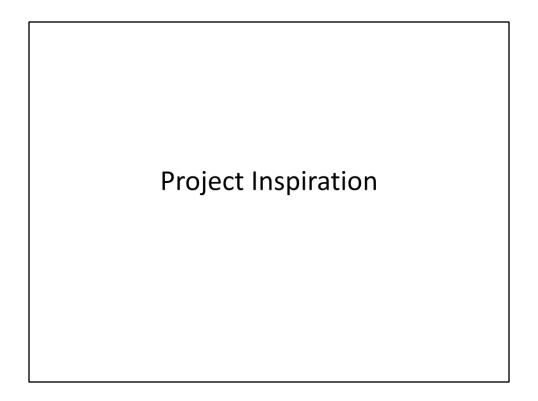
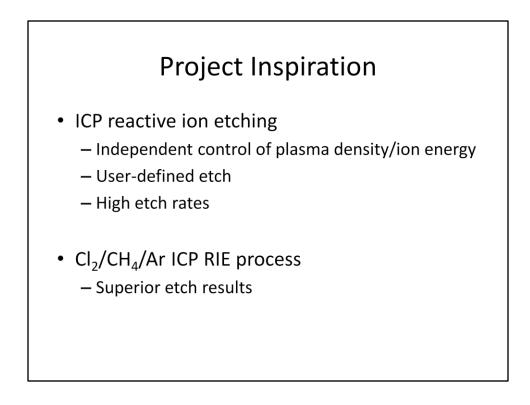
InP Etch Results – ICP RIE

Prepared by Matthew Haines

Note: Read notes found on each slide – they contain important process information!

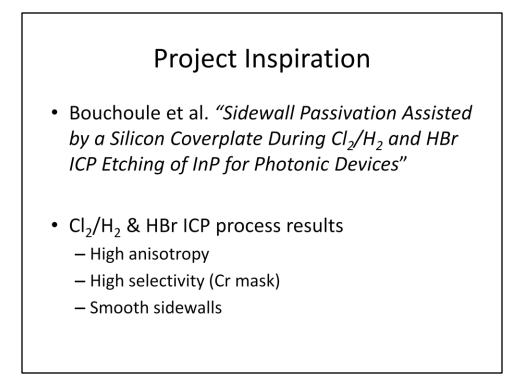




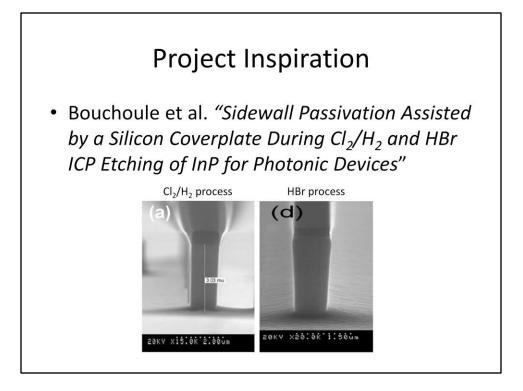
InP nanostructures can be used for numerous nanophotonic systems, such as Bragg mirrors, waveguides, etc.

Compared to typical RIE systems, ICP RIE systems enable independent control of plasma density (through coil power adjustment) and ion energy (through platen power adjustment). This enables the user to define the etch – one can ensure either an ionic-based etch or a chemical-based etch. Additionally, high etch rates are attainable with ICP RIE systems.

Cl2/CH4/Ar ICP processes have been shown to result in superior etch results relative to other etch mechanisms. This is due to surface passivation as well as increased indium volatility. The purpose of Ar is to increase plasma stability as well as to promote the desorption of etch products.



Both ICP RIE processes (using Cl2/H2 and HBr) resulted in high anisotropy, high selectivity, and smooth sidewalls.

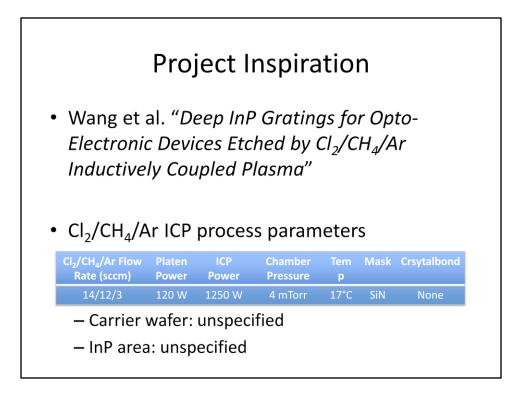


Both ICP RIE processes (using Cl2/H2 and HBr) resulted in high anisotropy, high selectivity, and smooth sidewalls.

Project Inspiration

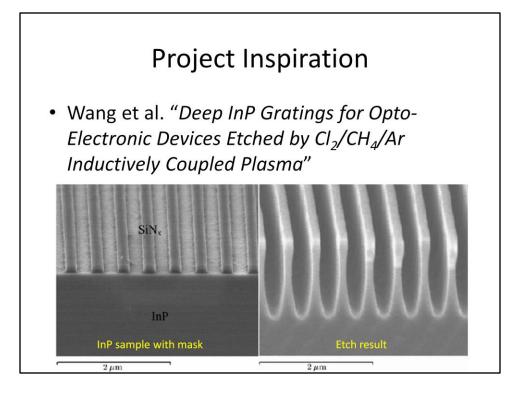
- Bouchoule et al. "Sidewall Passivation Assisted by a Silicon Coverplate During Cl₂/H₂ and HBr ICP Etching of InP for Photonic Devices"
- Major process drawbacks
 - 1. Si coverplate passivation source
 - 2. Inconsistent etch lack of temperature control

The use of a Si coverplate as a passivation source is industrially impractical; during future ICP RIE processes, 4" InP wafers will be used instead of silicon.



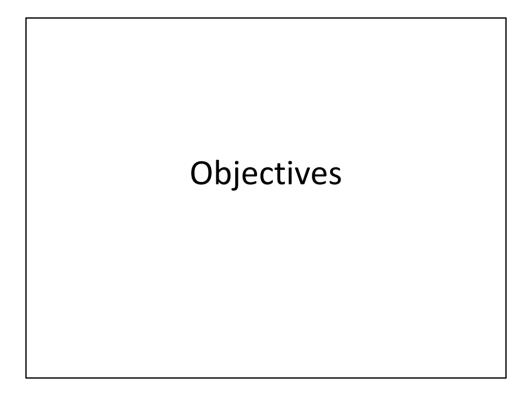
Project Inspiration

- Wang et al. "Deep InP Gratings for Opto-Electronic Devices Etched by Cl₂/CH₄/Ar Inductively Coupled Plasma"
- Cl₂/CH₄/Ar ICP process results
 - High anisotropy
 - High selectivity (SiN mask)
 - High aspect ratio
 - Smooth sidewalls



Project Inspiration

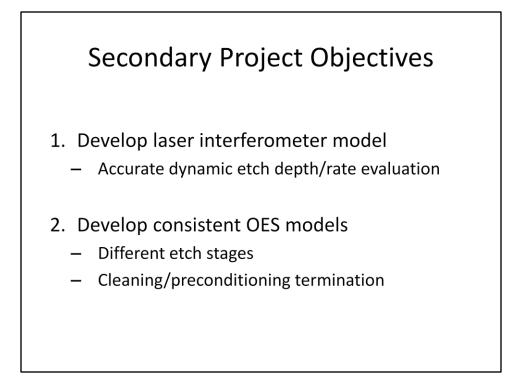
- Wang et al. "Deep InP Gratings for Opto-Electronic Devices Etched by Cl₂/CH₄/Ar Inductively Coupled Plasma"
- Major process drawbacks
 - 1. Inconsistent etch lack of temperature control
 - 2. Unspecified information carrier wafer, InP area



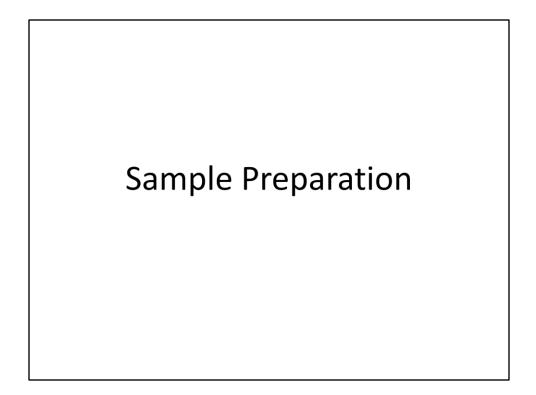
Primary Project Objectives

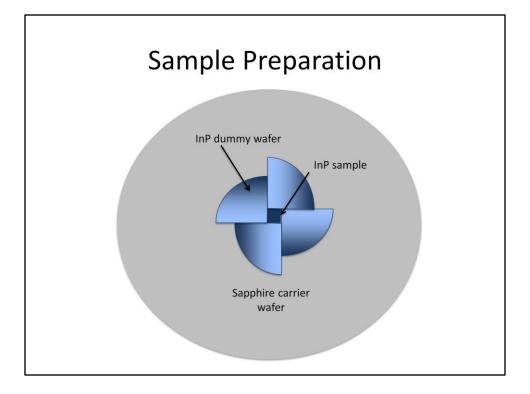
- 1. Develop consistent/controllable ICP recipe
 - Temperature control; crystalbond 590
 - Sapphire carrier wafer
- 1. Obtain InP nanostructures with
 - Smooth sidewalls
 - High anisotropies
 - High selectivities
 - Large aspect ratios

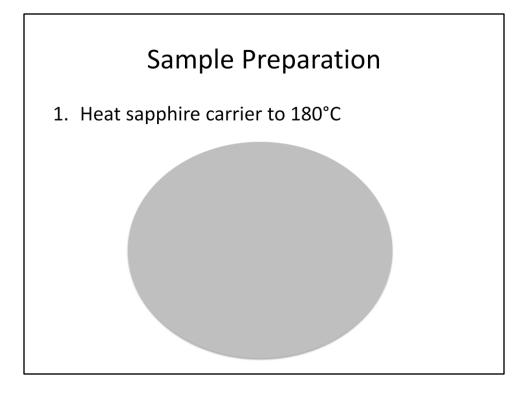
Sapphire carrier wafer usage ensures that passivation is gas-based, not carrier waferbased

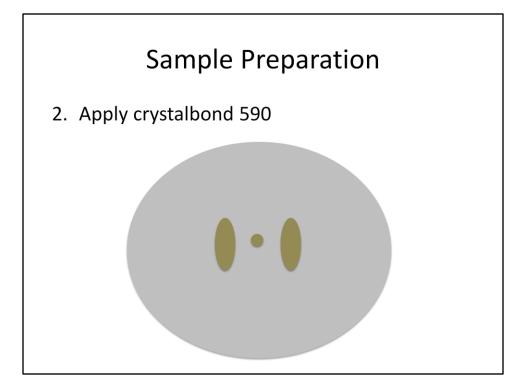


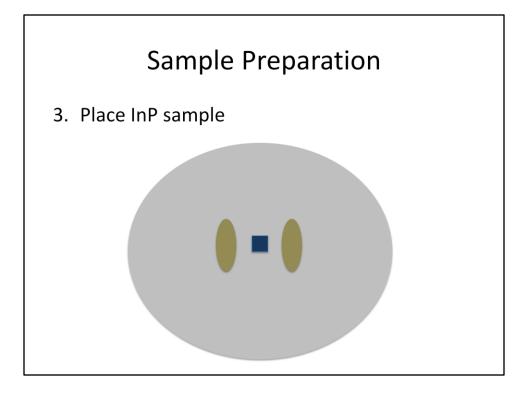
Note: The OES has thus far only been utilized to determine termination time during cleaning and preconditioning phases of the ICP process. However, it can be used for many more applications (i.e. radical generation rate determination, etc)

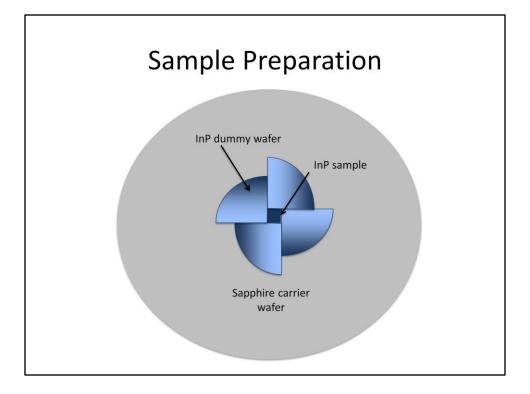


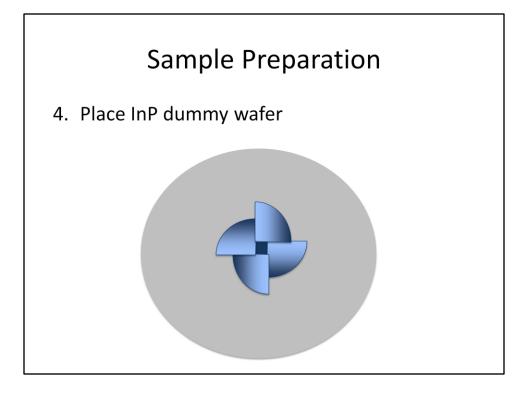


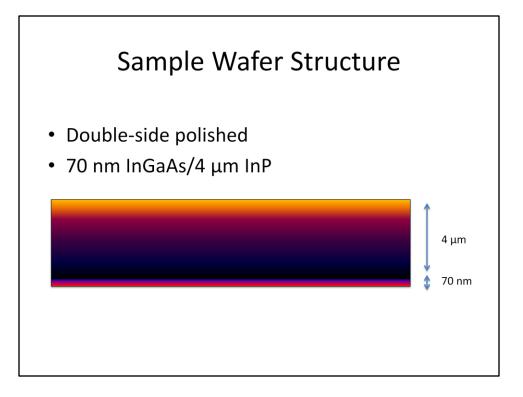


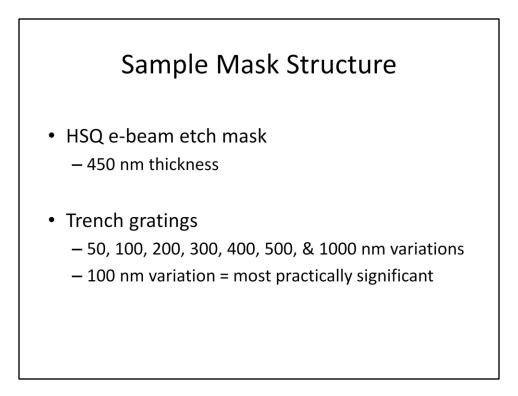




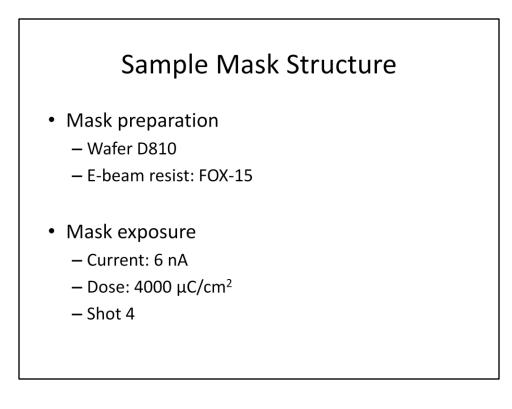




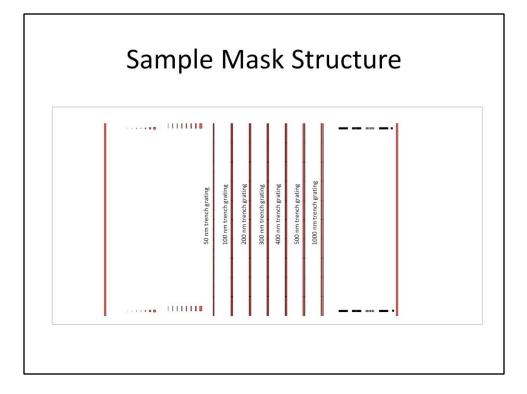




Note: 100 nm trench grating is most practically significant for Bragg mirror applications, and therefore will be utilized throughout the analysis section of this presentation.



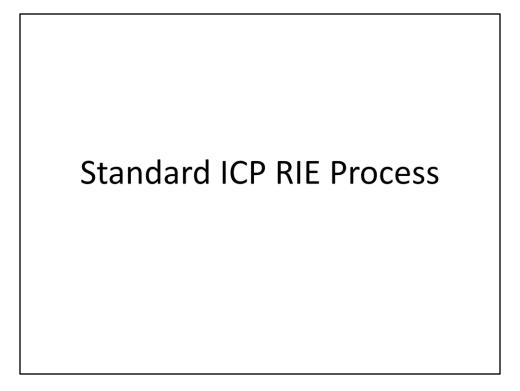
Note: 100 nm trench grating is most practically significant for Bragg mirror applications, and therefore will be utilized throughout the analysis section of this presentation.





Sample Cleaning (Following Etch)

- InP dummy wafers
 - 1. 7 min. $H_2SO_4/H_2O_2 = 4:1$ treatment
 - Removes crystalbond & polymer
- Sapphire carrier wafers
 - 1. 7 min. $H_2SO_4/H_2O_2 = 4:1$ treatment
 - Removes most polymer & other residue
 - 2. 5 min. O2 plasma treatment
 - Removes remaining polymer



Standard ICP RIE Process

1. Increase ICP temperature to 180°C

- 2. 10 minute O₂ plasma clean
 - Wafer: Sapphire cleaning/precondition wafer
 - Purpose: Reduce chamber contamination
- 3. 30 second O₂ plasma clean
 - Wafer: InP sample
 - Purpose: Reduce sample contamination

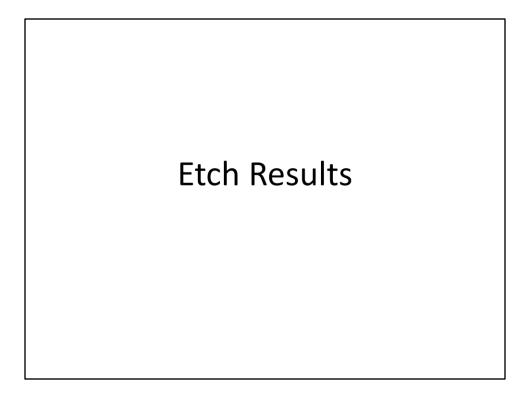
All relevant recipes can be found within the ICP directory on the designated III-V ICP etcher computer: Recipes\users\mhai

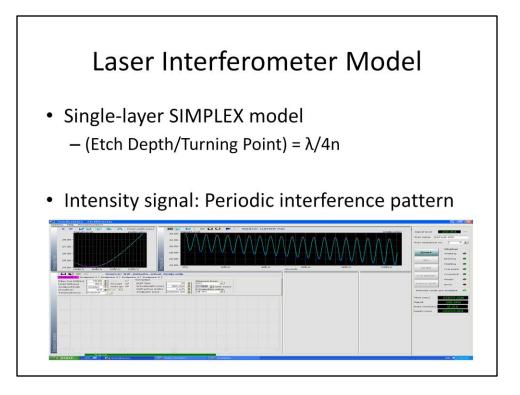
Standard ICP RIE Process

- 4. 2 minute O_2 plasma clean
 - Wafer: Sapphire cleaning/precondition wafer
 - Purpose: Reduce chamber contamination
- 5. 15 minute ICP chamber precondition
 - Wafer: Sapphire cleaning/precondition wafer
 - Purpose: Attain steady-state ICP chamber chemical composition

Standard ICP RIE Process

- 6. ICP reactive ion etch
 - Wafer: InP sample
 - Purpose: Etch InP sample
- 7. 20 minute O₂ plasma clean
 - Wafer: Sapphire cleaning/precondition wafer
 - Purpose: Reduce ICP chamber contamination





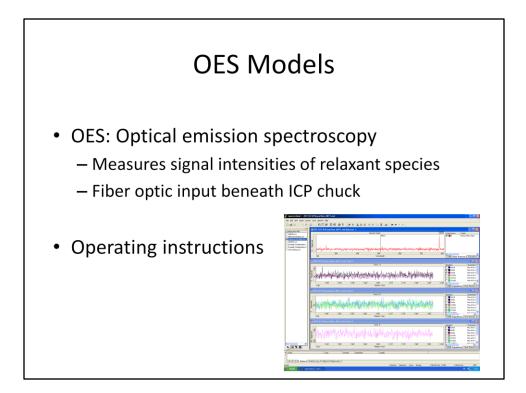
Since the etch process involves only a single materialistic layer, the single-layer SIMPLEX model can be effectively utilized in order to determine the dynamic etch depth of the indium phosphide samples. Should the etch process involve several materialistic layers, a full-scale model would be needed, which requires accurate knowledge of layer thicknesses as well as refractive indices.

In the above equation, λ refers to the light wavelength whereas n refers to the meterialistis refractive index (n~3.900 for InP at a temperature of 180C and a wavelength of 980 nm).

Laser Interferometer Model Relevent user-defined parameters Buffer size Filter cutoff Holdoff time Analysis mode Endpoint Refer to interferometer manual Provides parameter-related information

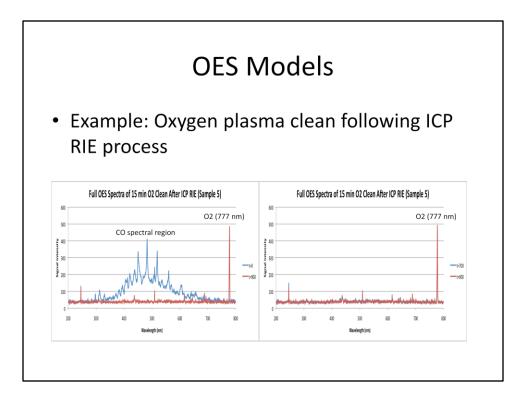
Since the etch process involves only a single materialistic layer, the single-layer SIMPLEX model can be effectively utilized in order to determine the dynamic etch depth of the indium phosphide samples. Should the etch process involve several materialistic layers, a full-scale model would be needed, which requires accurate knowledge of layer thicknesses as well as refractive indices.

In the above equation, λ refers to the light wavelength whereas n refers to the meterialistis refractive index (n~3.900 for InP at a temperature of 180C and a wavelength of 980 nm).



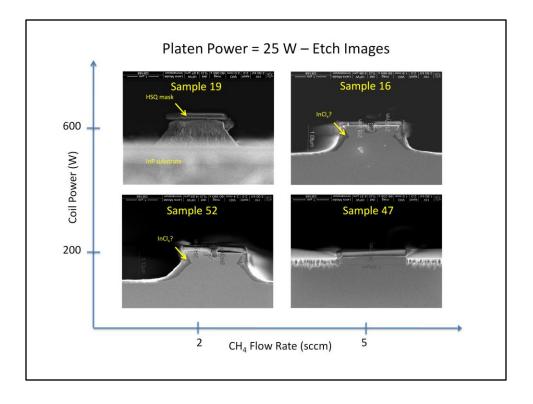
OES Models

- OES data collection
 Each ICP process stage
- Steady-state OES spectrum
 - Cleaning/preconditioning termination



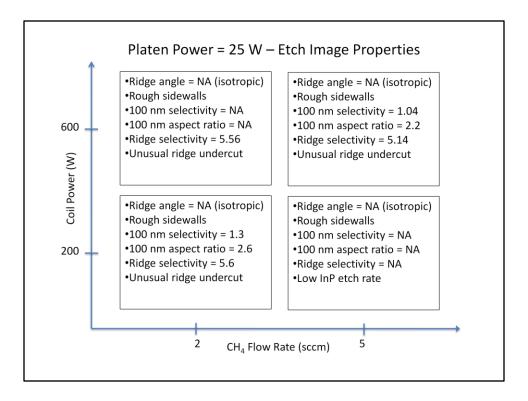
InP ICP Reactive Ion Etch						
Constant ICP Parameters						
- Etch time = 600 s						
Ar Flow Rate	Chamber Pressure	Temp.	Carrier Wafer	Crystalbond	Wafer Area	
2 sccm	1 mT	180°C	Al_2O_3	590 HMP	2″	
	nstant IC tch time Ar Flow Rate	tch time = 600 s	Ar Flow Rate Chamber Pressure Temp.	nstant ICP Parameters tch time = 600 s Ar Flow Chamber Temp. Carrier Rate Pressure Wafer	Ar Flow Rate Chamber Pressure Carrier Wafer Crystalbond Wafer	Ar Flow Rate Pressure Temp. Carrier Crystalbond Wafer Area

Note: Ar flow rate of 2 sccm utilized in order to obtain a stable plasma, low chamber pressure utilized in to order increase InCl volatility, high temperature utilized in order to increase InCl volatility, Al2O3 carrier utilized in order to achieve inert background environment, crystalbond 590 HMP used in order to achieve temperature control as well as sample adhesion, etch time selected based upon previous experimentation, 2" wafer size utilized as it is the largest commercially available InP wafer.

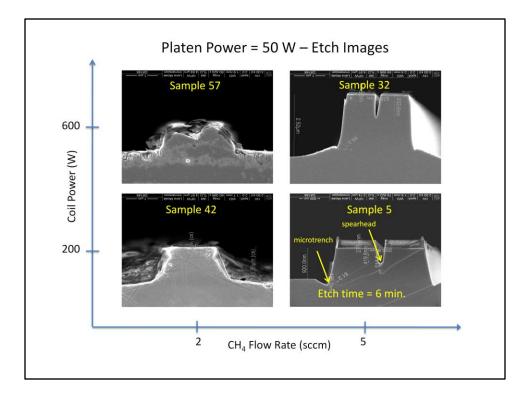


Top left hand corner – High coil power and low platen power, therefore very radicalbased etch. Hence there will be a large concentration of novolatile InCl on the surface of the InP sample, resulting in rough features. Initial undercut of nanostructure may be due to increased sticking coefficient of HSQ, which would localized radicals (chemical-based etch, hence undercut) at the InP-HSQ interface.

Top right hand corner – over passivation due to high methane flow rate; low etch rate Bottom left hand corner – over passivation due to low platen and coil power Bottom right hand corner – over passivation due to high methane flow rate, low coil power, and low platen power; very low etch rate; very rough surface



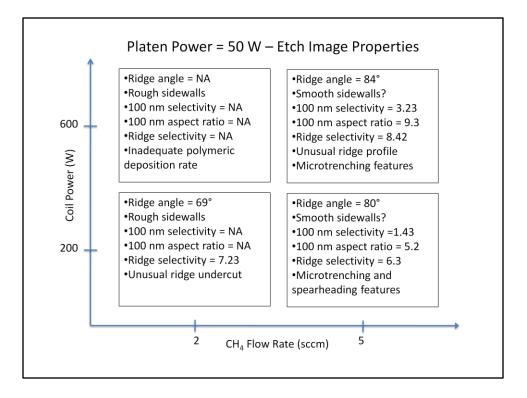
Note: 100 nm selectivity refers to the selectivity within the 100 nm InP trench. Take one of these information blocks – during presentation – and describe what it means.

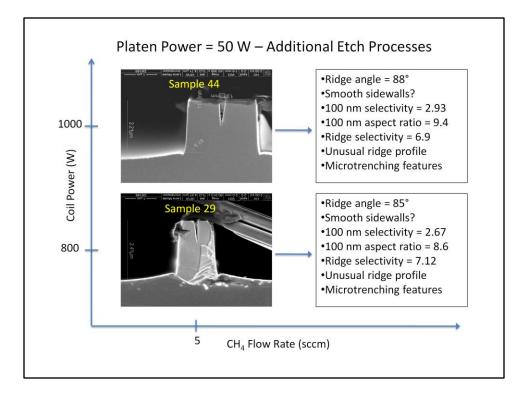


Top left hand corner – Sample was destroyed due to very high coil power, very high platen power, and low passivation rate. HSQ mask was completely removed from sample.

Top right hand corner – High coil and platen power balances the ion/radical ratio, resulting in smooth InP sidewalls, high degrees of anisotropy, high selectivities, and large aspect ratios. Strange vertex profile possibly due to micromasking effect. Bottom left hand corner – Strongly ionic based etch due to low coil power; ions damage sample surface.

Bottom right hand corner – Strongly ionic-based etch due to low coil power, resulting in microtrenching and spearheading features.





Top - High coil and platen power balances the ion/radical ratio, resulting in smooth InP sidewalls, high degrees of anisotropy, high selectivities, and large aspect ratios. Strange vertex profile possibly due to micromasking effect.

Bottom - High coil and platen power balances the ion/radical ratio, resulting in smooth InP sidewalls, high degrees of anisotropy, high selectivities, and large aspect ratios. Strange vertex profile possibly due to micromasking effect.

Conclusions

1. Low platen power – poor etch results

2. Low CH₄ flow rate – poor etch results

3. High platen power & high methane flow rate – superior etch results

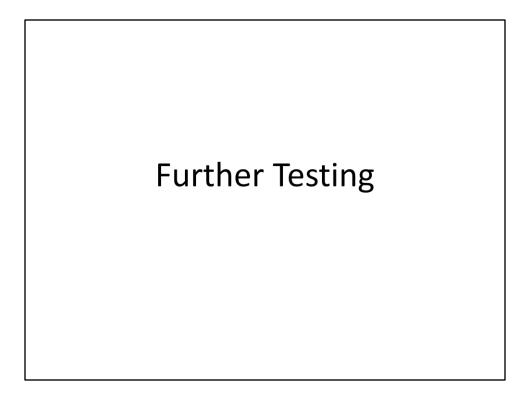
Where "poor etch result" indicates that the desired InP nanostructures features listed in the slide "Primary Objectives" have not been satisfactorily attained.

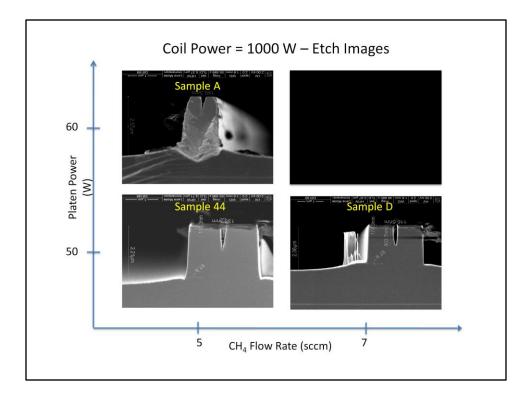
Conclusions

4. High coil power – reduced microtrenching

 InP etch profile – major dependence upon CH₄ flow rate & platen power; minor dependence upon coil power

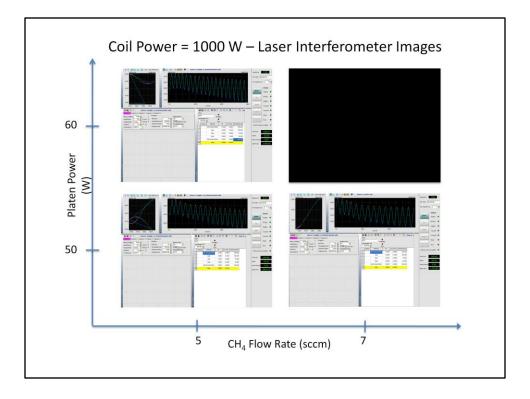
Note: Conclusion 5 refers to the observation that, should un-optimized platen power and methane flow rates be utilized, the resulting profile will not contain the desired nanostructures. Should un-optimized coil power be used, it is still possible to obtain the desired indium phosphide nanostructure profile.





Sample A – Increased platen power to 60 W results in minimal etch differences relative to those obtained from InP sample 44. The trench grating anisotropy appears to worsen, possibly due to scattering effects. Additionally, mask erosion appears to occur. This may be due to mask charging occurring predominantly at the HSQ/InP apexes (which would occur since ions impinging upon the HSQ would try to become grounded by traveling to the InP), which would result in distorted ionic bombardment trajectories. Consequently, mask erosion occurs.

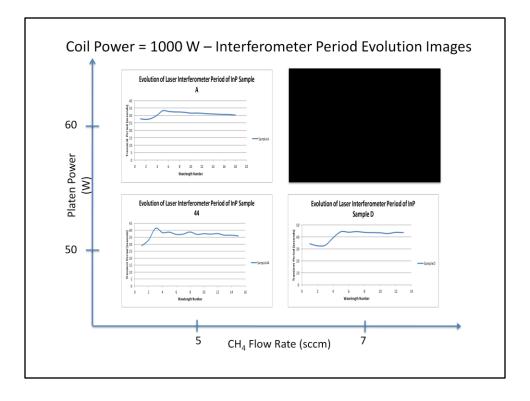
Sample D – Increased passivation (by increasing the methane flow rate) results in a lower InP etch rate, as expected. However, the anisotropy of the 100 nm trench grating significantly increases. This may be due to the increased quantity of methyl radicals molecules formed during the process. These radicals react with the InP, producing $In(CH_3)_x$ molecules, which are much more volatile than $InCl_x$ molecules. Hence increasing the methane flow rate results in a much more chemically (radical)-based etch. Hence, the trench grating profile of InP sample D differs from the triangular profile obtained from the highly ionic-based etch of InP sample 44 due to superior balance between chemical and ionic based etching.



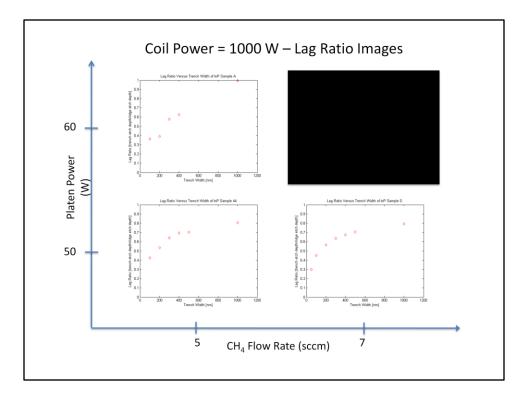
The etch rate of InP sample A is greater than that of InP sample 44 and D due to a higher coil power as well as low methane flow rate (relative to sample D).

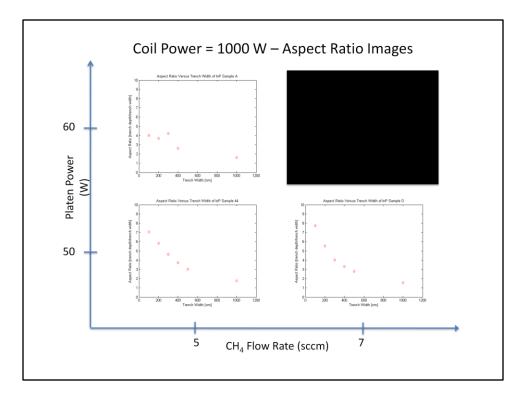
The InP etch rate of InP sample 44 is greater than that of InP sample D due to a lower methane flow rate.

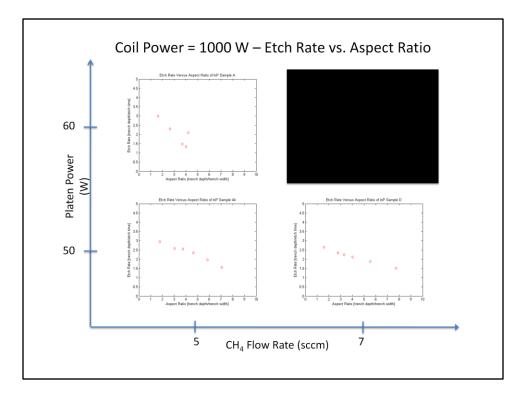
The InP etch rate of InP sample D is low due to a high methane flow rate.

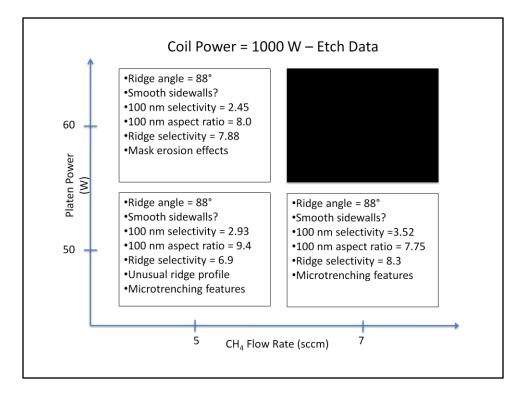


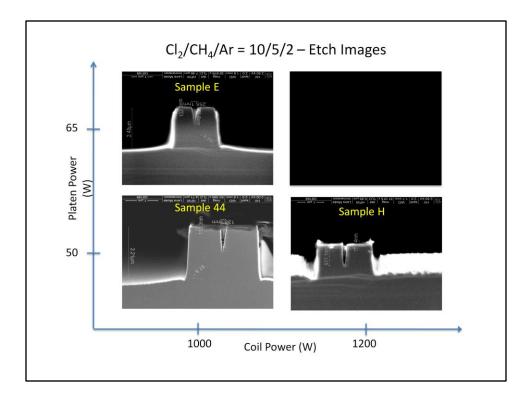
Analysis of the period evolutions of the laser interferometry intensity spectra of InP samples A, 44, and D reveals that the initial etch rate (at the beginning of the etch process) is substantially higher than the rates obtained during the remainder of the process. This may be due to the absence of sapphire carrier wafer preconditioning: At the commencement of the etch process, the sapphire has not been subjected to the deposition of polymeric material. As the etch progresses, polymeric material is deposited onto the sapphire sample. This organic layer is then sputtered - due to ionic bombardment - and is redeposited onto the InP sample, thus reducing its etch rate. Lastly, further inspection of the period evolutions reveals that the etch rates of all samples increases slightly as time progresses. This may be due to the gradual heating of the InP samples.





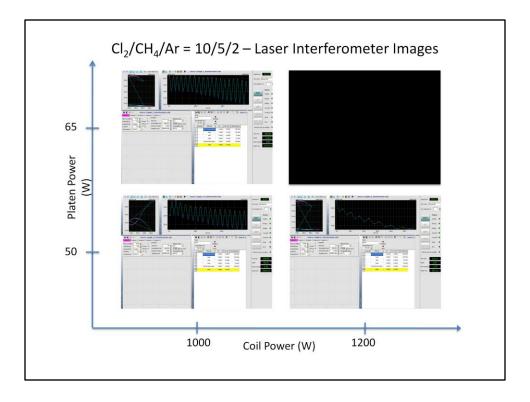






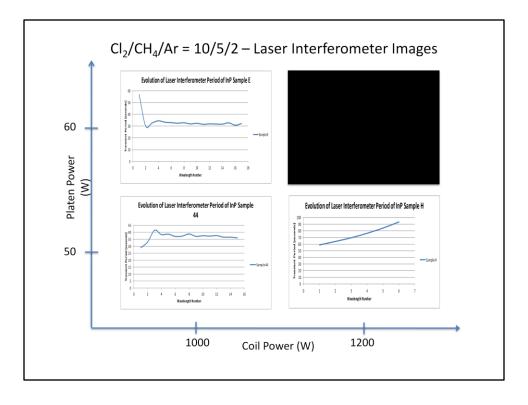
Sample E – Increased platen power to 65 W results in minimal etch differences relative to those obtained from InP sample 44. The trench grating anisotropy appears to worsen, possibly due to scattering effects. Additionally, mask erosion appears to occur. This may be due to mask charging occurring predominantly at the HSQ/InP apexes (which would occur since ions impinging upon the HSQ would try to become grounded by traveling to the InP), which would result in distorted ionic bombardment trajectories. Consequently, mask erosion occurs.

Sample H – Increasing the coil power increases the chemical component of the etch, producing additional chlorine radicals. These react with InP, resulting in nonvolatile InCl_x molecules. This effect should yield an increasingly rough surface, although such extreme roughness was unexpected.



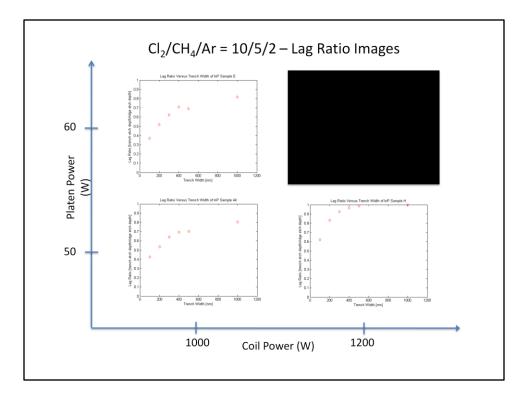
The etch rate of InP sample E is greater than that of InP sample 44 and D due to a higher coil power as well as low methane flow rate (relative to sample D). The InP etch rate of InP sample 44 is greater than that of InP sample D due to a lower coil power, and hence fewer nonvolatile InCl_x molecules present to disrupt the etch process.

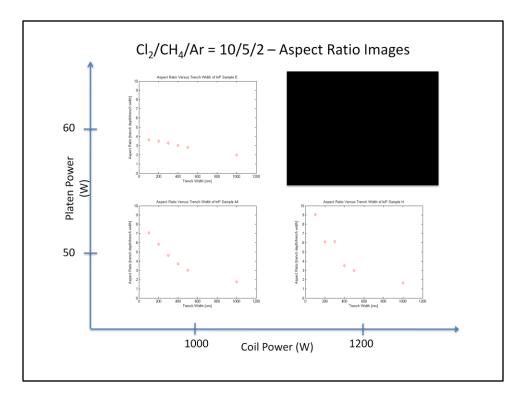
The InP etch rate of InP sample D is low due to a high coil power.

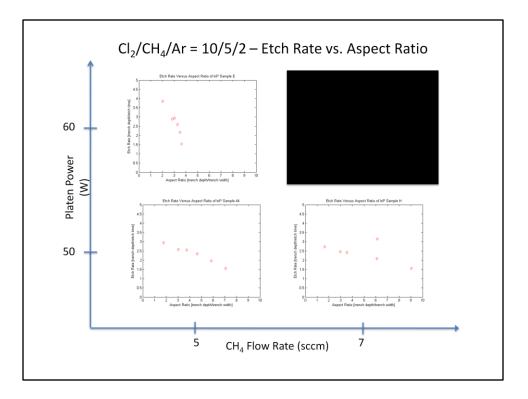


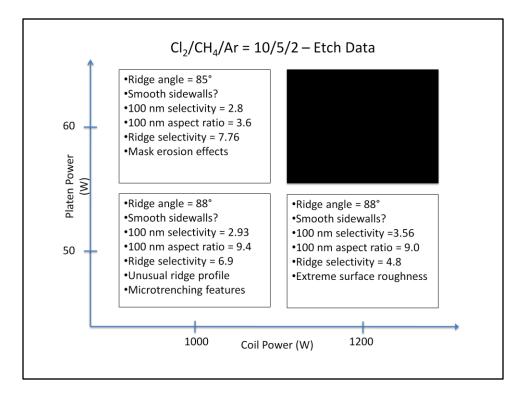
Analysis of the period evolutions of the laser interferometry intensity spectra of InP samples 44 and H reveals that the initial etch rate (at the beginning of the etch process) is substantially higher than the rates obtained during the remainder of the process. This may be due to the absence of sapphire carrier wafer preconditioning: At the commencement of the etch process, the sapphire has not been subjected to the deposition of polymeric material. As the etch progresses, polymeric material is deposited onto the sapphire sample. This organic layer is then sputtered - due to ionic bombardment - and is redeposited onto the InP sample, thus reducing its etch rate. Lastly, further inspection of the period evolutions reveals that the etch rates of all samples increases slightly as time progresses. This may be due to the gradual heating of the InP samples.

The initial, low etch rate of InP sample E may be due to inadequate thermalization; the etch rate increases as the sample temperature increases (due to ionic bombardment, surface chemical reactions, etc).



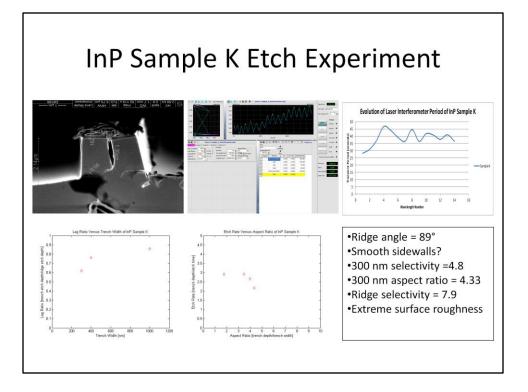




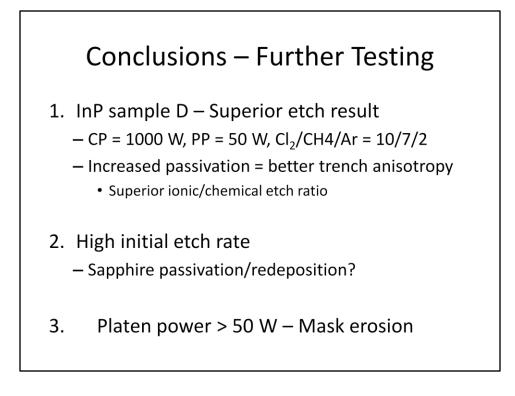


InP Sample K Etch Experiment

- Repeat Sample H Experiment
 - Unexpected extreme surface roughness
 - Verification required
- CP = 1200 W, PP = 50 W, Cl₂/CH₄/Ar = 10/5/2

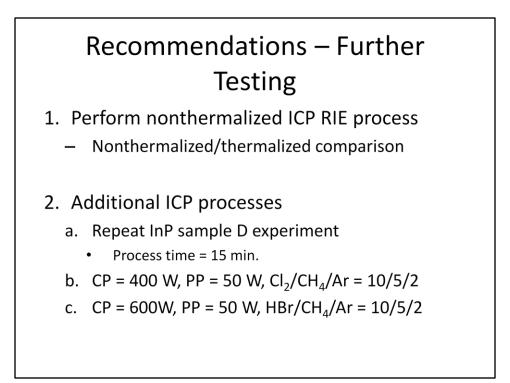


Sample K - Reduced surface roughness relative to InP sample H. Much more desirable etch profile and much more consistent period evolution profile. Experimental repeatability and consistency is therefore questionable.



Note: Sapphire passivation does occur, as evidenced by presence of polymeric material following the precondition stage.

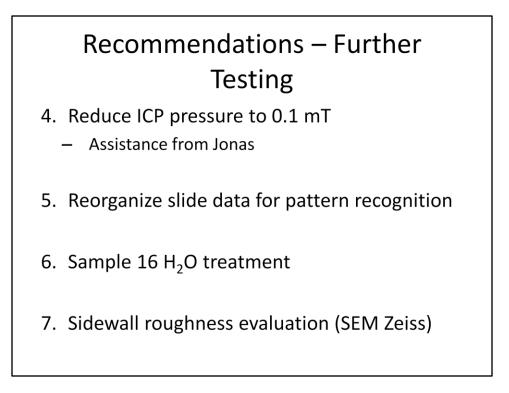
KY: Adjusted CP to 1000W



Repeat InP sample D as it is the best etch result obtained this far.

Perform additional process with CP of 400 W, as ideally the InP etch would be ionbased in order to attain a reduced lag effect. As can be seen from previous experimental results (samples 5, 32, 29, 44), as the coil power decreases, the etch becomes increasingly ionic.

Perform etch experiment substituting HBr for Cl, as HBr has a lower vapour pressure and is thus much more volatile than Cl.



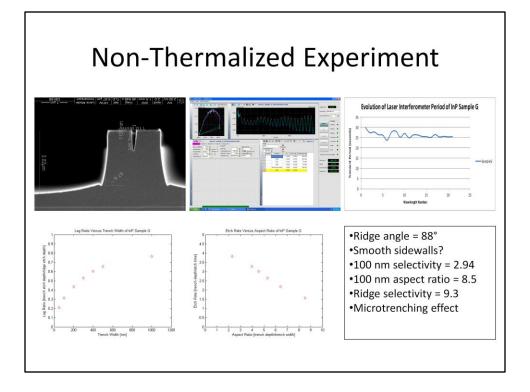
Repeat InP sample D as it is the best etch result obtained this far.

Perform additional process with CP of 400 W, as ideally the InP etch would be ionbased in order to attain a reduced lag effect.

Perform etch experiment substituting HBr for Cl, as HBr has a lower vapour pressure and is thus much more volatile than Cl.

Non-Thermalized Experiment

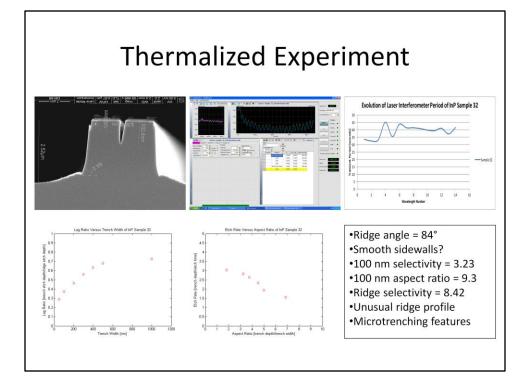
- InP sample G
- CP = 600W, PP = 50 W, Cl₂/CH₄/Ar = 10/5/2
- Non-thermalized
 Absence of crystalbond 590

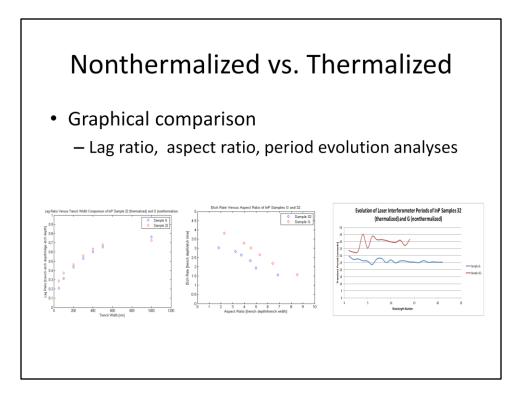


Sample G - Very similar to thermalized experiment. The etch rate during this experiment was higher than that of InP sample 32, likely due to significant sample heating. However, the etch rate was expected to increase during this process as the sample temperature increased; upon analysis of the period evolution of the laser interferometer signal, it can be concluded that this phenomenon did not occur. This observation suggests that significant sample heating did not occur during this etch process.

Thermalized Experiment

- InP sample 32
- CP = 600 W, PP = 50 W, Cl₂/CH₄/Ar = 10/5/2
- Thermalized
 Application of crystalbond 590

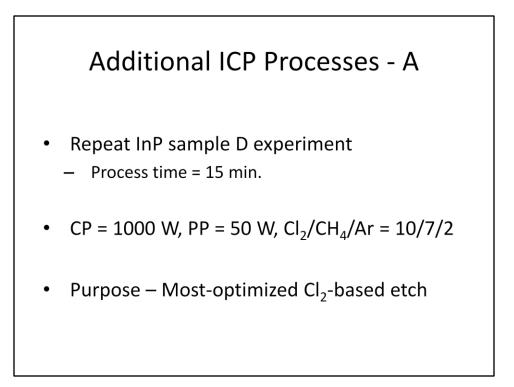




Analysis of the lag ratios of InP samples G and 32 reveals minimal difference. This suggests that sample heating does not significantly alter the lag ratio of the InP nanostructures.

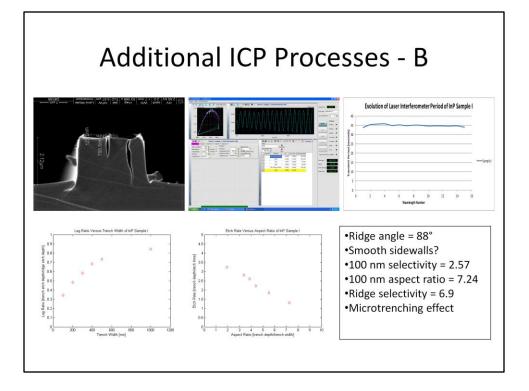
Analysis of the aspect ratios of InP samples G and 32 reveals a significant difference. This suggests that a particular trench of a non-thermalized will subjected to a greater etch depth than that of a thermalized sample, as expected.

Analysis of the period evolutions of InP samples G and 32 reveals a significant difference. This suggests that the etch rate of a non-thermlized sample is greater than that of a thermalized sample, as expected due to sample heating.

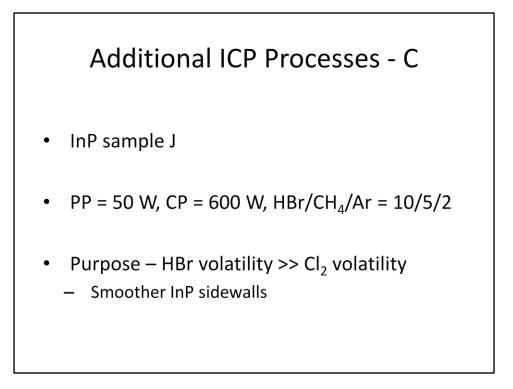


Additional ICP Processes - B

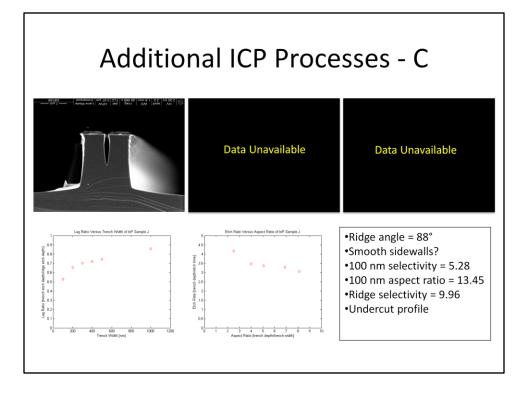
- InP sample I
- PP = 50 W, CP = 400 W, Cl₂/CH₄/Ar = 10/5/2
- Purpose Ionic-dominant etch desired



Sample I – Very anisotropic profile (although significant cracking occurred within this particular trench; this phenomenon was not observed in other trenches, such as 200 nm). Microtrenching effects occurred, likely due to the reduced coil power, resulting in a ion-dominated etch. Additionally, analysis of the period evolution data pertaining to the laser interferometer indicates the sample heating did not occur; this again may be attributable to a reduced coil power



Note: The above ICP parameters were selected for this experiment as they have yielded decent etch results utilizing Cl_2 -based chemistry.



Sample J – HBr/CH4/Ar-based ICP reactive ion etch has yielded very promising results with regards to anisotropy, selectivity, aspect ratio, and lag ratio, and a smooth etch profile. However, a significant and undesirable undercut profile is observed on the exterior and interior of the trench. In order to reduce this effect, the methane flow rate could be increased (resulting in superior surface and sidewall passivation) or the coil power could be reduced (resulting and a more ion-dominant etch and thus reducing the isotropic etch component).

Conclusions

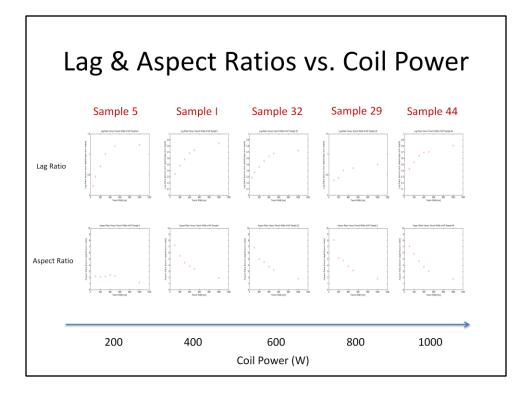
- 1. Nonthermalized etch rate > thermalized
 - Cause: Sample heating
- 2. Lowering coil power reduces chemical etch component
- 3. Promising etch results: HBr/CH4/Ar ICP RIE

Recommendations

- 1. HBr/CH₄/Ar ICP RIE process
 - CP = 600 W, PP = 50 W, HBr/CH₄/Ar = 10/7/2
 - Increased passivation = reduced etch undercut

2. HBr/CH₄/Ar ICP RIE process

- CP = 200 W, PP = 50 W, HBr/CH₄/Ar = 10/7/2
- Reduced CP = ion/anisotropic-dominated etch
- 3. Evaluate lag & aspect ratios vs. coil power



It appears that no discernable relationships exist between aspect and lag ratios as functions of coil power (with Cl2/CH4/Ar = 10/5/2, platen power = 50 W).

InP Sample N

- PP = 50 W, CP = 600 W, HBr/CH₄/Ar = 10/7/2
- Purpose Increased passivation = reduced etch undercut
 - Smoother InP sidewalls

