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Final Phase II Report for SBIR Contract # F49620-99-C-0002

Sponsored By BMDO
Organization: Solar Associates
Principal Investigator: Curt M. Lampkin
Report Date November 3, 2001
Contract Title: "Sputter Deposition Method for III-V semiconductors."

Table of Contents

Please keep in mind that the term "magnetron" can denote either a microwave source or a sputter deposition device. The context should help distinguish between them.

Summary

This contract had 3 goals:

Stage I

1. Build a thin film deposition system capable of low voltage sputtering of materials
2. Prove that at such low sputter voltages sputter yield are at least as high as standard magnetron sputtering
3. Sputter deposit InAs

Stage 2

1. Design auxiliary magnetrons
2. Build a prototype deposition system to use these auxiliary magnetron
3. Deposit InAs with real time control of stoichiometry

Stage 3

1. Build a pilot production deposition system
2. Deposit InAs with high electron mobility

Planned Accomplishments

Stage I

1. I built a proof of concept deposition system capable of low voltage thin film sputter deposition.
2. I proved that low voltage deposition is possible and rates were at least as high as standard magnetron sputtering
3. I sputter deposited InAs.

Stage 2

1. I designed and built auxiliary magnetrons
2. I built a prototype deposition system to use these auxiliary magnetrons
3. I deposited InAs with real time control of stoichiometry and achieved an electron mobility of 2000 Volts/cm^2-second.

Stage 3

1. I designed, but did not build, a pilot production deposition system including a load lock
2. I designed and acquired the parts and software for a computer control system but did not integrate it into the system.
3. No InAs with higher mobility was deposited

Unanticipated Accomplishments

1. I found that the surrogate magnetron is ideally suited for using the MEE method (mobility enhanced epitaxy). This can produce higher electron mobility films than had been anticipated. This is the major accomplishment of this contract effort.
2. I developed a microwave system for lowering plasma voltage. This was substituted for the electron beam emitter originally proposed.

To bring the process to full production capability, four additional process developments are needed:

1. In/As ratio control improved from +/- 25% to +/-1 %. .
2. Higher microwave power absorption
3. Process vacuum lowered from 10^{-5} to 10^{-7} torr
4. Substrate heating to 250 deg. C during deposition

The first 2 items require improved magnetron magnet design. The present magnets are 10 times more powerful (3000 gauss) than in standard sputter deposition systems. These high magnetic fields enable the low voltages and high rates. Such high fields, unless carefully designed, have a large volume enclosing magnetic fields over 50 gauss. I use microwaves to lower the sputter plasma

voltage. Fields higher than 50 gauss can enable microwaves to generate unwanted plasmas anywhere within the magnetic volume. Such unwanted plasmas degrade stoichiometry (elemental ratio) control.

Preliminary design studies have shown how to make the necessary magnet changes to lower the 50 gauss magnetic volume by 7 to 10 times. I have assembled such a magnet and plan on developing it when resources permit.

This process uses cams to synchronize sputter power to separate sectors of Indium and Arsenic. Present cams have an angular position accuracy of 10 degrees. Better angular accuracy is needed and will improve stoichiometry control proportionately.

Thirdly, lowering the process vacuum is not difficult. A liquid nitrogen cooling coil should be installed inside the vacuum chamber to absorb the water vapor. This will also decrease the oxygen levels. Oxygen is a known contaminant of InAs. It degrades the indium and arsenic target surfaces as well.

Finally, heating the substrate to temperatures of 250 degrees C is not difficult. A heated substrate may not be necessary but it might lessen the precision needed for controlling the Indium / Arsenic ratio control. I have a preliminary heater design which will fit the present deposition chamber.

The major effort in the program has been the development of a microwave source to lower the sputter plasma voltage. I originally proposed an electron gun. However, the electron gun had a fundamental flaw. It extinguished the plasma near its insertion point with a resulting non-uniform deposition. There was no known remedy.

In place of the electron gun, I designed a microwave system to inject energy into the sputter plasma. It took about 2 years. Fortunately, microwaves have proven to be very effective.

Patents

I anticipate or have applied for patents in at least these 4 areas:

a. The sector method for MEE
b. Arsenic vapor source
c. Microwave system
d. Auxiliary target design

Bell thin film test technology

One vital aspect of this project was the availability of my co-sponsor Bell Technology's test technology to test the InAs films. Bell's interest in the

success of this project was a great advantage as was the availability of people trained in evaluating InAs.

There were two main test methods. The first method was a 6 -point probe, which was used on freshly deposited films. It measured Hall signal voltage and equivalent device resistance under industry standard conditions of 0.1 Tesla and 0.010 amperes. From the resulting Hall voltage and resistivity readings I could calculate mobility using this equation.

$$\mu = V_H t / \rho B I$$

μ is the mobility, t is thickness ρ is resistivity, B is magnetic field strength and I is the current. Plugging the standard numbers into the equation I got

$$\mu = 2 \times 10^7 \, V_{Hall} / R$$

Where R is the device sheet resistance. In this particular equation mobility is independent of film thickness. I used this equation for all reported mobilities until mid 2001 when I discovered that the factor of 2 should be unity. Mobilities reported in status report #8 as 4000 were actually 2000. However the same factor applies to Bell's InAs also. My conclusion that I made material of equal mobility to Bell's is still true.

Near the end of the project, I found that the 6-point probe gave erroneously high Hall voltage readings if the film thickness varied by more than 20 % over the test span of 1 cm. Many of my films did vary this much either because of the extra plasma mentioned above or earlier from the earlier reported HHC (e-gun) non-uniformity. The mobilities of up to 4000 reported in status report #8 (dated July 5, 2000) were actually 2000. These mobilities values were from actual sensors made from my films so the 6-point probe error didn't apply. The higher mobilities reported in status report #9 (dated April 7, 2001) were wrong. They were measured only with the 6-point probe. As a reference point, Bell's own InAs films made by CVD vary in mobility from 900 to 2000 cm^2/volt second.

The second and most accurate method was to make actual sensors from the material and then probe test the sensors under computer control. The reported mobility of 2000 was measured by making actual devices.

Background

Phase I Precursor Contract Results.

The Phase I effort proposed that negative ions were damaging to thin films and that low plasma voltage would greatly decrease that damage.

The phase I effort attempted to lower the plasma voltage by simply using a very high magnetic field to contain the sputter plasma. The Phase I showed that the voltage magnetic field relationship was not linear but hyperbolic. The plasma voltage would not go below 200 volts for any magnetic field over 3000 Oersteds. A field of 5000 Oersteds was achieved and 7000 might be possible with extraordinary measures. For reference, 1 gauss = 1 Oersted in vacuum or in air.

However this effort did show that strong magnetic fields provided very low plasma dynamic impedance. Once the plasma ignited at 200 to 300 volts (depending on the material to be sputtered) then the current could be significantly increased with little further increase in plasma voltage. In many cases slight negative impedance was measured. Such low impedance was necessary for the efficient addition of external energy to the plasma.

Basis for Phase II Proposal

The literature described an electron beam as being effective in lowering plasma voltage for the standard low magnetic field sputter magnetron. (1). But with high plasma impedance, the voltage increased several hundred volts whenever useful currents were drawn.
Simultaneously, another technology became evident in the literature. Ion bombardment was known to improve thin film quality, most significantly by lowering the void fraction. The films were very dense, mechanically robust, and did not absorb water. Experience with ion bombardment stimulated further theoretical work. Muller (1) predicted the possibility of sputtering epitaxial films *if the* arriving ad-atoms themselves have 1 to 10 e.v. energy. (For reference, a typical thermally evaporated ad-atom arrives at the substrate with about 0.1 e.v. of energy).

When combined with the surrogate magnetron and the phase I results, I felt that with the confluence of all these new developments, the sputtering of thin films of semiconductors was possible.

Theory

Standard planar magnetron sputtering:

A planar magnetron is essentially a water-cooled box. One side of the box is made the material (called the target) to be deposited. There is a magnet behind the

target, which forms a round or rectangular closed magnetic "tunnel". This tunnel is termed "the racetrack". The magnetron is placed in a metal vacuum chamber with argon gas inside at a pressure of 1 to 10 millitorr. When a negative voltage of about -500 volts is applied to the target, a bright glowing plasma appears at the target surface.
The magnetic racetrack captures and traps electrons by forcing them into long helical paths around the racetrack. This lengthened path increases the collision frequency of electrons with argon atoms and improves ionization efficiency. Figure 1 shows a standard planar magnetron

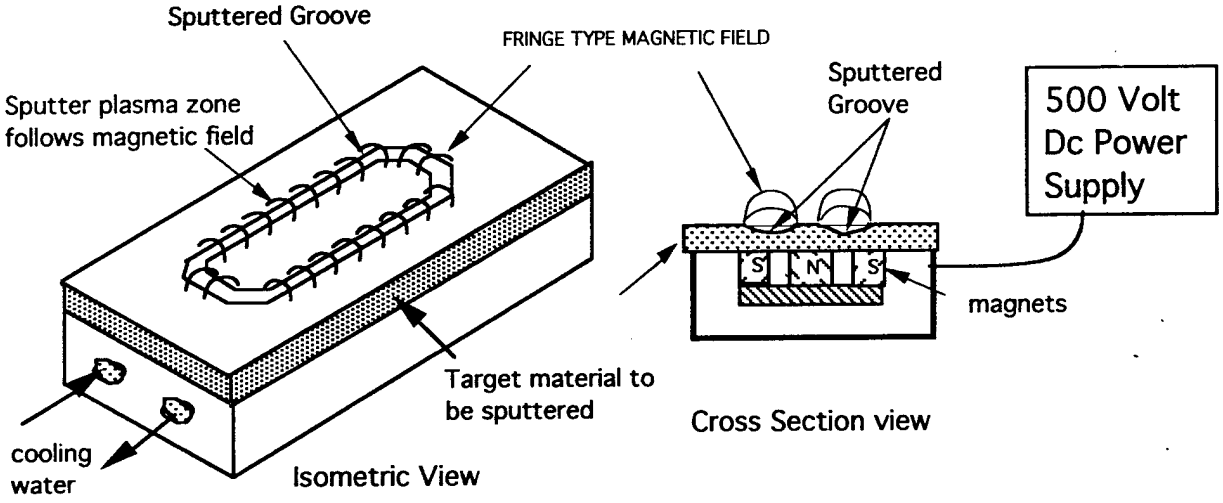
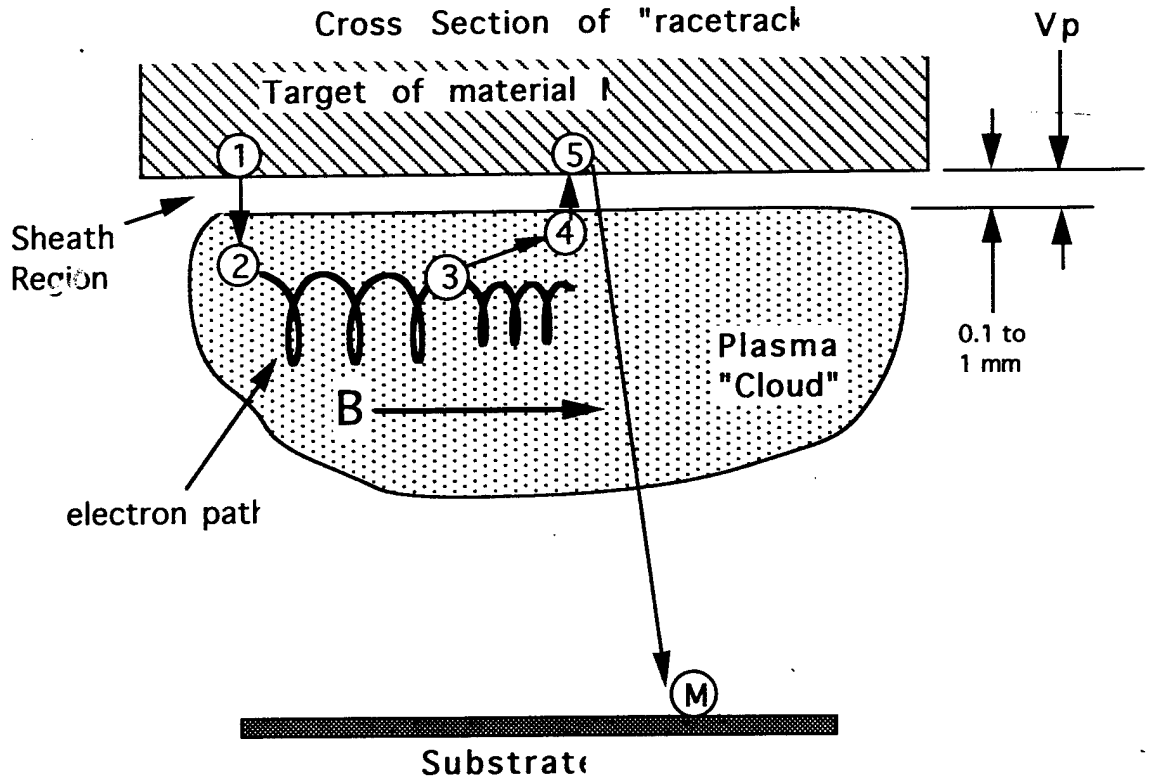


Figure 1 A planar magnetron

Positive argon ions generated by the spiraling electrons are attracted to the negative target. The impact knocks off (sputters) atoms of target material, which travel to the substrate and coat it.

Sputtering Theory

This next figure shows the essential parts of the sputter process.



The sputter plasma zone is a glowing "cloud" of neutral gas atoms, gas ions and electrons. It is contained by the magnetic "racetrack". It does not touch the target but is separated from the target by a "dark space" or "sheath" of 0.1 to 1 mm thickness. The plasma is 5 to 10 volts positive above chamber ground potential. The voltage Vp across this dark is equal to the target voltage plus the plasma potential. Inside the plasma there is no electric field.

The process starts with an electron emitted from the target surface at point 1. The electron is accelerated across the sheath into the plasma with the full energy of Vp. Once inside the plasma it sees no electric field and it starts to follow a helical path in the magnetic field at point 2. At point 3 it hits an argon gas atom and ionizes it. The ion diffuses inside the plasma until it reaches the sheath at point 4. There it is accelerated by the field from Vp and crosses the sheath to strike the target at point 5. There an atom of the target is knocked off or sputtered. It leaves the target at a random angle and may strike the substrate or the chamber wall.

In 5 to 10 % of the argon ion impacts, a secondary electron is also emitted. This electron is accelerated away from the negative target and enters the plasma where it creates more argon ions to maintain the plasma.

The sputter plasma voltage can be easily calculated. The sputter threshold voltage is approximately 25 electron volts depending on the material. It takes this much impact energy to remove an atom of target material by collision with a

positive argon ion. But only 8 percent (approximately) of ion collisions generate a secondary electron. To compensate for the small secondary electron yield, each secondary electron must be given enough energy to ionize 12.5 argon ions (1/0.08 = 12.5). These secondary electrons are the only means of generating more argon ions to sustain the plasma. It takes about 25 electron volts to ionize an argon ion, depending on the pressure and the power level of the plasma during operation. To maintain the plasma then, we must supply a target voltage of 12.5 x 25 or 312 volts. This voltage is needed to just ignite the plasma.

When additional current is drawn through the plasma, the voltage increases markedly indicating high dynamic impedance. The added power heats the gas and the resulting gas rarefaction effect [2] decreases the probability of an electron-gas atom collision. This is the situation in the standard sputter model. Such a model uses magnetic fields of about 100 to 300 gauss. Stronger fields provide no perceived advantage but do carry a penalty. The stronger fields erode the sputter groove more rapidly and the target must be replaced sooner, before the erosion groove penetrates the target.

If stronger fields could be used then the plasma volume would be smaller and more intense. This results from the smaller helical electron paths in such larger fields. The electron helical (or Larmor) radius is given by

$$R = 3.37\ (E^{0.5})/B$$

where E (in electron volts) is the energy of the electron perpendicular to the electric field and B is the magnetic field in Gauss. A smaller, more intense, plasma volume can operate at a lower pressure. Such a small plasma with smaller boundary area and operating at lower pressure probably sees less of the gas rarefaction effect. The dynamic impedance then decreases. Dynamic impedance is also the slope of the IV curve of the plasma. My previous work and the literature all show that higher magnetic fields lower plasma voltage and plasma impedance. My phase I effort showed a practical lower limit of 200 to 250 volts for the plasma voltage.

Rotating cylindrical magnetron

There is a second type of sputter magnetron, which can easily use very strong magnetic fields, the rotating cylindrical magnetron. Here there is no erosion groove as in the planar magnetron. Rotation of the targets cylindrical surface carries all parts of the surface through the sputter plasma completely eliminating any erosion groove.

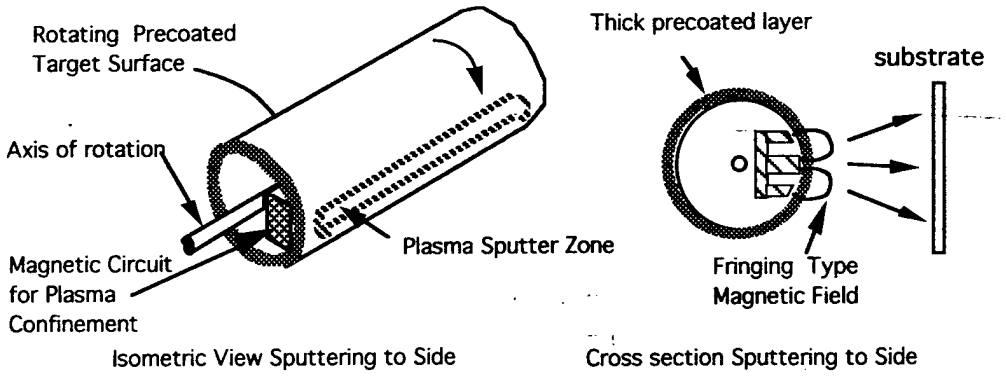


Figure 2. A rotating cylindrical magnetron

The material to be sputtered is pre-coated onto the surface of the cylinder. Rotation then continually moves material through the stationary plasma sputtering zone.

This type has three advantages. One is the aforementioned ability to use high magnetic fields. Second is high material utilization, 85% compared to only 25% for a planar magnetron. The third is less arcing. In a stationary planar erosion groove, partially oxidized or insulating material can accumulate at the edge of the "racetrack". These accumulations build up a positive charge and then discharge in an arc. The rotating cylindrical target has no racetrack "edges".

Problems with prior art sputtering

A. Negative Ion and High Energy Neutral Bombardment

Magnetrons can sputter deposit metals, metal alloys, and metal oxides efficiently but cannot sputter non-metals or many semiconductors. Metals remain neutral during the sputter process. But non-metals or semi-metals (such as arsenic or oxygen) can accept electrons and become negative ions.

These negative ions are immediately repelled from the negative target with the full energy of the target voltage (about 500 volts) and travel at high speed to the substrate where they damage the film. Along the way some are neutralized in the plasma region giving up the extra electron and becoming high-energy neutral atoms. Present data indicates that all non-metals generate these high-energy neutrals and negative ions to some degree.

Hybrid deposition methods avoid the high-energy neutral bombardment by sputtering the metal and thermally evaporating the non-metal. Hybrid methods also allow real time control of stoichiometry. The fact that hybrid methods are necessary and effective shows just how much damage is caused by high-energy

neutrals / negative ions. For example, efficient II-VI thin film solar cells cannot be sputtered at all unless the hybrid method is used.

Transparent conducting oxides (TCOs) also suffer from negative ion effects since Oxygen is very electronegative,

The need for adding external energy to the plasma.

As I showed above, magnetic fields cannot lower plasma voltage much below 250 volts. There are not enough secondary electrons to lower the voltage more than that. The remedy is to add more energetic electrons or to add energy to the electrons already present.

Low voltage sputter yield

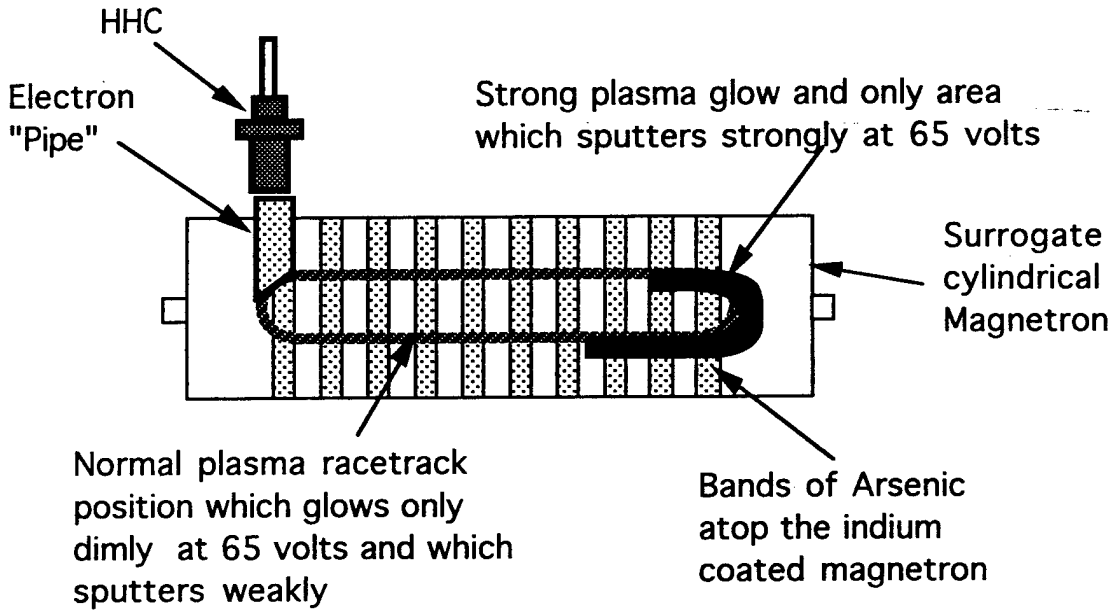
In the proposal for this contract I made a good case for expecting high sputter yield at low plasma voltages. This was proven with the HHC when I made InAs films at high rates. This work also verified the literature prediction that Arsenic sputters about twice as efficiently as Indium. This 2 to 1 ratio was used for the initial In/As ratio control calculations. Later I determined the ratio to be 2.5 to 1.

Methods

Electron Gun Approach

The electron gun adds electrons with about 25 to 35 ev of energy to the plasma. Each one of these electrons can ionize only one argon ion. To decrease the voltage by half, roughly 10 times as many of these low energy electrons must be added to the plasma as there are high energy electrons already present. The voltage decrease is proportional to the added electron current.

As this work progressed it became obvious that the e-gun was not satisfactory. The high magnetic field made it difficult to inject electrons into the plasma. This next figure shows the effect of the e-gun on the sputter plasma. The e-gun was biased about 50 volts negative with respect to the plasma. This meant the e-gun partially extinguished the electrostatic field necessary to support the sputter plasma.



The electron" pipe" was a metal tube, which contained and channeled the electrons into the high magnetic field region. Whenever others have used this scheme it has been with lower magnetic fields. Even so, they found it best to make a miniature tantalum hot hollow cathode and place it directly in the plasma. I could not duplicate that technology and used instead a commercially available electron source from Electric Propulsion Laboratories in Monument CO.
I assume other investigators saw the same plasma extinction effect but to a much lesser degree since they use much weaker magnetic fields,

As will be seen later microwaves were able to add energy to the electrons in the plasma.

The surrogate magnetron

A surrogate magnetron is shown next in this next figure. It is similar to the rotating cylindrical magnetron with one important difference. The surrogate is not pre-coated with the material to be sputtered but instead is coated in the vacuum chamber during the deposition process. This is the type used for this contract effort.

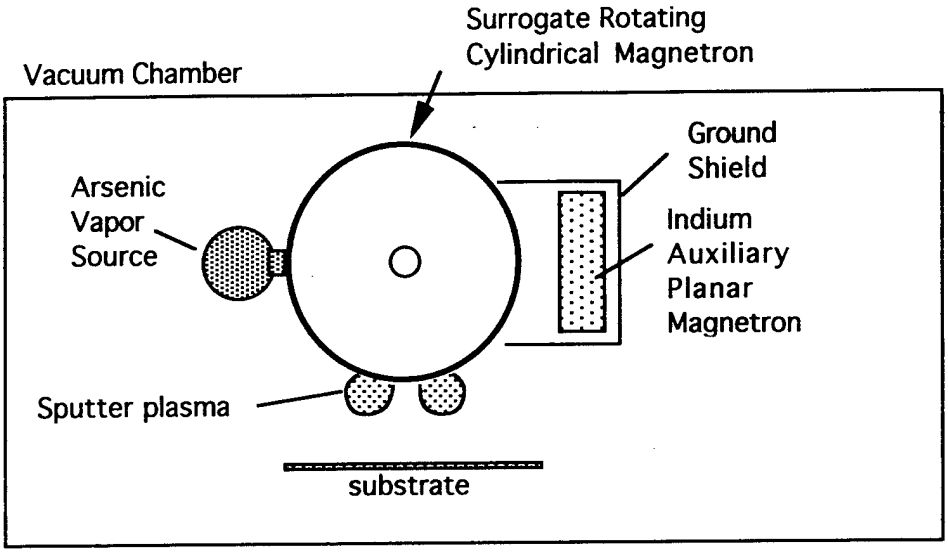


Figure 3 A surrogate magnetron

A vapor source and an auxiliary sputter target are shown.

Initially, the auxiliary Indium magnetron had not yet been built. Instead I had a surrogate coated with a thin layer (0.6 mm) of Indium. The experimental setup was as shown above but without the auxiliary Indium magnetron. I had no arsenic target either so I designed and built an arsenic vapor source. This vapor source could condense a 2 micron thick film arsenic onto the 0.6 mm thick Indium layer. The indium thickness of 0.6 mm did not significantly diminish the magnetic field.

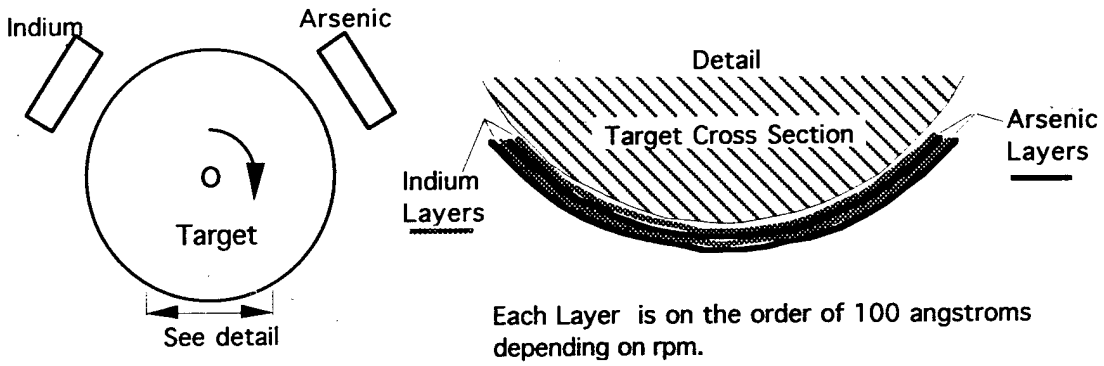
Methods for combining elements onto a surrogate target.

When using a surrogate magnetron, there are three ways to combine materials, in this case arsenic and indium. The three methods are:

- Jelly Roll
- Stripes
- Sectors

Jelly Roll Method.

The original intent was to use 2 auxiliary targets to deposit thin continuous layers in a "jelly roll" fashion onto the surrogate. This figure shows the relationship of the layers in the "jelly roll".



Each Layer is on the order of 100 angstroms depending on rpm.

The layer thickness here has been greatly magnified for clarity

A layer of Indium is deposited on top of a layer of Arsenic on top of Indium and so on. To start the process, the surrogate target would be rotated while the auxiliary targets built up several hundred layers. The surrogate target is ground potential at this point, not sputtering any material from itself. These initial layers would act as a buffer to correct for power variations during the sputter deposition from the surrogate. After the buffer coatings were built up, and while the auxiliary targets were continuously sputtering, the surrogate sputter power would be turned on and deposition begun.

The separate layers on the surrogate are thin, on the order of 10 to 100 Angstroms. Such thin coatings have three advantages. First, thermal conductivity is very high. This removes any thermal stresses and allows much high sputter power levels for higher deposition rate. Second, thin coatings allow the use of high magnetic fields, which in turn allows the high magnetic fields. This enables low voltages, which are the solution to the high-energy neutral bombardment problem. Third, layers of poorly electrically conducting materials such as lightly doped silicon or chalcogens could be sputtered with DC power.

There were two reasons for not using this " jelly roll" method. First, auxiliary target were not available until two years after the contract started. Second, it is difficult to use unless the sputter rates are uniform over the lengths of both the surrogate and auxiliary targets. The magnetic field design was acceptable. But these magnetic field designs require "tuning up" in actual use to get to the 1 to 2% uniformity needed. As it turned out, neither the arsenic or indium targets were usable as they were received. So no tuning up was possible. It could be done in the future when the target problems are solved.

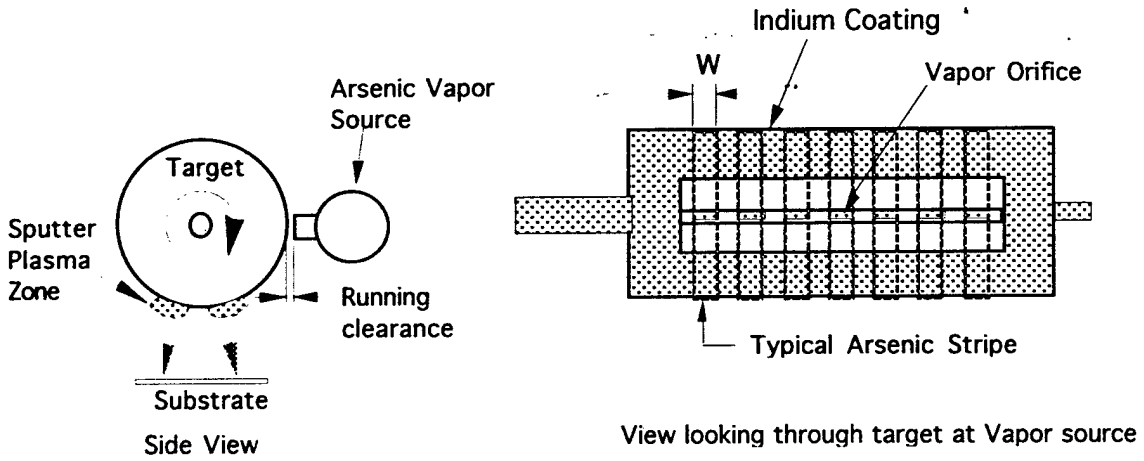
Because of this I had to develop substitute methods, first the stripe method and later, the sector method.

Stripe Method

The stripe method used a target pre-coated with 0.6 mm of Indium (as was mentioned above). An arsenic vapor source with a linear sequence of slits was placed very close, and parallel to, the surrogate target cylinder.
This thermal vapor source deposited about 2 micron thick stripes of arsenic onto an indium coated cylindrical target. I could vary the In/As ratio by mechanically controlling the size and number of baffles in the slot in the arsenic vapor source. The vapor source had a 1.5 mm slot 6 inches long. I simply placed small metal blocks or baffles into the 150 mm (6 inch) long slot. For instance, 7 blocks each 13 mm wide would have a total width of 91 mm. That left 8 spaces with a total width of 150 - 91 = 59 mm. The indium to arsenic width ratio is 59/91= 0.65. I discovered that at low plasma voltages the arsenic sputters 2.5 more efficiently than Indium. So this factor was taken into account.

However, this was a crude mechanical method and difficult to control. The metal blocks required a tedious installation process. Additionally, because of vapor leakage around the edges of the orifices the stripe width varied along the length of the target. The stripe width also varied with sputter deposition time. The In/As ratio varied throughout the deposition because the stripes were wider at the base than at the top. They became narrower as they sputtered away. Thus the stoichiometry varied on all three axes. The sum of these effects was that the In / As ratio could not be controlled to better than an estimated +/- 20%.

This figure shows the stripe method configuration.



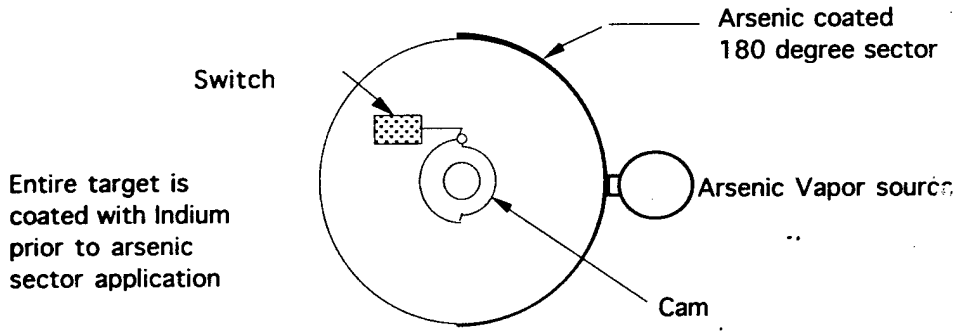
The stripe method eventually did make a thin film sample with mobility of 2000 when it was used with the microwave source. However the method was so crude that a better method was sought.

Sector Method

The sector method was developed near the end of the project. It solved the uniformity problems inherent in the "jelly roll" and stripe methods. It also had another, extremely important advantage. It made it possible to deposit separate monolayers of Indium on top of monolayers of Arsenic. This is called migration enhance epitaxy or MEE. Usually it can be done only with molecular beam epitaxy (MBE) or atomic layer epitaxy)(ALE system. With MBE the arriving ad-atoms have only thermal energy. But sputter MEE they have the 1 to 10 ev energy of sputtered ad-atoms. When perfected, this should allow MEE to be done at 100 to 1000 times faster deposition rates than MBE. It should also permit lower substrate temperatures during deposition.

The sector method got its name from the use of sectors of the desired materials on the surrogate magnetron.

The next figure shows the formation of the sectors. The present configuration is for experiments only. . But it could be scaled up as will be shown later. It used a pre-coated indium target, which is coated, in the deposition chamber with enough arsenic for 4 or 5 depositions. A cam and toggle switch arrangement controlled the drive motor. It rotated at a constant speed and reversed at each cam position.



The arsenic vapor source is a close fitting thermally heated can with a loading port for charging with arsenic pellets. It is indirectly heated with Infra-Red heat and operates at 400 degrees C. The narrow slit in the source was placed within 0.2 mm of the surface of the water-cooled surrogate magnetron. This was the same source used for the stripe method but without the metal blocks to configure the slot into separate orifices.

The surrogate was briefly pre-sputtered prior to coating to enhance arsenic adhesion. When the thermal source heated, arsenic vapors continually condensed onto the surrogate. Target rotation was limited to 180 angular degrees. A cam and toggle switch reversed the motor at each angular limit. This provided constant angular velocity necessary for a uniform arsenic coating over the 180

degree sector. The other 180 degrees was left uncoated and provided the indium for the deposition. The target rotated at 3 rpm for this arsenic coating.

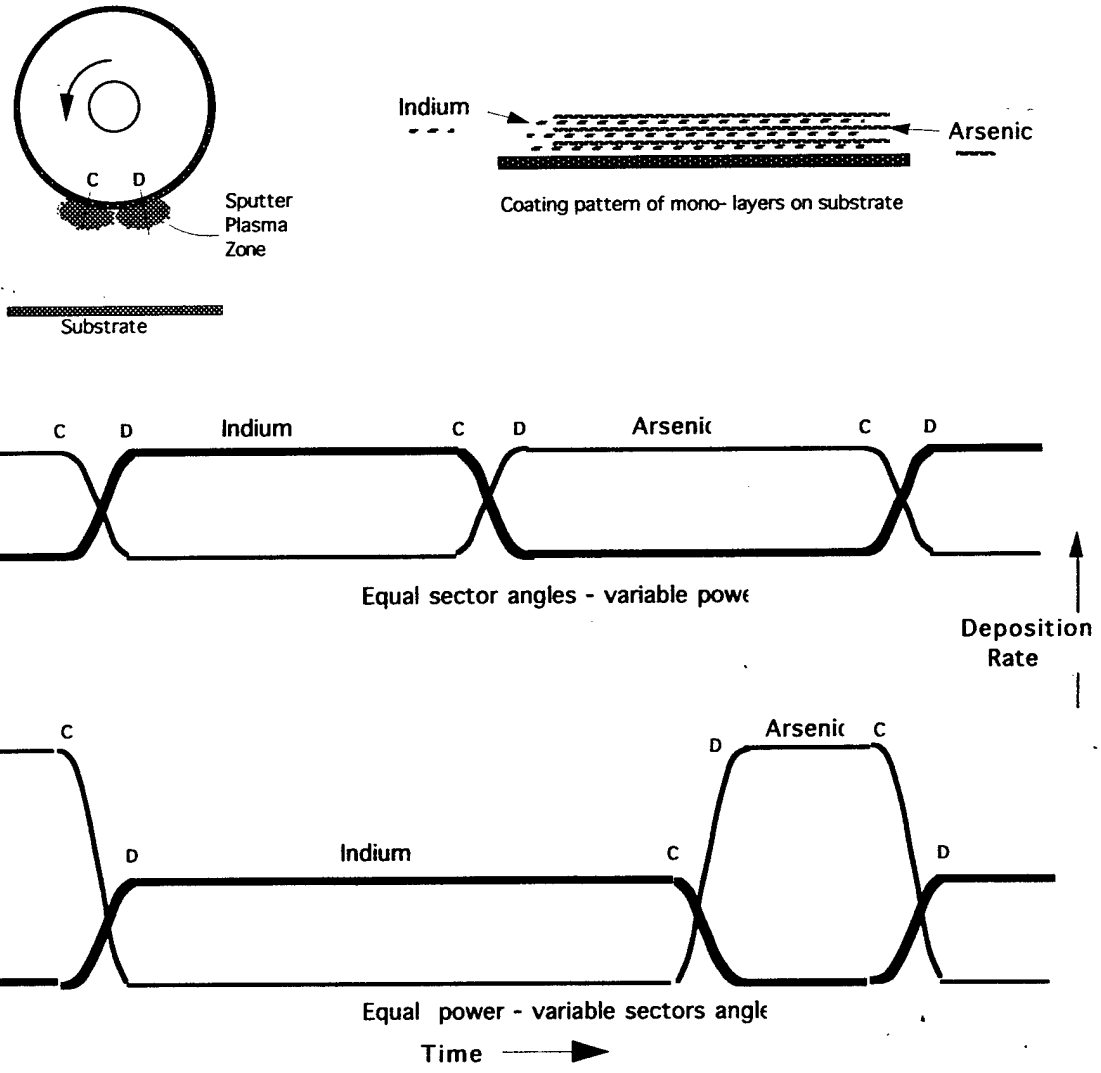
After the arsenic sector was applied there were 2 sectors on the target: 180 degrees of indium and 180 degrees of arsenic.

To deposit InAs, the cam was rotated 90 degrees. This was necessary since the arsenic vapor source was located at the " 3 O'clock" position while the sputter zone was at the bottom or the "6 O'clock" position. These cam positions were within 10 degrees of true position. It could and should be more accurately done but resources were limited.

During deposition the target rotated at 80 rpm. With present power levels 80 RPM is sufficient to get 2 monolayers per revolution; one of Arsenic and then one of Indium. When further research and higher power rated equipment allows increased microwave coupling levels, the higher deposition rate will require as much as 1500 RPM. My present rotating "O" ring shaft seal is limited to 500 RPM, but fortunately there are Ferrofluidic couplings rated to 7000 RPM

The target continuously rotated the indium and arsenic sectors sequentially through the sputter zone. The cam and switch were repositioned yet again and used to send different power levels to the indium and to the arsenic. The reason for different power levels requires explanation.
If the sector angular width were proportional to the sputter yield of each element then the indium sector would be 2.5 times larger. This is because the indium sputters 2.5 times less rapidly with equal power to both sectors. But while equal power would be easier to do, the boundary between the monolayers would be less abrupt. The arsenic sector would be narrowed to the point where the sector width and transition width were comparable.

To get the most abrupt boundary, the sectors must be of equal width and the power to each element adjusted as that element enters the sputter zone. The next figure shows a plot of transition width. Angle "a" is determined from magnetic field plots. It takes 45 degrees to make a complete transition through the sputter zone. Here the two rate vs. time diagrams show the alternate methods. It also shows that for maximum transition sharpness the equal sector angle method provided the narrowest transition width to deposition cycle ratio. This gave minimum "smearing" of the transition between layers. With equal power, the arsenic sector was not much wider than the transition width. The sector method is the only method being considered now.



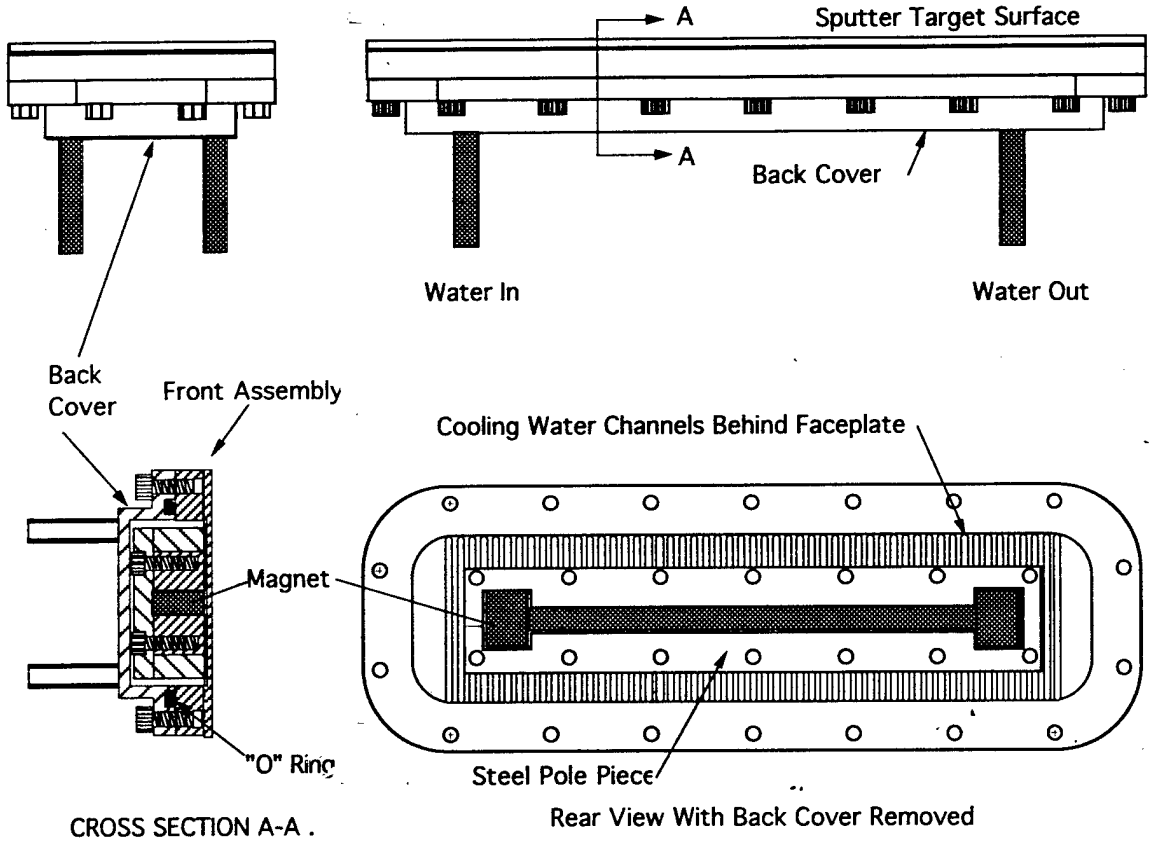
With the equal sector angle mode, deposition rate is controlled by varying power to the surrogate magnetron. As the Indium sector reaches the sputter zone power is raised to the maximum allowed. There is no limit for indium plasma voltage since indium is a metal and doesn't produce negative ions. Power is lowered when the arsenic sector edge reaches the sputter zone. The arsenic plasma voltage is the limit to power. Arsenic does produce negative ions and its plasma voltage must remain low. Fortunately, arsenic sputters 2.5 times more efficiently than indium. The Arsenic power is kept at the maximum while the Indium power is varied to precisely control the In/As ratio.

The outputs from two 500 watt sputter power supplies are sent to a relay switching circuit. The relay is operated by the cam and connects each power supply to the corresponding sector as required. The total switching cycle is about 0.8 seconds per revolution. A micro-switch driven by the cam can switch in less than 2 milliseconds. The circuit is a cross switch arrangement. When one

power supply is active the output from the other is sent to a dummy load resistor. This prevents power supply transients.

Auxiliary Sputter Target

The basic auxiliary target design is shown ion the next figure.



Assembly was simple. The magnets and pole pieces were attached to the front assembly. The back cover was then attached.
The magnet had a simple "dog bone" shape. This increased the deposition rate at the ends to match the sputter rate of the surrogate magnetron. The field at the target surface was about 600 gauss. The target was 4 mm thick for both Indium and Arsenic.

The original intent was to obtain a cylindrical surrogate entirely pre-coated with indium and then have arsenic stripes sprayed in a pre-calculated pattern. The stripe pattern would vary over the width of the surrogate to vary the stoichiometry (In/AS ratio) over the deposited film. One part of the film would supposedly then have good InAs. However, environmental regulations placed severe restrictions on the vendors and this striped target while quoted was never

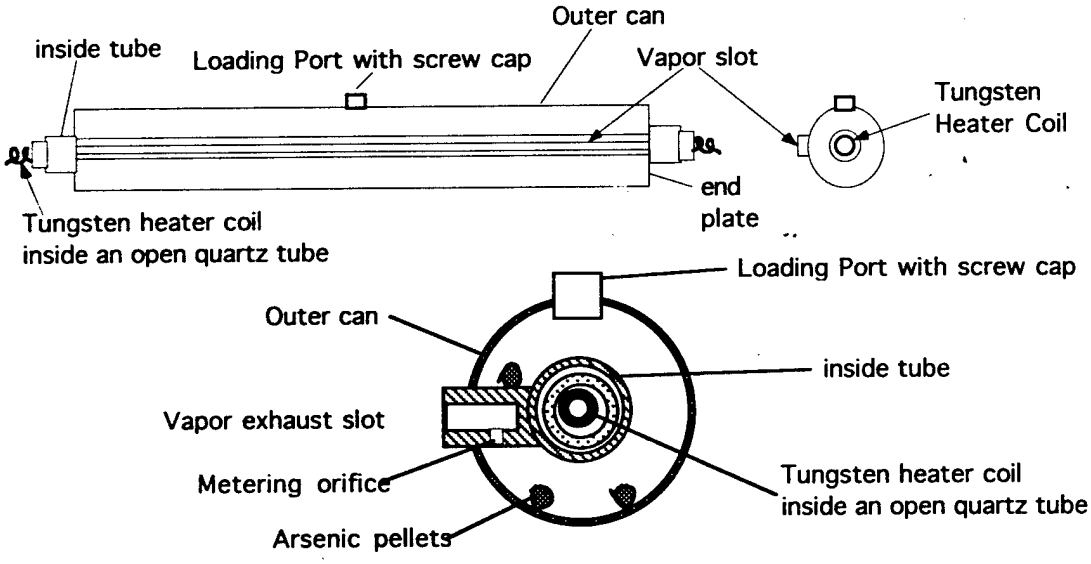
delivered. The vendor cancelled the purchase order. This striped target would have cut the time for the first deposition. It took over two years to get an auxiliary target. Again environmental regulations placed severe restrictions on the vendors. I inspected the auxiliary arsenic target when it finally arrived. The as-cast surface was non-uniform and visibly porous.

The arsenic target was used 20 times and then the surface became poorly conductive, probably because of oxidation. No further sputtering could be done with it because of severe arcing. This oxidation was probably caused by the poor vacuum of 1 $x10^{-5}$ torr. When the cryrogenic coil is installed the pressure should drop 100 times to 10^{-7} torr and the sputter life should extend at least that amount. 100 x 20 = 2000 sputter depositions. The sputter life may even be higher since there may be an oxidation threshold level.

The Indium target also oxidized with use but at a lesser degree as will be discussed later.

Arsenic Vapor Sources

Because of the unavailability of the auxiliary source or sprayed striped target, I had to design a vapor source, shown here.



Enlarged cross section of vapor source

The ends of the can are flat metal and completely seal the source. The only escape for vapor is through the metering orifices and thence through the slot. The vapors condense onto the target. A typical charge is 350 milligrams and it lasts for 3 or 4 depositions.

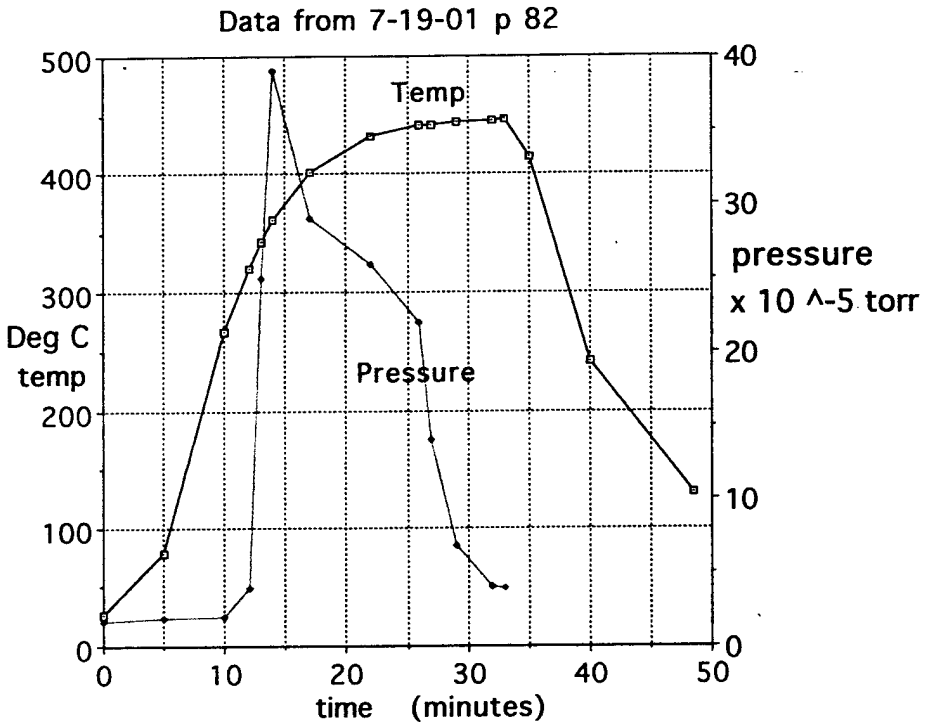
This vapor source has configurations applicable to both the stripe and sector methods. The stripes were made with small linear baffles placed inside the vapor source slot. For the sector method the slot was completely open.

I made two models of the vapor source. The original arsenic vapor source had cracked during the HHC effort. I designed a new stainless steel arsenic vapor source which was resistance heated at 75 amperes and 1 volt. Because it had a larger slit size the internal arsenic vapor pressure was lower. It did not work as well as the original IR heated source and more material escaped into the system. The new source had a much lower effusion rate than the old source. The problem may have been that I measured source temperature differently between the two sources. The old source may have a higher internal temperature but I won't know for certain without more tests.

I repaired the original arsenic vapor source and used it for all subsequent work. It took about 60 minutes to heat up, deposit arsenic and cool down enough so that sputtering could be done. About 2 to 3 % of the arsenic vapor escaped to the system. This was fortunate since I needed this pressure signal to monitor and learn the arsenic stripe and sector deposition processes. The micro ion pressure gauge was 5 to 10 times more sensitive to arsenic vapor that nitrogen or water vapor. There was a 1 micron trap on the exhaust of the system mechanical pump. The metal mesh became coated with pump oil and trapped the arsenic very efficiently although it slowed the initial pump down slightly. In the 3 years of work I have used, approximately 60 grams of elemental arsenic pellets. The arsenic effluent levels were much lower than for an equivalent CVD process.

Arsenic vapor source operation

The system was pumped down and the indium coated surrogate magnetron sputtered for a few minutes to present a clean surface for better film adhesion. The vapor source was then heated with 70 watts. The following shows the heating profile.



The system pressure was read with a Micro-Ion Bayard Alpert type ionization gauge. The temperature was measured with a thermocouple welded to the outside of the thin metal vapor source. I assumed that the pressure spike was from arsenic vapor. With no arsenic in the vapor source, the pressure never rose during the heating cycle.

When the pressure rise ended, the power was removed and dry nitrogen was bled into the chamber until the pressure reached 20 torr. This sped the cool-down of the vapor source. This was the procedure whether stripes or sectors were used.

Indium targets

Most work was done with a surrogate magnetron pre-coated with indium.

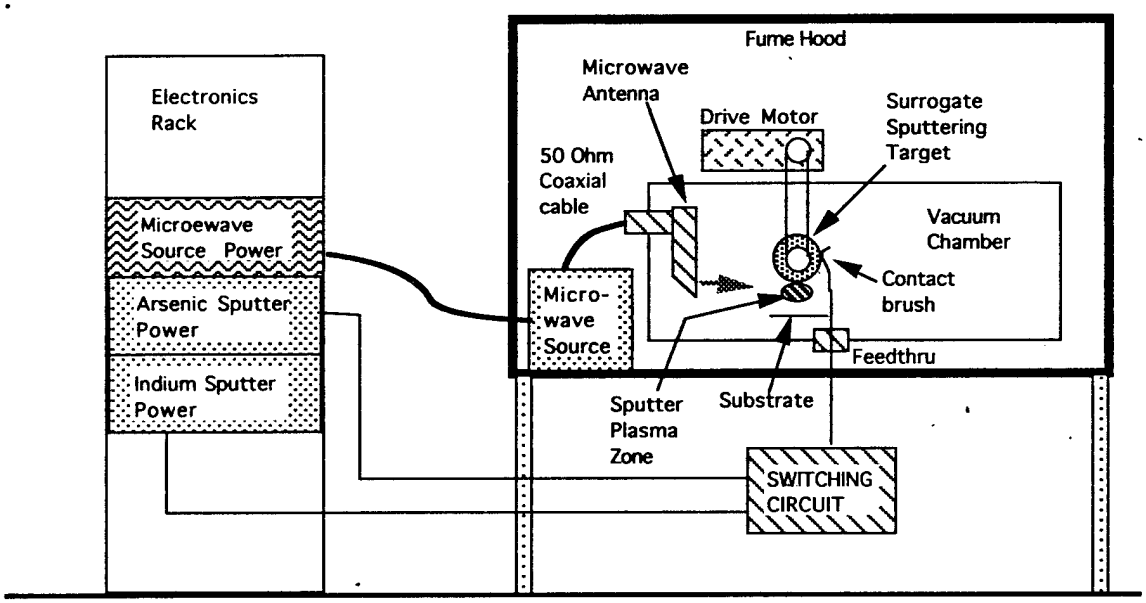
The indium auxiliary target was fabricated and used for several depositions but oxidation prevented its ultimate use. It was identical to the Arsenic auxiliary.

Deposition System Description

All experiments were done in a small stainless steel chamber. The entire system was housed in a 6 foot wide fume hood. An electronics rack stood adjacent. The

rack contained the turbo pump controller, pressure gauges, microwave source power supply, and the sputter power supplies.
The deposition chamber was a 10 inch diameter cylinder 27 inches long. It was turbo pumped and could reach 5x 10^{-6} torr.
There was an eleven inch diameter flange disk at each end for access to the interior. In the figure below, the left flange disk supported the microwave feed-through.
The feed through was made from the antenna end of an old microwave oven magnetron soldered into a brass QF flange

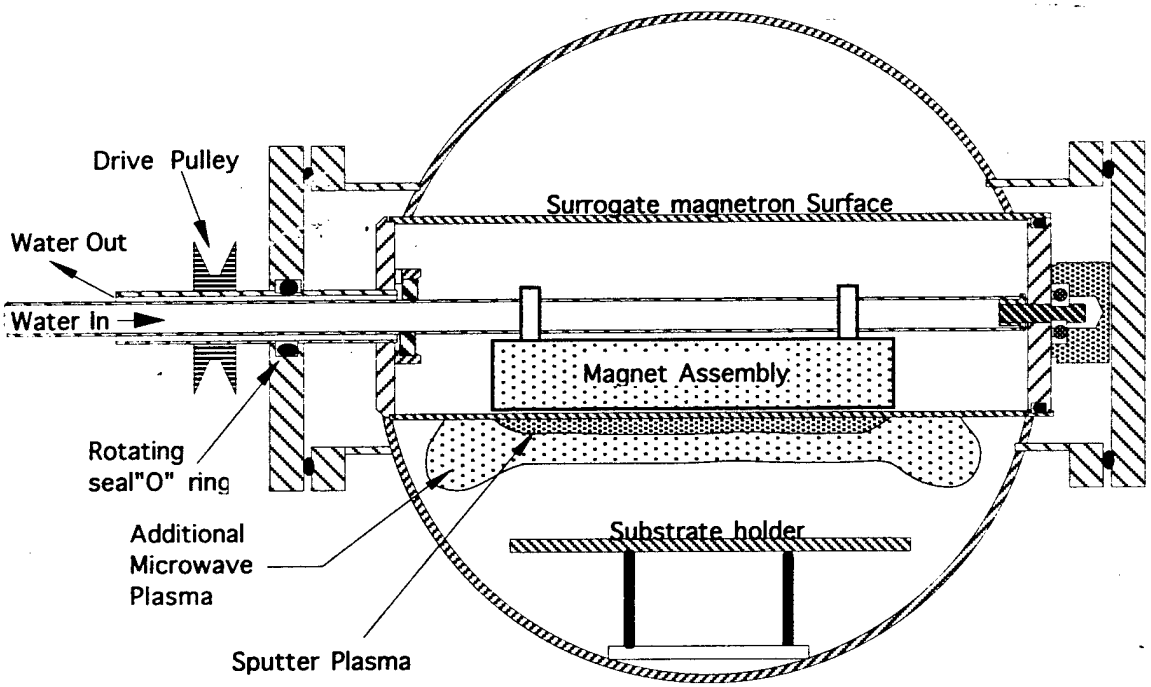
Not shown is an EPA HEPA vacuum cleaner rated for use with Arsenic dust. It gathered the dust that was generated during chamber cleaning. The chamber was cleaned once during the 3 year project.



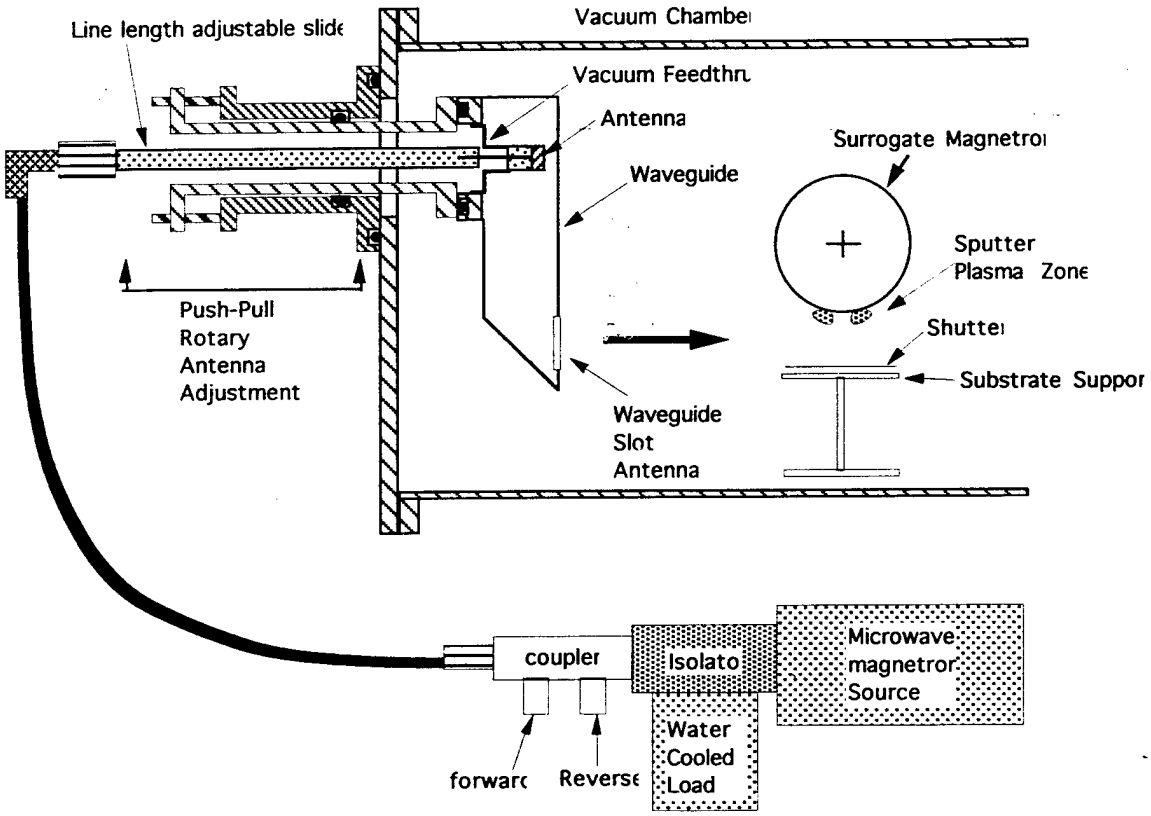
The vacuum pumps and instrumentation are not shown.

Surrogate cylindrical magnetron target.

The surrogate sputter target was a stainless steel tube, 75 mm (3 inches) in diameter and 250 mm (10 inches long. It has a magnet assembly inside supported by the cooling water tube. A cross-section of the chamber and surrogate target is shown next. The mechanical parts are slightly simplified for clarity.



Vacuum Chamber Cross section



Microwave System

Microwave power from a 120 to 1200 watt variable magnetron was fed to an isolator and then to a 600 watt co-axial cable. The isolator directed any reflected power into a water-cooled dummy load. This prevented reflected power from reentering the magnetron microwave source and damaging it. The cable carried the power to an adjustable slide co-axial line and then to the vacuum feed-through antenna into a waveguide. The microwaves were fed to the plasma with a slot antenna fed by the waveguide.

The bi-directional coupler read the forward and reverse power levels. This allowed tuning of the system. N type co-ax fitting rated at 300 to 500 watts are used throughout. Component power ratings were difficult to learn

The push pull-rotary antenna adjustment was used to tune the system. It allowed the slot antenna to moved closer or further away from the plasma and to be tilted 10 degrees in either direction With the stronger magnets with their larger magnetic volume there was a strong sensitivity of the system to tilt angle of the slot antenna. When I weakened the magnet to reduce the magnetic field volume that slot angle sensitivity greatly decreased.

A rotating vane was also used to tune the system. It was a half disc located at the opposite end of the chamber 38 cm (15 inches) away from the surrogate.

Two other methods of microwave feed were also used. First, a loop antenna was placed close to the surrogate at the 2 O'clock position. Second, the co-axial cable was fed directly into the end cap of the surrogate through a microwave slip ring. The loop antenna method was about equal in performance to the slot antenna and might be used in the future. Plasma power absorption with the direct feed was lower.

Chamber Absorption of Microwave power.

I fed microwave power to the system feed with no sputter power. This gave a base line absorption level for the system.

Watts in	Watts Absorbed
100	55 to 68
173	24 to 79

I could vary the absorbed or reflected power by moving the view port shutter. The absorbed power was the same at pressures of 2.5 and 5 millitorr.

I assume that one part of the chamber absorbed power more efficiently than the rest of the chamber since tuning could vary the absorbed power level. I don't yet know which part it is but it would be useful to know.

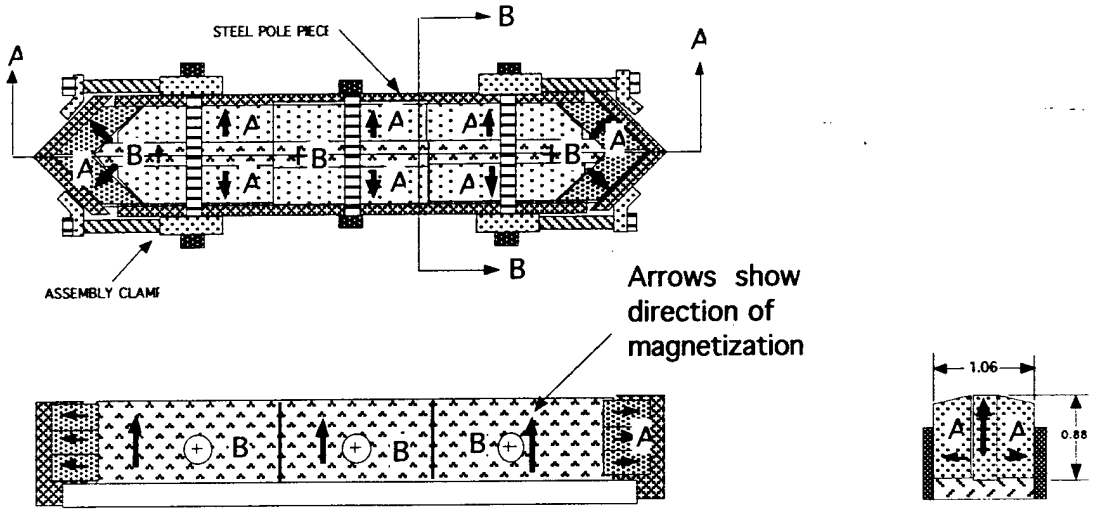
Chamber absorption might be a useful indication of system cleanliness.

Substrate absorption of microwave power

An InAs film will absorb microwave power and overheat if it is not resting on a metallic surface. The metallic surface protects the InAs film since the microwaves reflect at a node. The substrate should be within 1/20 wavelength of the metallic surface at the power levels I presently use. The higher the power the closer the substrate must be. This should not be a problem as the substrates always rest on or close to a metal surface.

Magnet Design

The next figure is of the original magnet, which produced the troublesome side or extra plasmas.



SECTION A-A SECTION B-B

Previous magnet design

Note the clamps and bolts that are necessary to assemble this magnet. It uses ND 39 material (Neodimium Iron Boride), which is very strong. The magnets tend to repel each other strongly unless clamped and bolted together. In fact, the magnets must be assembled loosely and then drawn together with the bolts.

Design of new magnet

This previous magnet had a large magnetic volume and a peak field of 3500 gauss. I realized it had to change if I was to remove the effects of the "side" plasma.

I then learned of the magnetic quadrupole principle from Dr Muraviev who works for me through a University of Central Florida contract. This principle states that to minimize the far field, the magnets must occur in matched pairs. If this is done then the far field is zero since a point in the far field sees two opposing identical magnets, which cancel each other. Only even numbers of magnets should be used.

As you can see, the above magnet design has 3 magnet directions. I have now removed the central B magnet and now have 2 identical A magnets facing in opposite directions. Dr. Muraviev calculated a 7 times decrease in the magnetic field in the region with the troublesome "side" plasma, or extra plasma, at the 4 O'clock position mentioned previously. The main field would also decrease, but only by 10%. In the actual assembly the old 3 piece magnet had a central B of 2600 Gauss and an end field of 2400. The new 2 piece magnet had a central

field of 2400 gauss and an initial end field of 1120 gauss. I was able to increase the end field to 1400 gauss but it had no immediate effect.

I reassembled the 2 piece magnet with closer spaced end pole pieces and raised the end field to 1400 gauss. I haven't done an analysis yet to find the problem. The new magnet is not as efficient as the old as far as power absorption. But if it eliminates the side plasmas then I could make better InAs at a lower rate to get more samples.

There is one other magnet design, invented by Dr. Muraviev, which uses a checkerboard of cubical magnets. This has an even lower far field and yet may be as strong as the old magnet array. I had no time to finish its design before the contract effort ended.

Results

Thickness Measurement

Film thickness was measured by weight. Most films were about 1/2 micron thick on a 2 inch x 2 inch x 0.010 inch thick 96% alumina substrate. 1/2 micron of InAs on a 25.8 cm^2 substrate has a volume of 1.25×10^{-3} cm^3 Its density is 5.67 gm cm-3, giving a total weight of 7.1 mg. Since the scale I used was sensitive to 0.05 mg, film thickness could be determined to 0.7 %.
Stainless steel thin films were used for profile and footprint determinations. This was easy to do since the surrogate cylinder itself is stainless steel. These film thicknesses were about 0.2 micron and density was 9 gm /cc. The total film weight was 4.5 mg and accuracy was 1.1%. For smaller substrates accuracy was proportionally worse.

I also had a need to measure the thickness of the films deposited by the auxiliary targets onto the surrogate target cylinder. For this I made 1 inch wide metal bands from 0.006 " thick stainless steel sheet spot welded into shape. They were a close sliding fit over the surrogate. I placed 7 of them in a row numbering them and pre-weighing them. Again the film thickness was measured by weight gain.

As I continued to use the indium target, the profile of the indium film deposited onto the surrogate became very non-uniform. It was uniform to 5% at the start and rapidly worsened to 2 to 1 variation. The cause was oxidation of the indium. The surface became rough and blackened. I could repair most, but not all, of the damage by burnishing the indium surface with a clean piece of cloth and isopropyl alcohol.

While sputtering at constant power I noticed a slow increase in the plasma discharge voltage. Voltage increased from 308 to 342 volts in 5 minutes of sputtering at 100 watts for a voltage rate of increase of 6.8 volts per minute. No microwave energy was used for the auxiliary targets.

When ice water was pumped through the indium target, its temperature dropped to 5 degrees C. during sputtering. Then the voltage rate of increase dropped almost in half, to 3.8 volts per minute. When I decreased the power to 50 watts, still with ice water flowing, the rate dropped even further, to 0.4 volts per minute.

From this I found that burnishing the surrogate target also lowered the plasma voltage. This explained some of the variability in the process. The surrogate target was not blackened but it did become rougher with usage. The surrogate surface oxidation was not as evident.

IV Curves

The current-voltage plot is the basic electrical measurement for the sputter plasma. It measures the coupling efficiency of the external energy sources, either e-gun or microwave as well as the performance of the magnet. It can also indicate the oxidation status of the arsenic and indium targets.

The first work was done with an electron gun, a model HCPEE250 hot hollow cathode (HHC) made by Electric Propulsion Labs rated at 10 amps with 2 sccm of argon flow.

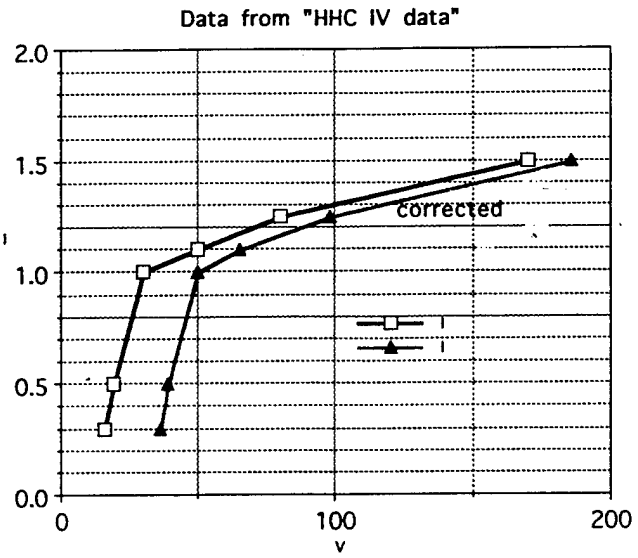
In order to inject electrons into the strong magnetic field I used a "pipe" of either metal or ceramic to channel the electrons from the HHC into the plasma. The pipe also prevented the HHC from being coated by the sputtered material

The situation was never completely satisfactory. The metal pipe would be attracted to the strong magnet and move out of position necessitating a very strong yet adjustable mount. The pipe would get red hot.

The HHC had 2 major problems. The first was the necessity for an anode biased at about 18 volts positive. Recall that the plasma is always a few volts (say 10 V) more positive than the most positive point in the system. The anode was in contact with the plasma. So even if the target voltage was - 65 V below ground, the accelerating sheath voltage was 65 + 18 + 10, which is 93 volts. Thus, the effective plasma voltage was always increased by the anode voltage. The anode was necessary with the HHC but was not necessary with microwaves.

This IV curve shows an HHC IV curve with and without correction for the anode

voltage.



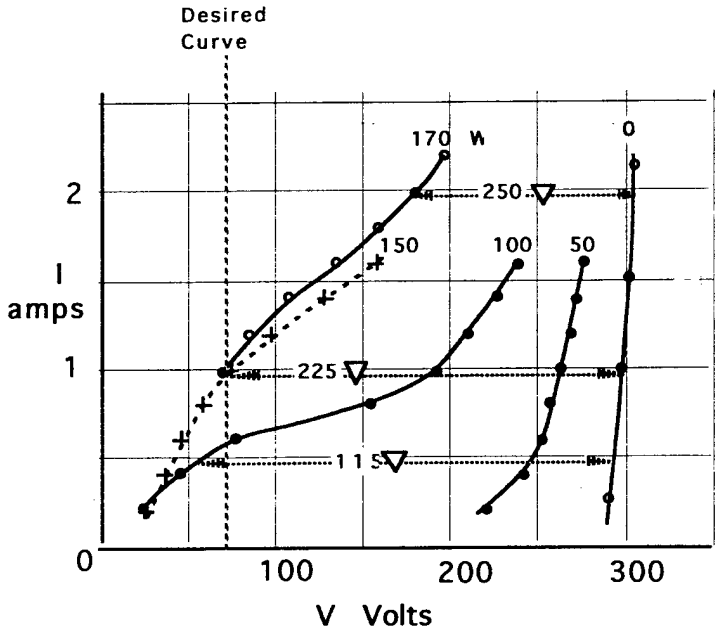
The second problem was that the HHC was biased 40 volts negative to ground. This weakened the electrostatic field near the target, which was also biased negative to ground.

Because of these problems all work with the HHC was stopped and microwaves were substituted as the method to add energy to the plasma.

Microwave Results

Initially I used a resonant cavity to confine the microwaves to the plasma region. This was not effective. The cavity disturbed the electrostatic field and degraded the IV curve. I removed it and the system ran without it.

An approximate theory of low voltage sputtering was developed. It simply subtracts the low voltage enhanced IV curve from the standard IV curve with no added energy. The next figure shows this. The numbers next to the curves are the injected microwave power levels. This data was taken with a stainless steel surrogate target. The delta lines show the actual power difference between the microwave supported curve and the 0 watts curve.



This concept seems more accurate at low sputter power. In fact the effect of microwave energy seems to become more efficient at higher sputter powers. At a nominal 170 watts of microwave power the curve is lowered by 250 watts. I could make each curve a vertical line by varying the microwave power at each point on the curve. But it would pass through the topmost point. And the lower part of the curve would be further away from the desired curve. My instrument calculations are only approximate for the microwave power data. So

Presently the microwave system power is limited by the line length adjustable slide. It is rated at 100 watts continuous and 500 watts intermittent. Even though the rest of the system is rated at 300 to 500 watts I cannot go over 200 watts CW. I have already burned out one slide.

The main sputter power supply is rated at 5 amps @ 2.5 KW. Assuming the un-energized curve voltage at 5 amps to be 325 Volts and 70 volts is desired, I need to inject 1275 watts of microwave power. [5 amps x (325-70) volts]. The microwave source is rated at 1200 watts but I can't use all of its power. The deposition rate would be 5 times greater if I could.

Deposition rates

The present deposition rate was 11 microns per hour with the stripe method so 55 microns per hour might be possible with this particular system. The stripe method did not seem as sensitive to side plasmas.

The sector method has a present deposition rate of 4 microns per hour. It has not yet been optimized because of its evident sensitivity to the side or unwanted plasma.

Film Deposition Results

Once a proper substrate cleaning procedure was developed, all subsequent films adhered well. The main contaminant was styrene out-gassed from the styrofoam storage boxes the substrates were shipped in. All films passed the tape pull test and could be heated to 550 degrees C in dry nitrogen with no adverse effects such as flaking or pinholes.

If films with excess indium were made, they appeared slightly lighter than stoichiometric films. However, when these films were heated to 550 degrees C in dry nitrogen the excess indium would form small globules of pure indium evenly spaced all over the surface. The film weight loss was less than the measurement limit of 0.05 mg.

If films with excess arsenic were made, the excess arsenic was emitted into the tube furnace exhaust vent. The resulting films in all case were well adherent but had low mobilities of less than 200. Weight loss for these films was from1 to 5%.

All films were deposited at room temperature. They were slightly warm to the touch after being removed from the deposition system.

The films were protected by a shutter during system adjustment prior to deposition. When the system was properly tuned, swinging back the shutter had little effect on plasma voltage. The shutter had to be electrically floating though. If it was grounded, when it opened, a large plasma voltage change occurred and the system had to be readjusted to compensate.

Film mobility results

I deposited one film dated 4/13/00, which was made into sensors. It had 8 sensors out of 200 with mobilities over 250 cm^2/volt second. The best had a mobility of 2045. (4.5 mv @22 ohms). These numbers came from Bell's process engineer. I did not know at the time that this film was contaminated with oil. The copper electrode adhesion was poor. Over the next several months the sensor properties degraded, presumably because the copper was coming off. A year later I attempted to have wire leads soldered to the sensors but they all pulled off during soldering.

The best films needed no heating to maximize sensitivity. They could be heated with little change seen after heating.

Discussion of results

Microwave Power absorption

The deposition chamber absorbed from 14% to 45 % of the incident power. This was with the surrogate magnetron installed but no substrate holder or shutter. Presumably, when tuning, I changed the ratio of power absorbed in the plasma to power absorbed by the chamber. Since chamber power absorption could be almost half of the incident power and I was limited in power to less than 200 watts, this was a serious issue. Waveguides would transmit much more power than co-axial cable. But I needed the cable for its ease of use. For the next system waveguides could be built into the chamber itself to increase microwave power levels and deposition rate.

Stoichiometry control

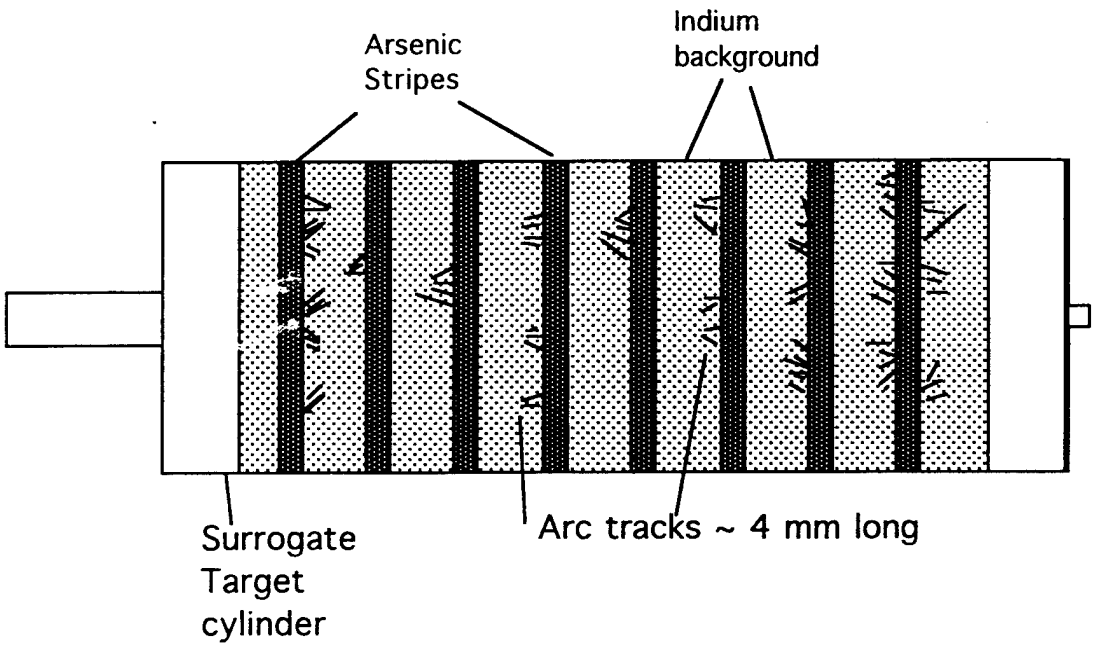
Deposition footprint uniformity is essential for good film composition control. The unwanted side plasma prevented this. The film thickness varied as much as 3 to 1 from front to back of the substrate. I now have a magnet design that has low magnetic field volume. I also know what the proper mechanical design requires for ease of magnet assembly and adjustment.

The sector method is capable of closer stoichiometry control but it requires more accurate cam alignment make it easier to align the surrogate target sectors with the plasma and the arsenic vapor source.
The sector method would also eliminate the effect of non-uniform magnetic fields.

The sector method provides the means for migration enhanced epitaxy (MEE) [3]. This is probably the most important development of the entire effort,

The stripe method of combining element should not be discarded but it is more difficult to implement in the present state of the art.

The jelly roll method has a defect, which I don't know how to solve. When I sputtered stripes at high plasma voltages the indium stripes showed severe arc tracks at the boundary with the arsenic stripes as shown here.



I assume these arcs might occur even at lower voltages but to a lesser degree. One possible reason is that plasma impedance depends, to some extent, on the material being sputtered. The secondary electron yields are different for arsenic and for indium. It's possible that current travels from arsenic stripe to indium stripe within the plasma. I don't know if this is harmful but to the extent it is variable it might affect stoichiometry control. The arc tracks I saw were certainly not uniform.

Effects of Non-Uniform Magnetic Fields

Non-uniformities in the surrogate magnet show up as either grooves or ridges. Higher magnetic field areas sputter more efficiently and remove material faster. Low magnetic field areas sputter less rapidly and build up ridges. The next figure shows how to estimate the magnitude of this problem.

Assume that by continuous coating of the auxiliary target and the vapor source a total thickness of 1/4 mm (0.010 ") was accumulated. It is shown here is a single coating for clarity. Whether it exists as a single coating or is slowly built up has no effect on this analysis. Assume the stronger and weaker magnets cause a plus and minus 25 % deviation in sputter rate from the average sputter rate in the target center.
Eventually the ridges are so thick that the target must be removed from the system and resurfaced with a lathe.

Two criteria determine when to resurface the target:

a. The ends of the material are never sputtered in order to protect the target itself from being sputtered.
b. . If the area of maximum sputter rate sputters too rapidly, the target base tube will then start to sputter and contaminate the film with stainless steel.

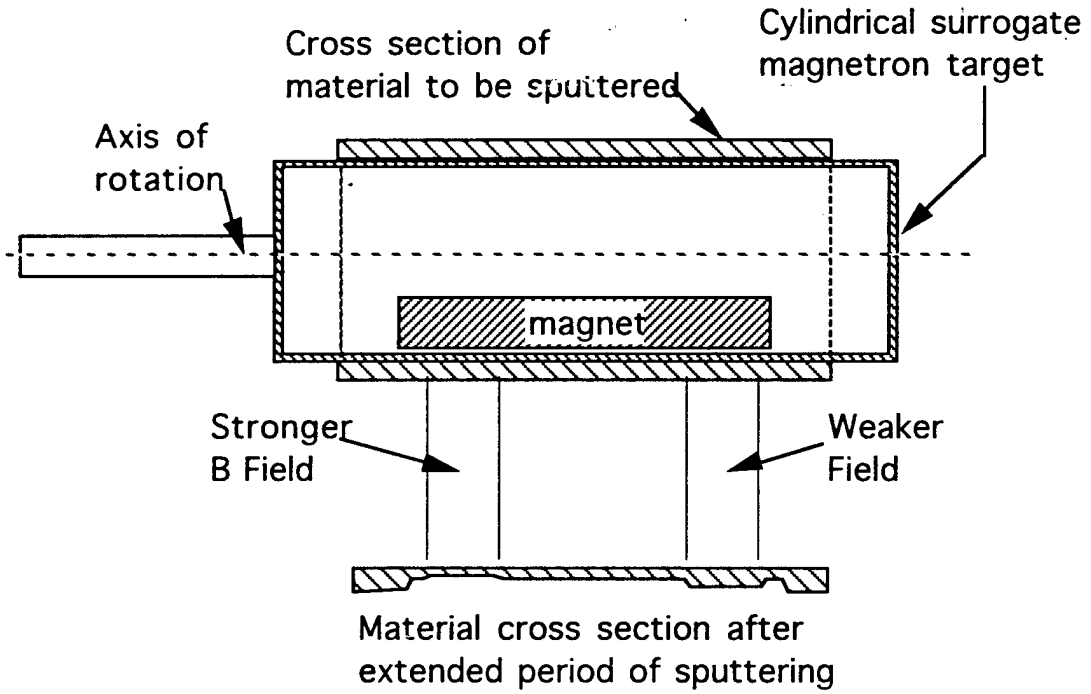
With the sector method, stoichiometry is independent of magnetic field uniformity. The only effect of such non-uniformity will be to make the film thicker under the strong field area and thinner under the weak field area. For this calculation:

- Assume a material thickness of 1/4 mm on a 7.5 cm. diameter tube, 15 cm active length
- Assume also that the target is running at 500 watts.

Material volume is then 88 cm^3. Because of the strong field area we use only 75% of this volume or 66 cm^3. InAs sputters at an efficiency of 5 x 10^{-7} cm^3 per Joule. To calculate the time needed to sputter this volume we write:

(66 cm3 x 1 joule x 1second/ 500 Joules) / (5 x 10^{-7} cm^3) = 2.65 x10^5 sec.
Thus the target needed resurfacing every 74 hours.

For a 1/2 micron thick film this represents an area of 132 square meters. This is enough for 20 years of production at Bell Technology's present level of production.



Effects of 10^{-5} torr vacuum

As soon as stoichiometry control is perfected then the effects of poor vacuum may become evident. Oxygen is a contaminant for III-V materials. Oxygen certainly harmed both the indium and arsenic auxiliary targets. Placing a cryrogenic LN2 coil in the system will certainly improve the vacuum from 10-5 torr to 10^{-7}torr. I just recently devised a way to install one in this chamber without interfering with sample insertion and shutter actuation.

This problem is a lower priority than getting good stoichiometry control and film uniformity.

Process Scale up Considerations.

The present deposition rate of 11 microns per hour is much higher than the competing processes of ALE (atomic layer epitaxy) or MBE (molecular beam epitaxy). ALE has a rate on the order of 0.1 microns per hour and MBE is not much faster. So this method is at least 2 orders of magnitude faster. And yet it has great promise of film quality sufficient for many semiconductor applications. It is also superior to MBE and ALE in that the arriving ad-atoms have 1 to 10 e.v. of energy rather than their 0.2 e.v of energy

The main concerns for scale-up and their proposed remedies are:

1. The changing arsenic and indium surface condition during sputtering
    a. Lower oxygen levels at 100 x lower pressures
    b. Periodic recoating of the surrogate with a thin molten indium coating. This would provide the sharpest sector transition edge. This is patented already.
    c. Use of the arsenic vapor source.
    d. Sputter plasma photoemission to compensate for changes in target surface state.
    e. Low temperature coolant will greatly improve stability.

2. Minimizing the sector transition angle for maximum abruptness of the mono-layers.
    a. Periodic recoating of the surrogate with a thin molten indium coating. This would provide the sharpest sector transition edge.
    b. Use of the arsenic vapor source

3. Scale up of the microwave delivery system.

a. Multiple antennas could be used if they were properly phased. Since the wavelength is 12 cm, phasing to 1/100 wave involves easy dimensional tolerances of 1 mm.

4. Scale up of the arsenic vapor source.
   a. The present design can be increased to a length of 1 meter with little change in design.

5. Difficulty in obtaining arsenic targets
   a. Use the arsenic vapor source

6. Leakage of arsenic vapor into the system.
   a. The leakage rate is a few percent
   b. The vapor source can be enclosed in a separate compartment.
   c. Note that the arsenic target would over-spray about 30% into the system. The vapor source is a more efficient source and it provides the sharpest sector transitions.

7. Increasing microwave absorption
   a. Use waveguides built into the chamber
   b. Monitor chamber base absorption. This should also be a measure of chamber cleanliness and indicate when cleaning is necessary.

8. Environmental concerns with arsenic
   a. Solid arsenic is much less dangerous than compounds such as arsine and arsenic trichloride.
   b. The main concern is dust when cleaning the chamber and chamber tooling. I have used a motor driven rotary wire brush encased in a small container and exhausted by an EPA HEPA filtered vacuum cleaner. This trapped almost all of the material. If the work is done in a fume hood with HEPA filters to catch any escaping particles there should be no concern. Larger deposition chambers will probably be cylindrical in shape and these are the easiest to clean. They can also be vented themselves by placing an exhaust duct at one end while the operator works at the other end. In this case no fume hood is necessary.
   c. Sandblasting could be effective but only if it can be radically improved over present devices.
   d. I note that in my arsenic hygiene research I find that arsenic is being considered as an essential trace element in the human diet. Recently it has been found to cure one rare form of cancer.

The discovery of the sector method has changed the system design rules. However this has made the task easier than first anticipated.

Bibliography

1 Muller " Role of Incident Kinetic Energy of Adatoms in Thin Film Growth. Surface Science 184:L375 (1987) Note... Chapter 21 of "Handbook of Plasma Processing Technology" Ed. By Rossnagel, Cuomo, and Westwood contains an excellent discussion of this subject of self bombardment, especially PP506-509. It also has a good bibliography.

2 " Thin Film Processes II" Ed by Vossen & Kern Page 57

3 Y. Homma et. Al. Appl. Physics Letters, 68(1), 63 [1 Jan 1996]