SEMICONDUCTOR PROCESSING RECIPES
Subjects are ordered alphabetically.

Au wet chemical etches
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13 g KI (potassium iodide) + 7 g Iodine crystals + 250 ml water.
After dissolving the KI in water, the solution may need to be heated to dissolve the iodine. Etch rate is approximately 2000 Angstroms / min. This Au etch does not attack photoresist.

Cleaning and rinsing procedures and hints
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Degreasing of samples:
First, put the sample in a beaker filled with acetone. Second, take the sample out of beaker with tweezers and rinse with a methanol squirt bottle. Third, put the sample on a filter paper and blow dry. Caution: Do not let acetone or methanol dry on wafer surface. Rinse with methanol when there is still acetone on wafer.

Rinse in water:
When rinsing a sample in a water-filled beaker, let the DI water overflow into the sink so that particles on the water surface are washed away.

"Spinning dry" versus "blowing dry":
Drying a sample or wafer by "spinning dry" leaves very clean surfaces. Spin-dried samples are used for epitaxial growth, atomic-force microscopy, or other processes in which the surface quality is very important. Blowing dry is a quick and convenient method which is not as clean as spinning dry but sufficiently clean for most purposes.

Cleaning contaminated beakers:
Here is a cleaning procedure suitable for cleaning beakers: Fill beaker to the rim with 25 % H2SO4 and 75 % H2O (by volume) and leave etch in beaker for 60 minutes. Subsequently thoroughly rinse with H2O.

Cleaning contaminated samples:
Caution: This procedure is not suitable for all samples. Procedure: Clean contaminated sample in 25 % H2SO4 and 75 % H2O (by volume) for 10 minutes in an ultrasonic bath. This should remove most contaminants.

GaAs wet chemical etches
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6 ml H2O + 2 ml H2O2 (30%) + 2 ml H3PO4
This etch is frequently used for mesa etches. Etch rate is approximately 1.5 microns / minute.
Note: Always add acid last to the solution to avoid heating.

76 ml H_2O + 2 ml H_2O_2 (30%) + 2 ml H_3PO_4.
This etch is frequently used for gate recess etches. Etch rate is approximately 1200 Angstroms / minute. Note: Always add acid last to the solution to avoid heating.

10 ml Citric acid + 10 ml H_2O_2 (30%)
This etch is used for gate recess etches. Etch rate is approximately 2 Angstroms / sec for GaAs and 4 Angstroms / sec for $Al_{0.30}Ga_{0.70}As$. This etch has anisotropic etching properties. For more information see Ralph Williams "Modern GaAs Processing Methods" (Artech House, Boston, 1990) Note: Always add acid last to the solution to avoid heating.

GaAs ohmic contacts for n-type materials
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Sample preparation procedure for lift-off:
Leave for 15 sec. in HCl / H_2O = 1 / 1 solution for removal of oxide layer formed on the GaAs surface. Blow dry with nitrogen gas. Mount in evaporator and pump down.

Metallization:
Typical metallizations are AuGe / Ni / Au with 500 Angstroms / 400 Angstroms / 500 Angstroms film thickness. Evaporation rates are 1 - 5 Angstroms for AuGe, 1 - 3 Angstroms for Ni and 1 - 5 Angstroms for Au. The AuGe is a eutectic with a Au / Ge = 88 / 12 weight percent distribution. Required pressure before evaporation is $< 2 \times 10^{-6}$ Torr.

Annealing:
Typical annealing conditions are 420 DegC - 450 DegC for 30 sec.

GaAs ohmic contacts for p-type materials
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Sample preparation procedure for lift-off:
Leave for 15 sec. in HCl / H_2O = 1 / 1 solution. Blow dry with nitrogen gas. Mount in evaporator and pump down

Metallization:
Typical metallizations are AuZn / Au with 500 Angstroms / 500 Angstroms film thickness. Rates are 1 - 5 Angstroms for AuZn, and 1 - 5 Angstroms for Au. The AuZn is mostly Au but contains a few percent of Zn. Required pressure before evaporation is $< 2 \times 10^{-6}$ Torr.

Annealing:
Typical annealing conditions are 400 DegC - 440 DegC for 30 sec.

GaAs Schottky contacts
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Sample preparation procedure:
Rinse with acetone. Rinse with methanol. Blow dry with nitrogen gas. Leave for 15 sec. in HCl / H_2O = 1 / 1 solution. Blow dry with nitrogen gas. Mount in evaporator and pump down

Metallization:
Typical metallizations are Ti / Au with 500 Angstroms / 1000 Angstroms film thickness. Rates are 1 - 3 Angstroms for Ti and 1 - 5 Angstroms for Au. Cr / Au with 500 Angstroms / 1000 Angstroms film thickness. Rates are 1 - 3 Angstroms for Cr and 1 - 5 Angstroms for Au. Required pressure before evaporation is $< 2 \times 10^{-6}$ Torr.

GaAs substrate thinning
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The thinning of GaAs substrates can be achieved by chemically assisted mechanical polishing the substrates in a bromine-methanol solution.

Sample thinning procedure: Attach the sample to the center of a 2-inch diameter quartz disk using crystal wax. Also attach 4 small pieces of a scrap GaAs substrate close to the edge of the disk. Mix a 5 % bromine-methanol solution, e. g. mix 20 ml of bromine with 400 ml of methanol. Take an 8 inch by 8 inch glass plate, cover it with a 12 inch by 12 inch filter paper, and put it on a cafeteria tray in a fume hood. Soak the filter paper with the Br-methanol solution and start a gentle rotating motion to polish the sample. Periodically rinse sample in DI water and then measure the thickness of the substrate until desired thickness is reached. When you get close to the desired GaAs substrate thickness, you may want to further dilute the Br-methanol solution, to obtain a smooth polished surface. Further dilution can be achieved by spaying additional methanol from a squeeze bottle on the filter paper. During polishing, you may need to replenish the Br-methanol solution and/or replace the filter paper if it gets torn.

Warnings: 1. Use double glove protection, that is, wear latex gloves plus the black chemical gloves over the latex gloves. 2. Always wear a lab coat. Br-methanol may etch holes in your clothes. 3. It is a good idea to newly mix Br-methanol solution every time you use it. If you want to keep the readily mixed solution in a bottle, occasionally check for pressure build-up in the bottle. 4. Keep work area in hood clean and organized, since the Br-methanol solution can stain the plastic surfaces of the hood. 5. Never get acetone close to the polishing experiment. Acetone reacts violently with the Br-methanol solution.

$Ga_{0.47}In_{0.53}As$ wet chemical etches
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76 ml H_2O + 2 ml H_2O_2 (30%) + 2 ml H_3PO_4
Etch rate is approximately 900 Angstroms / minute. Note: Always add acid last to the solution to avoid heating.

20 ml H_2O + 1 ml H_2O_2 (30%) + 1 ml H_3PO_4

Etch rate is approximately 3300 Angstroms / minute. Note: Always add acid last to the solution to avoid heating.

8 ml H_2O + 8 ml H_2O_2 (30%) + 1 ml H_2SO_4
Etch rate is XXX Angstroms / minute. H_2SO_4 will not attack photoresist, if sufficiently diluted and if resist is hard-baked. This etch can be used with photoresist. This etch selectively etches $Ga_{0.47}In_{0.53}As$ and it does not etch InP. Note: Always add acid last to the solution to avoid heating.

GaN annealing in N2
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If GaN is treated with acids, the hydrogen coming from the acids may diffuse into the sample. The sample can be annealed at 775 deg C for 300 seconds. As a result the contact resistance decreases significantly. For annealing procedures see Goetz et al. Appl. Phys. Lett. XX XXX (199???)

GaP wet chemical etches
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Mix 2 ml bromine with 200 ml methanol (1 % Br-methanol solution). Etch GaP in beaker without stirring. Etch rate is approximately 0.15 micrometer / minute without stirring the solution. Note: Stirring the solution increases the etch rate.
Note: It is a good idea to newly mix Br-methanol solution every time you use it. If you want to keep the readily mixed solution in a bottle, occasionally check for pressure build-up in the bottle. Suitable masking material: Crystal wax can be used as a masking material.

InP selective wet chemical etches
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10 ml H_2O + 10 ml H_3PO_4 + 30 ml HCl
Etch rate is approximately XXX Angstroms / minute. This solution etches InP but not arsenides such as GaInAs. Note: Always add strongest acid last to the solution to avoid heating.

10 ml H_3PO_4 + 10 ml HCl
Etch rate is approximately 4000 Angstroms / minute. This solution etches InP but not arsenides such as GaInAs. Note: Always add strongest acid last to the solution to avoid heating.

Material Data Safety Sheet (MSDS)
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MSDS provide important information on any material used in semiconductor processing laboratories. Consult MSDS before working with chemicals. MSDS are in a binder in the processing laboratory or can be found on the internet. Example: http://siri.uvm.edu/msds/.

Ni etch
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Ni on GaN and similar materials can be etched with the acid mixture:
We found the following etch to be good: $H_2SO_4 : H_2O = 1: 1$

We found the following etch to be very good especially for samples with annealed Ni (at 400 to 500 degree C): $HNO_3 : CH_3COOH : H_2SO_4 = 5 : 5 : 2$
It takes about 10 to 20 minutes to remove Ni contacts

Un-annealed Ni contacts cam be removed in diluted HNO_3. Use $HNO_3 / H_20 = 1 / 30$. It etches away 500 Angstroms Ni contacts in 1 minute. Ultrasonic bath will remove particle contaminants. Then use $HCl / H_2O = 1 / 1$ in ultrasonic bath.

Ni contact annealing
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???? Ni contacts become low-resistance contacts when annealed in air at 550 deg. C. See report by XXX et al. in Appl. Phys. Lett. XX, XX (1999)

Photoresist
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Shipley 1350 J is a positive photoresist suited for standard lithography
Spinning: 5000 rpm for 30 seconds. Typical thickness 1.4 - 1.6 microns

Shipley 1813 is a positive photoresist suited for small features
Spinning: 5000 rpm for 30 seconds. The typical resist thickness is 1.3 microns

Shipley 4110 is a positive photoresist suited for small features
Spinning: 5000 rpm for 30 seconds. Typical thickness 1.0 - 1.2 microns

Pre- and post-baking:
Typical pre-baking times: 20 minutes at 90 DegC. Typical post-baking times: 20 minutes at 120 DegC. Alternatively, pre- and post-baking can be done directly on the hot plate of a hot-plate oven. One minute on a 90 DegC hot plate is typical for post-baking. Note that the sample must be in direct contact with the hot plate (i. e. no glass slide must be between sample and hot plate).

For lift-off processes:
There are several possibilities to get good lift-off: (i) Use softer post-baking for lift-off processes, for example 20 minutes at 90 DegC. (ii) If resist does not lift off in acetone use a gentle ultrasonic bath. Reduce the 110 V supply voltage to the ultrasonic bath with a "Variac". (iii) The top layer of the resist can be hardened by a 4 minute soak in monochlorbenzene (= photoresist hardener). The monochlorbenzene soak should be done after exposure before development.

Exposure:
Before exposure, remove photoresist bulges from the edges of the sample with a Q-tip soaked in acetone. Typical exposure times are 15 - 50 sec. Make sure sample is in intimate contact with mask.

Development:
Use Microposit developer and water in a 50 / 50 volume percent mixture. The active ingredient in Microposit developer is ammonium hydroxide. Typical development times are 9 - 15 sec. After development rinse thoroughly (at least 30 sec) in DI water.

Photoresist remover:
Photoresist can be removed by an acetone rinse followed by a methanol rinse. If photoresist is not removed by acetone, for example after extensive baking or plasma etching, photoresist remover can be used. The active ingredient in photoresist remover is KOH.

Si wet chemical etch with anisotropic characteristics
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Anisotropic etching of silicon: Silicon (Si) etches anisotropicly in KOH, NaOH, or TMAH. That is, crystal surfaces etch at a very different etch rate. The (001) plane of Si etches much faster than the (111) plane. The etch rate ratio can be as large as a factor of 1000. This ratio and the etch rate also depend on the molar concentration of the aqueous etch and the temperature of the etch solution.

The (111) planes and the (001) planes form an angle of 36 degree.

Etch characteristics and etch rates: Some of the differences in etch characteristics between KOH, NaOH, and TMAH are: KOH has less (001)-vs.-(111) etching selectivity than NaOH. NaOH provides a better surface finish than KOH. TMAH does not etch the mask and has less (001)-vs.-(111) etching selectivity than NaOH.

Typical etch rates for one molar solution of KOH in water at T = 20 DegC (room temperature) is 0.6 microns/hr for the (001) surface and an estimated 0.001 microns/hr for the (111) surface.

Typical etch rates for one molar solution of NaOH in water at T = 20 DegC (room temperature): Si (001) at 0.6 microns/hr; Si (111) at an estimated factor of 10^3 smaller; the SiO2 mask at about 0.0006 microns/hr.

Typical etch rates for one molar solution of NaOH in water at T = 50 DegC: Si (001) at 10 microns/hr; Si (111) at an estimated factor of 1000 smaller; the SiO2 mask at about 0.02 microns/hr.

Typical etch rates for one molar solution of TMAH in water at T = 50 DegC: Si (001) at 7 microns/hr; Si (111) at an estimated factor of 100 smaller; the SiO2 mask at a negligibly low rate.

All etches have a thermally activated behavior with an activation energy of Ea = 0.7 eV. The etch rate depends on temperature according to

$$r = r_0 \exp(-E_a / (kT))$$

With an activation energy of Ea = 0.7 eV, the etching rate doubles every 8 DegC.

Mask: The etch mask commonly used for KOH etching of Si is the thermal oxide of Si, i. e. SiO2. A sufficiently thick mask is needed since the etch also attacks the SiO2, albeit at a much lower rate.

Si - Schottky contacts on Si

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Platinumsilicide Schottky contacts:
Clean Si wafer in BOE before evaporation. Evaporate 1000 Angstroms of Pt. Anneal at 650 DegC in an inert atmosphere (e. g. N2 or Ar) for 10 - 20 minutes for platinumsilicide formation.

SiO2 wet chemical etch

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Buffered Oxide Etch:
BOE (Buffered Oxide Etch) is 1 part 49 % HF and 7 parts 40 % ammonium fluoride. BOE does not attack photoresist. Etch rate is approximately 250 Angstroms / min.

Silver epoxy

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Manufacturer: World Precision Materials Inc., Sarasota, Florida
Silver epoxy # 4898 usable for up to 400 deg. C., Volume resistivity 0.0002 Ohm cm
Mixing ratio: 1 : 1 by weight
Curing times:
15 minutes at 120 deg. C
90 minutes at 80 deg. C
12 hours at 50 deg. C
Potlife of mixed epoxy at room temperature: 3 days

Surface roughening

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Surfaces of semiconductors can be roughened to increase the amount of light that can escape from the semiconductor. Surface roughening alleviates the "light escape problem" occurring in semiconductors with a high refractive index.

Relevant references: Schnitzer et al. "30 % external efficiency from surface-textured LEDs" Appl. Phys. Lett. Vol. 63, p. 2174 (1993). Windisch et al. "Systematic photoluminescence and electroluminescence study of high-efficiency surface-textured thin-film light-emitting structures" Proc. SPIE Vol. 3279 p. 94 (1998). Windisch et al. "LEDs with 31 % external quantum efficiency by outcoupling of lateral waveguide modes" Proc. SPIE Vol. 3279 p. 94 (1998).

Sample preparation: A monolayer of polystyrene can be deposited in two ways: spin-deposition or deposition by pulling it through a polystyrene beads/water solution.

Polystyrene beads are sold by Duke Scientific Inc., Palo Alto, Calif. Phone (650) XXX-XXXX

Ti wet chemical etch

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Buffered Oxide Etch:
BOE (Buffered Oxide Etch, usually used for etching SiO2 on Si). BOE does not attack photoresist. BOE etches a 200 Angstroms Ti film within a few seconds.

Ti evaporation

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Electron-beam evaporation of Ti:
A very good vacuum is required during Ti evaporation. A high oxygen and water partial pressure have deleterious effects on Ti evaporation. The pressure should be in the 10^-7 Torr range before the evaporation is initiated. When Ti evaporates, the pressure should drop due to the Ti gettering effect. Once the pressure has dropped, the shutter can be opened to deposit Ti on the sample.