Dry Etching of InSb Using OX-35 Etcher E241 – Spring 2018

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I. Project Motivations and Objectives

The use of indium antimonide (InSb) is seen in various applications in the semiconductor industry from high performance infrared detection systems, photodiodes and fast switching transistors. Primarily, InSb has been patterned using wet etching despite the limitation of this process, including mask undercut and corrosive etch products. By finding a method that allows for dry etching of InSb substrates, we can produce higher quality features at smaller scales without the existing defects that exist in the current process.

The objective of our project was to attain a smooth anisotropic wall on an InSb substrate. Looking through literature, there have been a few attempts with limited success to solve this problem, due to the non-volatile products of the etch (Pusino *et al.*, 2016; Abautret *et al.*, 2015; Zhang *et al.*, 2009). Our focus was primarily at observing the effects of ratio between the flow rate of methane and hydrogen, the flow rate of argon, and the chamber pressure, by quantitatively and qualitatively analyzing the smoothness and angles of the etch features. To characterize these features, we will be using tools such as the AFM and SEM, with the goal of obtaining a smooth etched surface with vertical side wall by the end of the quarter.

Our target etch characteristics are as follows:

- · Sidewall angle: 85 degrees minimum
- · Roughness below 1 nm
- Etch rate: 20~50 nm/min

II. Literature Review

There has been little published in finding a dry etch method for InSb. Currently there exists reactions that utilize Cl plasma, however it has resulted in the byproduct of InCl which is very non-volatile and leaves many defects and a residue that requires a high temperature purge cycle (Pusino *et al.*, 2016; Abautret *et al.*, 2015; Zhang *et al.*, 2009). Another primary reaction has used the gas mixture of CH₄, H₂, and Ar in varying quantities. This chemistry has been understood for III-V semiconductors and has been used in the etching of GaSb. However, it has been reported that by altering the ratio of CH₄ to H₂, we can greatly



Figure 1 InSb etch profile with different etch recipes from Pusino *et al.*(2008); (*left*) CH4/H2/Ar being 15/50/5 sccm, (*right*) CH4/H2/Ar being 9/36/3.

vary the etch features. It has also been shown that by varying pressure, ICP and Bias power, and the flow of the non-volatile Argon gas, we can control the etch rate and fine tune it for our needs. Figure 1 from Pusino *et al.* (2016) illustrates the etch characteristics which varies with different etch recipe. Current features are on the order of microns and we hope to be creating features on the scale of twenty to thirty nanometers by optimizing the conditions aforementioned in the future.

III. Design of Experiments (DOE) and Revisions

Several parameters were considered when designing our experimental plan and matrix. From our literature review, we were aware that CH_4/H_2 ratio, Ar flow rate, chamber pressure, ICP power, and RF bias power affected etch characteristics. However, since our goal is to create a smooth, anisotropic etch, and since literature states that the ICP power has little effect on etch anisotropy, we kept it at around 600W. Thus, our initial plan was to focus on four specific variables: CH_4/H_2 flow ratio, Ar flow rate, chamber pressure, and RF bias power.

Initially, we divided our DOE into four different studies for each of the variables that we were exploring. We approached the experiments as a simple factor optimization process – by changing one variable at a time to optimize to the best condition. Our methodology is illustrated in Fig. 2. and in detail is:

- 1. To study the effect of CH_4 and H_2 flow rate and ratio on etch characteristics while holding Ar flow rate, pressure and RF bias power constant. By starting at the literature optimum point, we hope we can fine tune the CH_4/H_2 value.
- 2. To study the effect of CH₄ and Ar flow rate and ratio on etch characteristics while holding pressure and RF bias power constant and keeping CH₄/H₂ ratio at the previously optimized value from study 1.
- 3. To study effect of pressure and RF bias power on etch characteristics while holding CH₄/H₂/Ar flow rate and ratio constant at the previously optimized value from study 1 and 2. This last study is intended to be a full factorial experiment such that we are able to see the interaction effect between pressure and RF bias power.



Figure 2 Initial DOE for Study 1 and Study 2

However, as we learned more about the etching tool, we decided not to go through with this DOE for several reasons. First, we assume that the ICP RIE tool from the literature is similar to ours such that we can use their reported optimum point as our starting point and fine tune each of our parameters. Upon discussion with our mentors, we realized that although the OX-35 etcher is an ICP RIE etcher, it is not exactly the same model as in the literature. Using the reported optimum point would not necessarily be an ideal start to fine tune the parameters. Therefore, this DOE is better for fine tuning each parameter once we have the idea of where the tool optimum point is. Finally, using this DOE will not allow us to see the interaction effect between each parameter clearly.

Our second DOE is designed to be the full factorial design that will allow us to see the interaction effects between factors. It involves four two-level factors (one upper parameter and one lower parameter for each factor) that we were exploring, i.e., CH_4/H_2 ratio, Ar flow rate, pressure, and RF bias power, resulting in 16 different cases. We obtained the upper and lower bound parameters of each factor from our preliminary experiment.

From our preliminary study we determined that the etch profile, or more significantly the vertical wall angle, is unaffected by the RF bias power. This is shown in the SEM images in Figure 3 and the results on Table 1. Hence, we eliminated this variable to simplify our DOE. In addition, we determined that 200W RF Bias power gave a more desirable etch profile. This elimination of the RF bias power from our explored variables resulted in a final 8 cases for our DOE, as shown by Table 2.



Figure 3 SEM images of etch profile from recipe: (*a*) 13/32/5 sccm, 15 mTorr, 200 W, and (*b*) 13/32/5 sccm, 15 mTorr, 150 W.

Parameters				Resulting etch characteristics		
CH4/H2 (sccm)	Ar (sccm)	Pressure (mTorr)	RF Bias Power (W)	Etch depth (nm)	Etch rate (nm/min)	Vertical angle (deg)
13/32	5	15	200	338.2	67.64	84.1
13/32	5	15	150	344.1	68.82	83.4

Table 1Results of increasing RF bias power

Overall, we have explored the effects of three variables: CH_4/H_2 flow rate, Ar flow rate, and chamber pressure, consequently varying these variables between 13/32 to 5/40, 5 to 20 sccm, and 10 to 20 mTorr respectively. In short, we initially designed our DOE to focus on one parameter at a time to find the most desirable etch profile, however such optimization method did not account for the interaction effects between variables and is not suited for the tool we use. Hence, we altered our DOE to account for these interaction effects, resulting in the final eight cases shown in Table 2.

Case	CH4/H2 (sccm)	Ar (sccm)	Pressure (mTorr)	RF Bias Power (W)	ICP Power (W)
1	13/32	20	20	٦ ٦	
2	13/32	20	10		
3	5/40	20	20		
4	5/40	20	10	200	600
5	13/32	5	20	200	600
6	13/32	5	10		
7	5/40	5	20		
8	5/40	5	10		

Table 2Final DOE Table

IV. Fabrication Process

We initially debated between using a soft mask (photoresist) versus a hard mask, such as SiN and SiO₂. We decided to opt for the hard mask because literature claimed that it results in a more vertically angled, smoother etch. Etch rate and selectivity test of different hard masks, e.g., SiN and SiO₂, are explored in our early preliminary test using the etch recipe from the literature. Finally, we chose to use SiO₂ with our hard mask because of its high selectivity and well established etch recipe.

The fabrication steps in our work are 1) SiO_2 hard mask deposition, 2) lithography pattern transfer, 3) SiO_2 hard mask dry etching, and 4) InSb substrate dry etching. This process is summarized in Fig. 4 and the related tools are listed in Table 3.

1) SiO₂ hard mask deposition

In this first step, we use CCP-DEP which is a Plasma Enhanced Chemical Vapor Deposition (PECVD) tool to deposit 230 nm thick SiO_2 layer on top of the pieces substrate. With the recipe SiO-350-0, the temperature, pressure, and resulting deposition rate are ~350°C, 10 mTorr, and 60-70 nm/min, respectively. The deposition time is usually around 3 minutes and 20 seconds. Based on our finding, it should be noted here that this step might be accountable for the roughness (2-5 nm) of our top substrate surface. We believe that this roughness might occur from either the pinholes formed from PECVD allowing ion bombardment to etch through the hard mask even though only 1/3 of the hard mask is etch through during InSb etching, or the temperature is too high for InSb which has melting point at 527°C.



Figure 4 Fabrication process diagram using hard mask; blue: InSb substrate, green: SiO₂ mask, and orange: SPR 3612 photoresist.

Table 3	Fabrication proces	s and main tools	s involved in	each process
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Fabrication Step	Process	Tools
Step 1: Hard mask deposition	Hard mask deposition	PlasmaTherm CCP-Dep
Step 2: Resist coating	Photoresist coating	Yes Oven, Headway
Step 3: Patterning	Patterning and developing	Heidelberg
Step 4: Hard mask etching	Hard mask etching	OX-RIE
	Photoresist removal	Matrix and Wbflexcorr (SRS-100)
Step 5: InSb etching	InSb Etching	OX-35
	Hard mask removal	Wbflexcorr (BOE 6:1)

2) Lithography pattern transfer

With the hard mask deposited onto our substrates, we primed and singed them using Yes Oven and then coated the resist using Headway. Shipley 3612 resist is used and the spin speed is set to be around 2250 rpm to obtain resist thickness of 1.6 μ m. We then prebaked the substrate at 90°C on hot plate for 2 minutes. Heidelberg maskless direct write lithography tool is used to expose light at 405 nm wavelength to transfer our pattern onto the resist. With the resist thickness of 1.6 μ m, Dose and Defocus in exposure step are set at 100 mJ/cm² and -2, respectively. After exposure, we post-exposure baked our sample at 115°C for 1 minute. The samples are then soaked in MF-26A developer for 2 minutes and washed with DI water. Post bake at 115°C for 2 minutes is done in the last step. It is worth noting that since our smallest feature line is 1 μ m width, it pushed Heidelberg to operate at its resolution limit at 1 μ m. We witnessed the *underexposure* issue of this 1 μ m line and decided to focus our attention on lines wider than 2 μ m instead. Dose and Defocus test matrix would be helpful if we ultimately want to get a good result for the 1 μ m lines.

3) SiO₂ hard mask dry etching

OX-RIE which is a Reactive Ion Etcher (RIE) tool is used to etch the SiO_2 hard mask down to InSb substrate layer. The following recipe is used,

Ar: 30 sccm / CHF₃: 45 sccm / CF₄: 15 sccm / 100 mTorr / 500 W / 20°C

The measured etch rate for SiO_2 and photoresist are 230-250 and 60-70 nm/min, respectively. Several issues are encountered at this process such as under etching of hard mask leading to grass and incomplete hard mask etch despite long over etch time. We will discuss these issues in detail later. However, to solve the incomplete hard mask etch, we found that the poor thermal contact between the piece substrate and the carrier wafer is the main cause. Area on pieces with poor thermal contact makes the heat to accumulate and therefore make either make the photoresist to burn or the SiO₂ dry etch to not be as expected. We solve this problem by putting sufficient *Santavac Diffusion Pump Oil* on the piece back side, and (might not be as important as the oil) separating etch into the following steps,

1. Etch for 30 sec / 2. Hold for 2 min / 3. Etch for 30 sec / 4. Hold for 2 min / 5. Etch for 25 sec.

The total etch time is 1 minute and 25 seconds which corresponds to 25-30% ever etch time. After SiO₂

etch, to remove the remaining photoresist, Matrix which is an O_2 plasma etch tool is used to strip resist using O_2 plasma. Long strip recipe with 5 minutes O_2 plasma is used. To further clean the resist residues, we soaked the samples into resist stripper SRS-100 for 20 minutes.

4) InSb substrate dry etching

OX-35 which is an Inductively Coupled Plasma (ICP) Reactive Ion Etching (RIE) tool is used to etch the InSb substrate. The recipes used are those according to DOE shown earlier in Table 2. This step is the main step in our experiment and special attention has been put in. To ensure good chamber condition, we run a chamber clean step, i.e., *Cham Clean Def Cl2 SF6 O2*, every time there are another user before us. Before each run except the first run, we run the short chamber clean step in which each step in *Cham Clean Def Cl2 SF6 O2* is shorten to 3 minutes, and the chamber conditioning step which is the recipe that we want to use. These two processes are run with dummy wafer. After InSb etching, the samples are soaked into the 6:1 Buffer Oxide Etchant (BOE) for 15 minutes to etch away the remaining SiO₂ hard mask. This finishes our fabrication process for the samples. Images of some tools and the pattern used in the lithography step is also shown in Fig. 5.



Figure 5 Tools and pattern used (not all included): (*a*) Nanospec, (*b*) Pattern used in lithography, (*c*) and (*d*) CCP-DEP, (*e*) Yes Oven, (*f*) OX-RIE, and (*g*) OX-35

V. Characterization

To evaluate the quality of our fabrication and etching processes, we utilized various characterization tools such as Optical microscope, Nanospec film thickness measurement, Alphastep 500 profiler, Sensofar S-Neox optical profiler, SEM and AFM imaging. Optical microscope is used to observe sample generally to ensure the quality of pattern and features after each fabrication step, i.e., lithography, SiO₂ hard mask etching, resist stripping, InSb etching, and SiO₂ hard mask removal steps. Nanospec is also used in parallel with optical microscope to measure film thickness in each step. The rest of the characterization tools can be categorized into quick and detail characterization. Alphatep and S-Neox profiler are two tools we used to quickly and roughly get the etch characteristics information such as etch depth and etch rate. With this information, we could immediately choose which direction to move forward with our DOE.

SEM and AFM are two tools we used for detailed characterization. We chose these tools because they can image features such as side wall angle and roughness. Starting out the project, we put priority in ensuring that the etch is anisotropic and the wall is as vertical as possible with smooth etched trench and side walls. Throughout our project, we found cross sectional SEM immensely useful in evaluating the quality of the etch qualitatively and quantitatively. Cross sectional SEM allowed us to measure the sidewall angle quantitatively and to visualize the smoothness of etched floor and top substrate surface, and the sidewall roughness qualitatively. While top-down SEM allow us to measure the sidewall roughness, or Line Edge Roughness (LER). To quantitatively measure the Line Edge Roughness or LER, we measure the peak to peak width of the wavy line as seen from the top-down SEM images and report the rms value of this roughness. By picking out successful samples through SEM, we were able to save time and only characterize the best samples through AFM imaging. AFM gave us quantitative measurements for the roughness, or Rq value, of the etch at the top surface and bottom of the trench. This Rq value is measured using XEP data acquisition software which carries out a root mean square analysis over the area of interest. These measurements and characterization methods were vital to our project and helped us decide where to further allocate our time, where to move forward with our DOE, and to help gather data for the final results of our project. Figure 6 summarizes the characterization tools and their results.



Figure 6 Results from characterization tools employed in this project

VI. Results / Findings

Ultimately, we were able to reach our goal of a vertical wall angle above 85 degrees, without an undercut present and a reasonable etch depth and etch rate. From the conditions explored in our DOE, the etch angles ranges from 70.1 degree to 86.5 degree. The best vertical wall angle case with the angle of 86.5 degree is obtained by a 13/32/5 sccm etch flow of CH₄/H₂/Ar, pressure of 10 mTorr. As previously mentioned, our ICP power and RF bias power were kept at a constant value of 600W and 200W, respectively.

When characterizing the vertical wall angles, we used the cross-sectional SEM images as stated earlier. Several angle measurements are made for each cross section of one sample. At least two cross section are measured per sample. The results are averaged and reported as the vertical angle of that sample. Line edge roughness (LER) is reported as a rms of roughness value, while the AFM etch floor roughness are reported in Rq rms value as mentioned in previous section. A summary table of our results is shown in Table 4, with the three of our best vertical etch angles highlighted. Figure 7 also shows the SEM cross-sectional images of three of our best vertical angle cases.

Parameters			Resulting etch characteristics				
CH4/H2 (sccm)	Ar (sccm)	Pressure (mTorr)	Etch rate (nm/min)	Vertical angle (deg)	Line Edge Roughness (nm)	<i>AFM</i> , Etch Floor Roughness (nm)	
13/32	20	20	72.5	84.5	65.1	-	
13/32	20	10	64.5	80.1	46.0	-	
5/40	20	20	74.7	86.0	47.4	1.8	
5/40	20	10	51.9	70.1	35.4	-	
13/32	5	20	76.0	74.5	49.5	-	
13/32	5	10	63.9	86.5	56.6	1.2	
5/40	5	20	64.4	83.8	56.6	1.3	
5/40	5	10	65.7	84.6	53.0	0.9	

Table 4 Summary of results



Figure 7 SEM images of three of the best vertical wall angles, achieved by
(a) 5/40/20 sccm CH₄/ H₂/Ar flow rate and 20 mTorr chamber pressure,
(b) 13/32/5 sccm CH₄/H₂/Ar flow rate and 10 mTorr chamber pressure,
(c) 5/40/5 sccm CH₄/H₂/Ar flow rate and 10 mTorr chamber pressure.

Data Analysis: Effects of each individual factors on etch characteristics

Now that we have all the results, we implement data analysis to find any relationship among factors of dry etching, i.e., CH_4/H_2 ratio, Ar flow rate, pressure, and RF bias power, and etch characteristics such as vertical wall angle, etch rate, and LER. First, we look at individual effects of the etch parameters on the resulting etch profiles. Linear regressions between each factor and etch characteristics of interest are carried out. Since we only have eight data points in total with two data points for each factor, the linear fit is not intended to be a prediction line, but rather a general trend line. As shown in Fig. 8, an increase in chamber pressure increases vertical wall angle, etch rate, and LER. Similarly, an increase in CH_4/H_2 ratio increases the etch rate and LER, however it has little effect on the vertical wall angle. Overall, we can determine that to increase vertical wall angle, we should be increasing pressure or decreasing Ar flow rate. While to minimize LER we should be decreasing pressure, or increasing Ar flow rate, or decreasing CH_4/H_2 ratio.



Figure 8 Effect of (*top row*) pressure, (*middle row*) Ar flow rate, (*bottom row*) CH₄/H₂ ratio on vertical wall angle, etch rate, and Line Edge Roughness

Data Analysis: Overall effect without interaction terms

We then use linear regression without interaction term among each factor to find the relationship with each etch characteristics (response.) The regression is in the form,

Response =
$$b_1 + b_2$$
 (pressure) + b_3 (Ar flow rate) + b_4 (CH₄/H₂ ratio)

where b_1 is the intercept term and $b_{i,i>1}$ are the linear coefficient for each factor. Figure 9 shows the coefficient estimates of each factor (b_i) and their corresponding probabilities. The fact that none of the probability value of these factors are below 0.05 (at 95% confidence level) indicates that they are not statistically significant to any of the etch characteristics. The strongest finding from this linear regression fit is the positive dependence of etch rate on pressure; i.e., if pressure increases the etch rate will increase.

Data Analysis: Interaction effects

To explore more about the interaction effect, we look into the interaction between each factor in our DOE using JMP data analysis software. Figure 10 shows an example of the interaction effect between pressure and CH_4/H_2 ratio on the vertical wall angle. At high Ar flow rate (20 sccm), difference in pressure makes the vertical wall angle to change differently with increasing CH_4/H_2 ratio. Similarly, this effect is obvious at low Ar flow rate (5 sccm).



Figure 9 Linear regression fit of etch characteristics and factors without interaction terms using JMP data analysis software



Figure 10 Interaction effects between pressure and CH₄/H₂ ratio on the vertical angle

Data Analysis: Overall effect with interaction terms

To account for these interaction effects, we fit another linear regression with the interaction terms. The regression is in the form,

$$Response = b_1 + b_2(pressure) + b_3(Ar \ flow rate) + b_4(CH_4/H_2 \ ratio) + b_5(CH_4/H_2 \ ratio \times Ar \ flow rate) + b_6(CH_4/H_2 \ ratio \times pressure) + b_7(Ar \ flow rate \times pressure)$$

where b_1 being the intercept term and $b_{i,i>1}$ are the linear coefficient for each factor; Figure 11 shows the coefficient estimates (b_i) of each factor and their corresponding probabilities. Fortunately, the regression



Figure 11 Linear regression fit of etch characteristics and factors with interaction terms using JMP data analysis software

shows that for the vertical wall angle, all of the interaction terms are statistically significant. Theoretically, this gives us a starting model to get a desired vertical wall angle; however, needs to be experimentally verified to ensure the validity of the model.

Data Analysis: Effects of individual factors on AFM surface roughness

AFM analysis has been instrumental in our understanding of the effects of etch variables on trench floor roughness. As shown in Fig. 12, we see that as pressure increases, the floor roughness increases. As Ar flow rate increases, the floor roughness stays quite constant, and as CH₄/H₂ flow rate increases, the floor roughness stays quite constant, and as CH₄/H₂ flow rate increases, the floor roughness slightly increases. From these trends, we can conclude that chamber pressure has the most prominent effect on the trench floor roughness of our etches.

At the beginning of the quarter it was our initial goal to uncover the etch chemistry which delivered the most anisotropic etch of InSb and the smoothest. We discovered this case to be 5/40/5 (CH₄/H₂/Ar) delivered with 200 W RF bias power at a chamber pressure of 10 mTorr, shown in Fig. 13. To quantitatively measure the roughness of the piece, AFM characterization and analysis was done, as described in the previous section. The trench of the sample had a roughness of 0.852 nm, lower than our reproduced literature condition of 1.735 nm and under our 1 nm goal we set at the beginning of the course. The smoothness of our trench has given us an etch chemistry recipe that we will continue to use as we move towards smaller features.



Figure 12 AFM trench surface roughness results from pressure, Ar flow, and CH₄/H₂ ratio



Figure 13 (*left*) AFM imaging and (*right*) SEM image of InSb trench with smoothest trench floor (5/40/5 sccm CH₄/H₂/Ar flow rate and 10 mTorr chamber pressure recipe.)

VII. Issues Faced

Grass etch floor

An issue that we faced during the etch process was a severe roughness on the trench floor. This problem is illustrated by Fig. 14, and this phenomenon is known as a "grass" issue, as the resulting rough surface looks like grass. This was caused by a lack of over etch on the hard mask etching process on the OX-RIE, leaving islands of un-etched hard mask on the InSb surface, which acts as a micro mask and then translates to an uneven etching process during the subsequent InSb etching. We resolved this issue by increasing our hard mask over etch percentage to 25-30%.



Figure 14 SEM images of grass issue prevalent after the etch process.

Rough top surface of substrate

In our preliminary studies, we also observed that the top surface of the InSb was extremely rough, to the extend in which we could see the roughness through an optical microscope, as shown by the Fig. 15 above. We realized that this was caused by a too thin hard mask layer being slightly etched through during the InSb etching process This was a relatively easy issue to fix, as we simply increased our mask thickness from 160 nm to 225 nm. We therefore keep in mind that at least more than a half the hard mask must remain at the end of the etch to prevent this issue.



Figure 15 (*left*) Optical Microscope image and (*right*) S-Neox image showing the roughness of the top surface of the InSb substrate

Line Edge Roughness Issue

As mentioned in previous sections, the LER on the sidewalls has been a prominent concern and recurring issue in the etch samples. This may be caused by polymer deposition onto the sidewalls, causing a rough surface etch, or because of the etch parameters used, such as the RF bias power. This issue is being further investigated.



Figure 16 Line Edge Roughness seen in AFM (left) and SEM (right)

VIII. Conclusion

The goal of this project is to create a smooth, anisotropic etch with vertical sidewalls on InSb substrate using the OX-35 etcher. We have obtained a recipe that achieve a trench vertical wall angel of 86.5 degrees and a trench floor roughness of 0.85 nm, which align with the objective parameters that we specified in the beginning of the quarter. Through our data, we have shown that increasing pressure and decreasing Ar flow rate would increase the vertical wall angle, while decreasing pressure, increasing Ar flow rate, and decreasing CH4/Ar ratio would minimize the line edge roughness. We have also shown through the JMP software that there are prominent interaction effects between pressure and CH_4/H_2 ratio on the vertical wall angle. Future work would focus on validating the model found from linear regression with interaction terms, improving the sidewall roughness or line edge roughness, further improving on surface roughness, and decreasing the feature size to the nanometer scale using e-beam lithography.

IX. References

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