Pres.
45 s/cycle

Optical Inspection:

sample appears damaged

next time trying
XeF_2
N_2 to help with cooling
time 75 s/cycle
+70s delay :

in order to see if it is the material or the recipe.
Maybe the layer got to hot ?

23.11.2022
J. Zacher

24.11.2022

Inspection of sample 2.2 under the SEM, front side of the effect of the XeF_2 etch.

sample_2.2_after_XeF ...

image 09.tif: device HfC layer cracked, pits from XeF_2 etch visible

image 70.tif: thin trenches

image 71.tif: section of trench

image 72.tif: 50% sections left to r. 7µm, 2µm, 3µm, 5µm, 10µm

image 73.tif: 7µm trenches

image 74.tif: 2µm trenches

image 75.tif: 3µm trenches

image 76.tif: zoom on middle trench of image 75

image 77.tif: probably the worst device

image 78.tif: probably the best device

image 79.tif: edged of device section

QS the sample 2.4 onto a carrier to get it ready for the etching in the APS.

Temperature of hotplate 100°C

28.11.2022

Removed 2 layers from carrier to prepare for PR removal.

Noticed film of PR looked uneven

PR removal:

5' bath 1 5' bath 2

2x H_2O Rinse according to timers

Optical inspection: looks bad SiO_2 delamination? Or maybe just PR residue?

Tried 1' 500 W O_2 Plasma for removing PR

looked better => 1 more minute

even better => 4 more minutes

QS 135 @ 770°C onto carrier wafer

29.11.2022

Add Kapton tape as extra fixation on 2.4.

IBE Med : 2 min

EPD @ 27' -> stopped at 23'
: ensure slight over etch

Optical: looks okay similar to before IBE

30.11.2022

Cleaved off samples from 2.rest

2.5 / 2.6 / 2.7

each chip ≈ 15mm x 15mm

Tip: 2 inch carriers are the perfect size for this size of chips and allow for easier pickup with tweezers.

PR coating of 2.5/2.6/2.7:

Resist: AZ ECI 3027

Recipe

Surface preparation:

vapor - HMDS (HMDS Standard)

Spin coating:

Recipe: CHIP_6000

done - 1 chip at a time

Softbake 90°C : 100s

Tip: Immediately set setpoint after turning on the hotplate. It will heat up to >150°C by default and cooling is slow!

setpoint ≈ 97°C gives 90°C measured with thermometer

Chuck Homing Error: Solution press "Init" repeatedly until you get no error!

PR exposure:

Tool MLA
Dose: 215 $\frac{mJ}{cm^2}$
Expose Mode: high quality
Laser: 405 nm
Laser Power: 100%
Defoc: 0
Design: "Advanced Lines and Cantilevers"

Careful to make sure lines are as perpendicular to wafer edge as possible in order to allows for easiers inspection later.

PR development:
PEB: 90" @ 100°C
Developer: AZ 726 MIF
Rinse: H_2O

Post development bake: 100°C on 5'

Check first when coming to the machine + Chromium version of wafers to save time

30.11.2022

Questions:

- Can a carrier wafer be reused?
- Cleaning after cleaving?
- Why does the same design end up with different # of stripes?

7.12.2022

Q6735 samples

2.5 / 2.6 / 2.7 onto a carrier wafer @ 100°C

7.12.2022

T. Sauer

Removed 2.4 from carrier and cleaned backside with acetone using "Qtips"

Unfortunately scratched the sample while doing so.

SEM ispection of 2.4

SEM gave me errors again!

Gun Vacuum

check first when coming to the machine

+ Chamber Vacuum

=> allows to save time

Staff solved it!

Has some issues with SiO_2 charging up. Ask Marco for advice.

6.12.2022

Inspection of 2.5 / 2.6 / 2.7 under optical microscope before removal from carrier after etching of SiO_2

Removal from carrier @ 100°C

Cleaned backside of chips with a bit of acetone on a foam swab.

7 min O_2 Plasma in Tepla 300 recipe 3

Inspection under optical microscope:

Samples still look okay

Tried PR removal in wetbench unfortunately wetbench was contaminated by previous user → some sticky residue built on the chips. Tried a second time but did not help

Discussion with J.P, J.D, L.G, M.L.

possible solutions:

- Tepla O_2 Plasma
- Remover in beaker

I tried remover 7165 in beaker in wetbench zone 12. Will leave it over night. With sample 2.5

Tried 4' of O_2 Plasma in Tepla 300 with sample 2.6

Looked better → tried 4 more minutes

Dektak XT Measurement

Sample 2.6:

1. Measurement across "trenches" at 50%
2. Measurement across "walls" at 95%
3. Measurement across edge of step square

Sample 2.4:

1. Measurement across edge of step square

6.12.2022

J. Sauer

7.12.2022

Removed sample 2.5 from remover 1165 bath.
→ looks much better

only very few flakes visible under microscope and not on major features.
Sample suitable for IBE

Mounted 2.5 and 2.6 on a carrier (for IBE) with QS 135 @ 100°C

IBE of samples 2.5

Med Power
20°
Flowcool 25

Beam V 500
Beam mA 800

EPD @ 20 min
stopped @ 23 min

Etch rate SiO_2 according manufacturer
72 nm/min at high power (@ 0°)
Factor for medium power 0.52
⇒ 37.44 nm/min at med power

IBE of sample 2.6:

same but −50°

Dektak measurements:

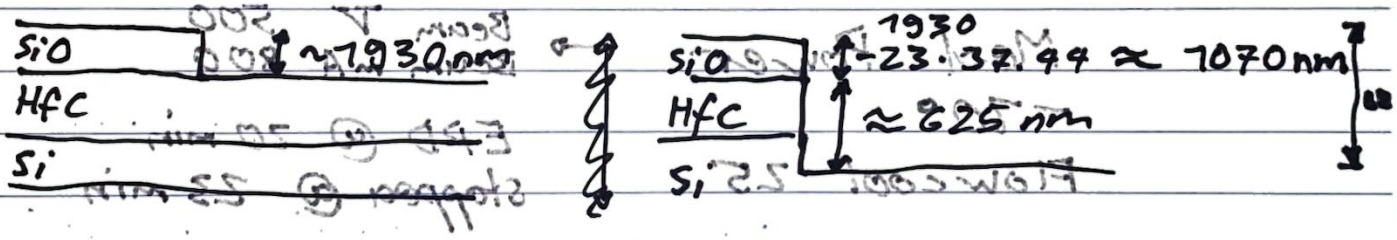
Sample 2.5

1. measurement across step edge
2. measurements across 50% lines

Sample 2.6:

1. Measurement: step edge
   Δz 1695 nm

Calculation etch rate from sample 2.6



etched Hfc + Si $\approx$ 625nm

of this Hfc $\approx$ 543nm

$\frac{625}{23} \cdot 20$ ← time of EPD

Gunk removal sample 2.7

O_2 Plasma Tepla 300

7' looked better → 7' better but marginal

→ put into 4765 over night

8.12.2022

SEM: Goals:

- get thickness of SiO_2 layer as deposited → 2.7
- get etch depth of HFC layer and look at sidewall profile → 2.5
- see if: APS chips "attacked" HFC → sample 2.7

SEM sample 2.5

looked at samples under the microscope, look okay no damage visible.

Looks good: clean etch, good sidewalls, nearly perfect etch depth

sample 2.7

Not enough time → next time

9.12.2022

PR coating of 2 samples: 2.8/2.9/2.10

Surface preparation: Vapor HMDS standard

Resist: AZ ECI 3027 Lot: DEAA420488-2041 Exp: 05/2023

Recipe Coater: CHIP_6000

Softbake: 90°C 90"

Make sure to „init" coater to avoid problems.

looked at samples under the microscope, look okay no damage visible.

Lesson learned: always check if there is resist first thing when you come. Because I had to wait for J.D. to bring new PR and then let it warm up to room temperature.

PR exposure

Design: lines & cantilevers

Dose: 275 $\frac{mJ}{cm^2}$ Defoc: -7

Laser: 405

Power: 100% Expose mode: high quality

PR development:

PEB: 90" @ 100°C

Developer: AZ 726 MIF 7'

H_2O 7'

PDB: 5' @ 100°C

12.12.2022

Training Ellipsometer

HfC films to thick

T. Bauer

13.12.2022

PR removal sample 2.3

Wetbench removal standard

Put chip into bath. Timer pressed timer start. Timer did not start. → called staff and removed chip from bath. Staff suggested using timer 2 also for first bath. Did this.

Optical inspection sample 2.3 after PR removal.

Dektak XT measurements sample 2.3

Measurement Step 2: saturation in the end

Always did 2-point level

19.12.2022

Mounting 2.8/2.9./2.10
on carrier in preparation for
etching in APS

QS 735 @ 700°C

Training µEtch:

Sample 2.5

goal: remove SiO Hardmask before imaging to be able to see if footing is present.

Recipe 3

Pressure: 725 Torr
N_2: 7000 sccm
EtOH: 400 sccm
HF: 575 sccm
1 cycle 300 seconds

15.12.2022

SEM Sample 2.5

SiO seems to be still on there

Sample 2.7

Okay

Training APS

Samples 2.8 / 2.9 / 2.10
mounted on 9 carrier

Trainer: Tanguy

Goal: Etch trough SiO layer

Recipe: SiO_2 PR

Time: calculated:

Let's do:

Tip: in case one needs to stop before Endpoint

not visible change on EPD after time run out

complain, smMain for EPD graphs

Note: After etching two chips look more where one still has the typical PR interference fringes

16.12.2022

Removing from carrier using hotplate @ 100°C

PR removal:

7' O_2 Plasma Tepla 300

5' Remover 7765

5' Remover 7765

2x H_2O Rinse

Note: Timer was broken, for both 7 so had to look at the clock → might have been ± 30 seconds.
when taking out chips looked: "grunky" → did another: round.
Chips stuck together on tops of each other in the holder / used to used another holder in the second round.

Still not good → Tepla 'Gigabatch 7' O_2 High better but not completely gone → 7 more minute
Letter again but still some residues → 7 more minute

SEM Sample 2.6

19.12.2022:

QS Samples 2.8/2.9./2.10 onto carrier wafers.

Added ~~kapton~~ to all three samples

IBE of 2.8:

-70° MED Power
EPD @ ≈ 22' stopped @ 24'

(start step 0° Flowcool 5 5 seconds)

IBE of 2.9 : Nan Recipe

- -10° t = 16'
- -40° t = 8.5'
- -70° t = 8.5'

} all at MED Power

1' of cooling between and at the end

20.12.2022:

SEM sample 2.8

Idea: Before next SEM wash sample in water to get rid of debris

µEtch Sample 2.8
Bake @ ≈ 170°C ca. 5'
Recipe 3: 900s
1 cycle
Needle Valve @ 12

~ 125 nm/min

22.12.2022

SEM 2.8 after µEtch

22.12.2022
F. Zauner