

Chapter 3

Hybrid Integration using the Epitaxial Lift Off Method

3.1 Introduction

The epitaxial lift off (ELO) technique finds its roots in the earlier works of Konagai, Sugimoto, and Takahashi where they worked on a "peeled film" technology for solar cells in 1978 [KoS78] and Wu, Coldren, and Merz where they examined the etch rates of $\text{Al}_x\text{Ga}_{1-x}\text{As}$ films as the Al mole fraction was varied from $x=0$ to $x=0.4$ [WuC85]. It was only until 1987 that Yablonovitch and co-workers popularized the ELO method and made it a viable alternative to some of the other monolithic/hybrid thin film integration methods [YaG87]. Although the other methods, described in section 3.1.1, are capable of integrating extremely thin layers of GaAs with Si, the ELO technique has proven to be one of the most versatile in allowing GaAs integration with almost any substrate, including nonplanar and transparent substrates.

3.2 General Description of the ELO Method

The epitaxial lift off method, as its name implies, is the complete separation of large area epitaxially grown AlGaAs films from their growth substrates and subsequently bonding these films to various other substrates [YaG87]. This technique utilizes a thin AlAs layer between the epi-layers of interest and the GaAs substrate. The high etch selectivity of AlAs over GaAs ($\approx 10^7 - 10^8$) or low Al mole fraction AlGaAs in 10% hydrofluoric acid is used to completely undercut the films of interest, thus separating them from the GaAs substrate. Since AlAs is extremely reactive and has been observed to decompose upon exposure to air, it is expected that an acid such as 10% HF will attack AlAs quite vigorously. The approximate etch rate of AlAs in 10% HF is $50 \mu\text{m}/\text{min}$ to $60 \mu\text{m}/\text{min}$ [DeP90] whereas the upper limit for GaAs appears to be no greater than $1 \text{\AA}/\text{hour}$ [YaG87]. Through the use of Apiezon W "black" wax as an HF resistant carrier, the selective etching of these extremely thin films can be facilitated as well as allowing them to be easily manipulated and bonded to alternative substrates without any damage. Upon application of the Apiezon W wax to the sample and insertion into 10% HF, the tension in the Apiezon W wax lifts up the edges of the epi-layers, thus allowing the by-products of the etch to exit the

etch front and allow new etch to enter, as shown in Figure 3.1. The methods of applying the wax to the sample will be discussed in section 3.2. The main by-product of the etch is H_2 , but other gases may form including AsH_3 . In fact, the mixture of H_2O and AlAs can result in the formation of AsH_3 . (As a note, since this process utilizes HF and can cause the possible formation of AsH_3 , however small the amount may be, the etch should always be performed in a fume hood.) To avoid cracking of the film and excessive bubble formation, the 10% HF etch environment is kept at $0^\circ C$. The importance of tension in the black wax and its ability to induce a radius of curvature of several centimeters in the epitaxial film was first investigated and understood by Yablonovitch and co-workers. The radius of curvature translates to lifting the epitaxial film up at the edges, thus changing the etch dynamics. Without taking into account the curvature in the film, the etch flux is equated to the diffusion flux of the by-products of the etch as follows:

$$vt3N = -Dt \frac{dn}{dz} \quad (3.1)$$

where v is the etch rate, n is the molar volume of the dissolved by-products (primarily H_2), N is the molar concentration of the AlAs release layer, t is the thickness of the AlAs, and D is the diffusion constant of H_2 [YaG87]. The above equation assumes 3

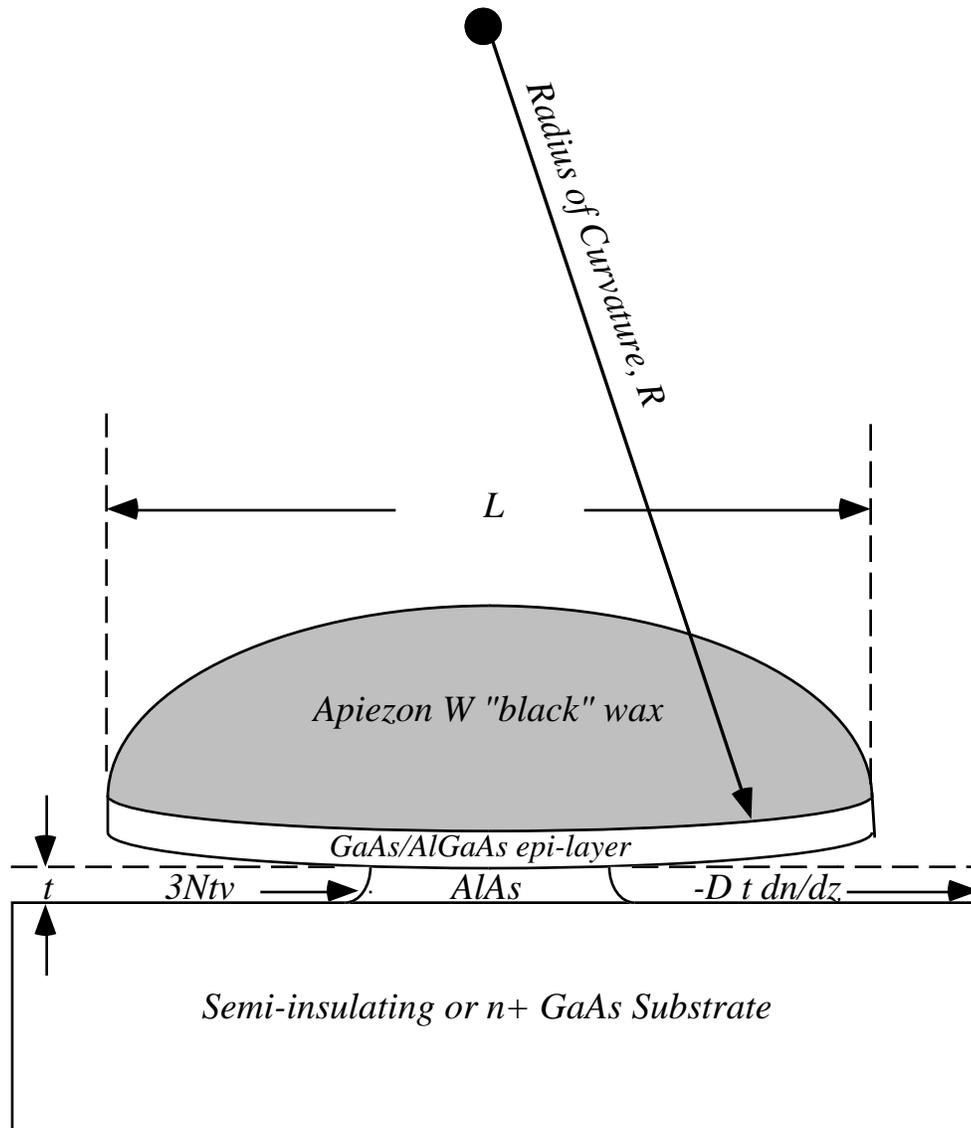


Figure 3.1 Illustration (not drawn to scale) of the ELO technique as an epitaxial film is being separated from its original growth substrate. The tension in the Apiezon W black wax induces a radius of curvature, R , of several centimeters in the epitaxial film which promotes removal of etch by-products from the etch front. As expressed by Yablonoitch, the etch flux is $vt3N$ and the diffusion flux is $-Dt \, dn/dz$ when not taking into account the curvature[YaG87]. L is the length of the AlAs channel that needs to be etched out and t is the thickness of the AlAs release layer.

moles of H₂ is produced for every mole of AlAs. If one integrates the above equation over the total length of the channel, L, and integrates over the total molar concentration of H₂ that can be produced, the maximum speed that the AlAs is removed can be expressed as [YaG87]:

$$v = \frac{Dn}{3LN} \quad (3.2)$$

Note that the total length along the channel, L, that has to be etched is a function of the dimensions of the Apiezon W black wax. Now, if the radius of curvature is included in the above analysis, equating the etch and diffusion fluxes is given as follows[YaG87]:

$$vt3N = -D \left(t + \frac{z^2}{2R} \right) \frac{dn}{dz} \quad (3.3)$$

Integrating this equation in the same fashion as above, the maximum speed that the AlAs can be removed is given as:

$$v = \frac{1}{3\pi\sqrt{Rt/2}} \frac{Dn}{N} \quad (3.4)$$

Therefore the total effective channel length that needs to be etched out is now expressed by $\pi\sqrt{\frac{Rt}{2}}$ which results in a much smaller value than the actual physical channel length defined by the wax.

Upon complete lift off, the sample is transferred to a surrogate substrate where it is bonded by various methods such as van der Waals (VDW) bonds, adhesive-based bonds, and metal/alloy-based bonds. Once the substrate is bonded to the alternative substrate, the Apiezon W wax is removed with a solvent such as trichloroethylene (TCE). This author has found that trichloroethane (TCA) works just as well and is not as toxic as TCE. In addition, toluene can also be used to

remove the wax, but since TCA is in wider abundance, it was used in this research. The following sections in this chapter describe in more depth the procedures used for the epitaxial lift off of a wide variety of films.

3.3 Sample Preparation for Epitaxial Lift Off

The epitaxial growth of the device structure is obviously the first step in preparing the sample for ELO. The AlAs release layer, as indicated earlier, does not have to be very thick and is usually kept within 100\AA to 500\AA . Whereas MBE growth of the active device layers in most of this research occurs at $\approx 600^\circ\text{C}$, the growth of the AlAs release layer was usually higher at 630°C in an effort to keep the surface as smooth as possible before growth of the actual device layers. In addition, for two terminal devices, where the heavily doped n-type or p-type GaAs substrate acts as one terminal, the AlAs can also be very heavily doped n-type or p-type with very little impact on the electrical properties of the device, as shown in Figure 3.2. The typical Si doping concentration in the AlAs release layer is $\approx 6 \times 10^{18} \text{ cm}^{-3}$.

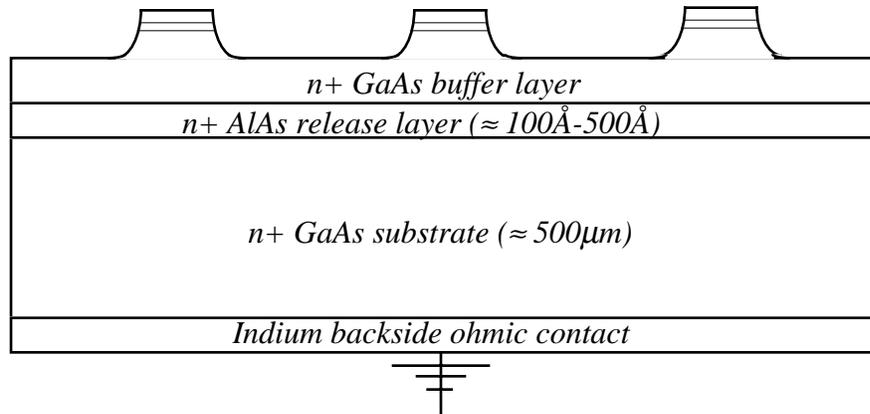


Figure 3.2: Illustration showing typical cross-section of ELO layer structure for two-terminal mesa isolated device that can be tested both before ELO and after ELO. Note that mesa etch must be controlled such that the etch does not penetrate the AlAs release layer.

Therefore, after device fabrication, one can test the devices both before ELO and after ELO. This allows a direct observation of the impact of ELO on the characteristics of any such two-terminal device. If an ELO device is grown on a semi-insulating GaAs substrate, then the AIAs can be grown nominally undoped.

There are many ways in which to apply the Apiezon W wax to the sample before lift off. In addition, the wax can be applied both before or after device fabrication depending on the particular process flow that is being used. The majority of this work is based on devices that have been fabricated first and then lifted off from their substrates. There are three types of Apiezon W wax, with different properties as shown in Table 3.1 [Bid81] :

	Wax W40	Wax W100	Wax W
Approximate Softening point, °C	45	55	85
Temperature for application, °C	45-50	80	100
Coefficient of expansion per °C, over 20°C-30°C	0.00063	?	0.00062
Permittivity	2.9	2.7	2.8
Volume resistivity, ohm/cm ²	5.055 x 10 ¹⁵	1.64 x 10 ¹⁵	6.31 x 10 ¹⁵

Table 3.1: Physical Properties of Apiezon W Waxes (from [Bid81]).

These waxes are of high purity and have a strong gettering action on greasy or chemical impurities. This gettering action is based on the very high average molecular weights of these hydrocarbons resulting in an ability for strong adsorption [Bid81]. Furthermore, these waxes are chemically resistant to acids such as HF, HCl and HNO₃. The simplest way to apply the wax to an ELO sample is by melting it directly onto the sample. With already processed devices, it was found that melting the wax directly onto the sample did not hurt the metallization or the device structure at all. With the use of a teflon coated tool, the wax and its shape can be manipulated and pressed onto the ELO sample. In addition, if the Apiezon W wax is dissolved in TCA and allowed to dry, it forms a very soft paste that can be applied to ELO

samples without having to heat the sample. Furthermore, if the wax is dissolved in TCA and not allowed to dry, the liquid mixture of TCA and wax can be spun onto the ELO sample in a similar fashion as spinning photoresist on a wafer. The advantage of using a solvent to soften the wax is that shaping the wax becomes easier. Rectangular shaped wax carriers are reported to work the best due to their radius of curvature [YaH90], but square or oval shapes with lengths of up to 2 cm also have been found to work. The disadvantage of using solvents to soften the wax is that the wax needs to be annealed to drive off all the solvent so that the wax becomes rigid, hard and dome-shaped. The dome-shape is believed to assist in lifting the film up at the edges and provides a smooth round surface which allow a vacuum tool to easily pick up or manipulate the sample. After application of the black wax to the ELO sample, exposure of the AIAs at all cleaved edges must be verified. Typically, the sample edges are cleaved up to the edge of the black wax or a teflon coated tool can be used to press the wax to the edges of the sample.

For mesa isolated device structures which contain heterojunctions consisting of $\text{Al}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$ (with $x \geq 0.5$), care must be taken when performing ELO on these samples since the 10% HF can also attack the device itself. Here the black wax must be applied such that there is uniform step coverage over the mesas with no gaps in between the wax and the devices. Upon application of the black wax to these mesa isolated devices, see Figure 3.3, the wax is re-annealed or re-melted to obtain the dome-shape in the wax carrier, as shown in Figure 3.4. The height of this dome on the chip is usually ≤ 0.25 cm. In addition, the mesa isolation etch must not go down through the AIAs release layer or the ELO method will not work since the AIAs release layer itself is blocked from the 10% HF etchant. Several samples were lost in this work due to unexpected high etch rates which went through the AIAs release layers.

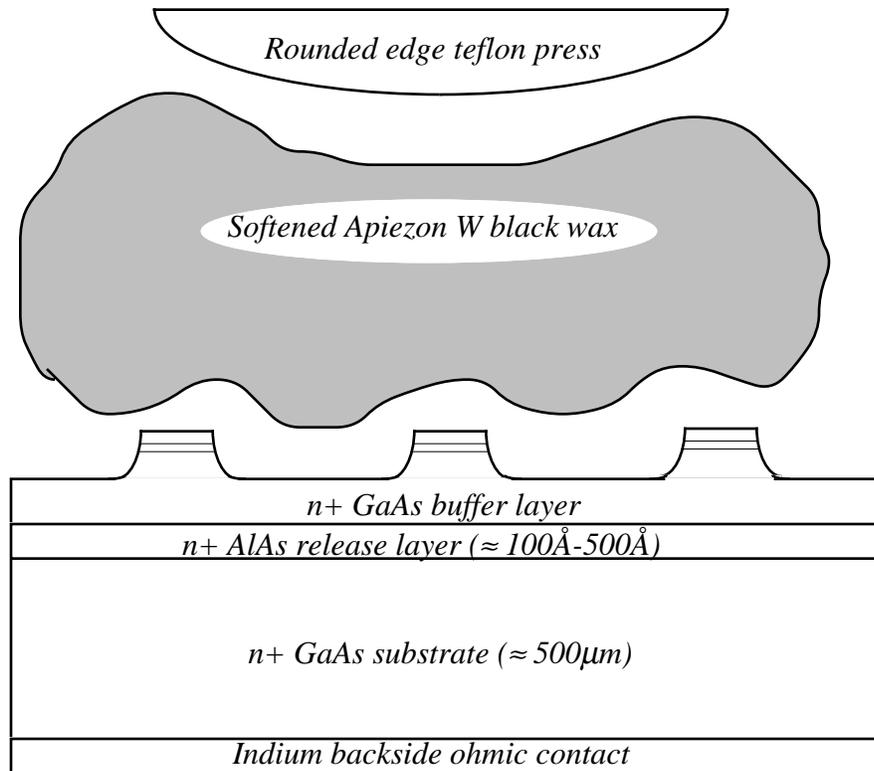


Figure 3.3: Illustration of the application of Apiezon W black wax to a mesa isolated AlGaAs/GaAs heterojunction device. To make sure that good step coverage of the black wax over the mesas occurs, a teflon press is used to press the softened wax over the edges of the mesas. The wax can be softened either by raised temperature or through the use of solvents such as TCA and toluene.

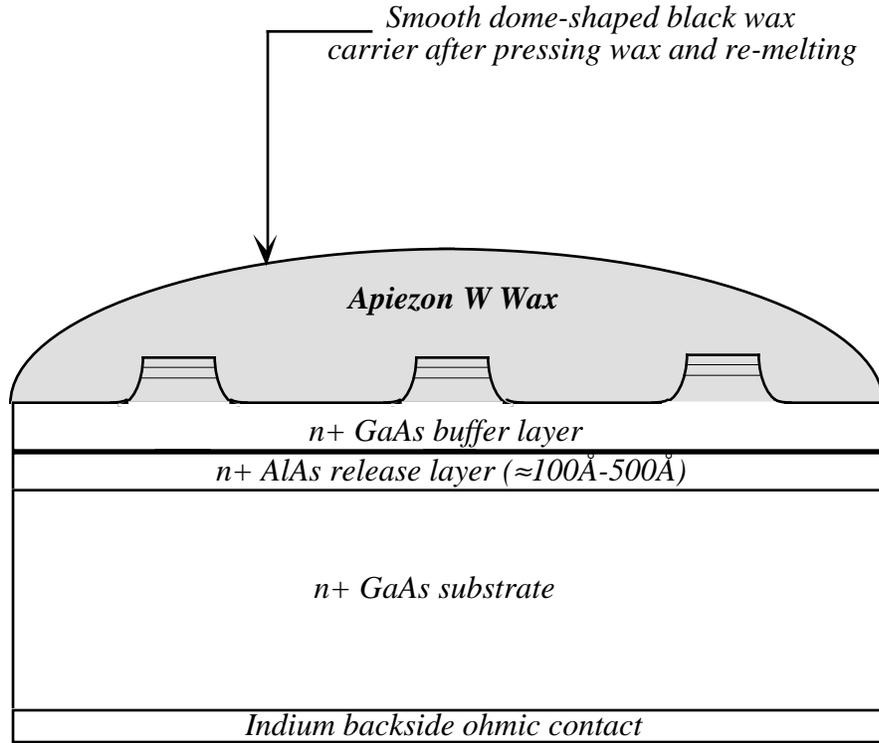


Figure 3.4: Illustration of dome-shape in black wax carrier on mesa isolated AlGaAs/GaAs heterostructures. The AlAs release layer is exposed at all edges before inserting the sample into the 10% HF etch.

3.4 Epitaxial Lift Off and Manipulation of ELO Films

Once the black wax carrier has been properly formed on the sample, the sample is placed in a 10% HF etch bath that is usually kept at 0°C. The low temperature is used to prevent excessive H₂ bubble formation which can hinder the complete undercut of the AlAs release layer. The time required to achieve complete lift off depends on the surface area of the ELO film and cannot be determined from the extrapolated etch rates of AlAs which represent an upper limit. ELO films with a surface area of 1cm² usually take about 5 to 6 hours for complete separation from the substrate. Films as large as 4cm² have been separated from the substrate in one overnight period (≈ 12 hours) while other samples have taken up to 48 hours to separate from the substrate. As the 10% HF etches the AlAs exposed at the edges of

the cleaved substrate, the tension in the black wax causes the film to peel upward at the edges, as shown in Figure 3.5.

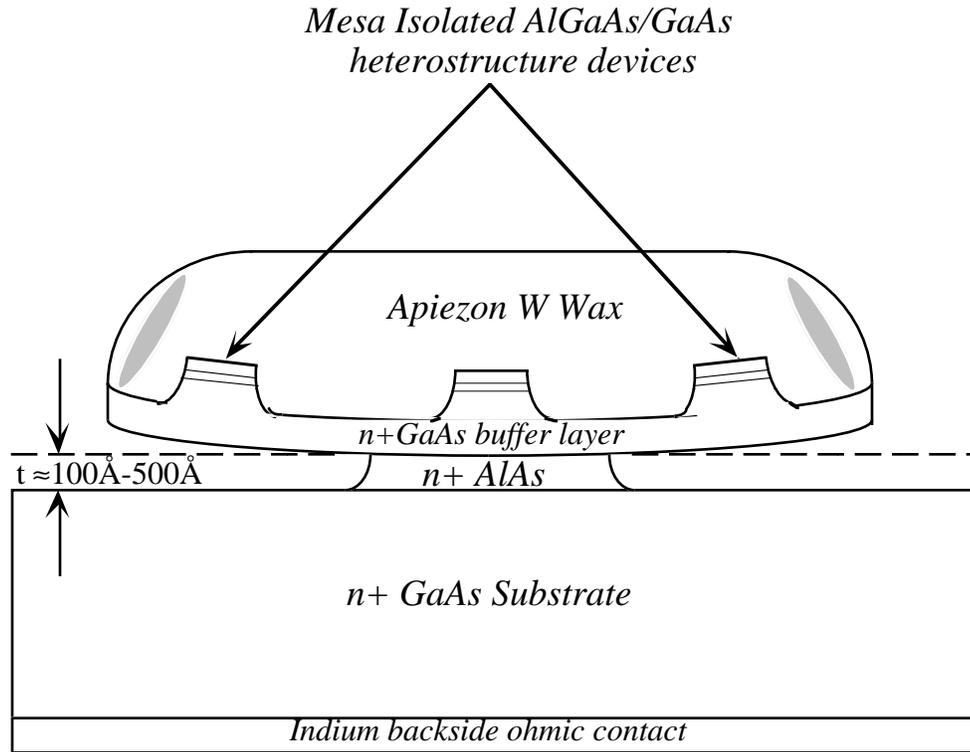


Figure 3.5: Illustration showing the edges of a mesa isolated AlGaAs/GaAs heterostructure (i.e., AlAs/GaAs double barrier resonant tunneling diodes, AlGaAs/GaAs double heterostructure LEDs) being peeled back by the tension in the Apiezon W black wax as the AlAs release layer is being etched out by 10% HF.

With the larger ELO films, care must be taken that the film does not separate from the substrate and reseat back onto the substrate. In these situations, the ELO film may re-adhere to the GaAs substrate and become very difficult to remove. In ideal situations, once the ELO film has completely separated from the substrate, the black wax carrier and the ELO film will float to the top of the 10% HF. In most situations, though, the wax carrier and film will separate from the substrate and then slide away from the substrate but still remain underneath the etch solution. In an effort to circumvent this

problem, the ELO film can be suspended in the etchant with a thin piece of HF resistant teflon or wire, as shown in Figure 3.6.

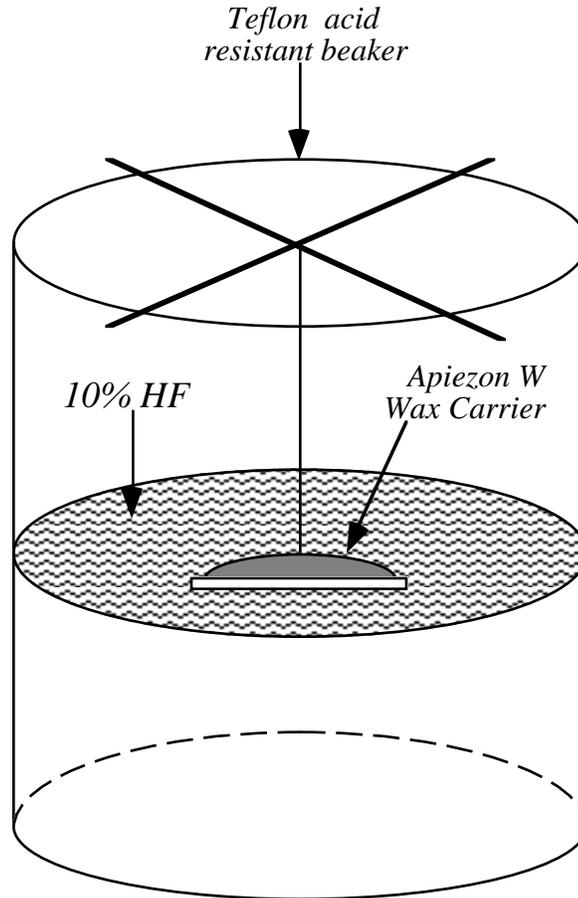


Figure 3.6: Illustration showing the suspension of an ELO sample in the 10% HF. Upon complete separation, the substrate drops away from the ELO film.

With the ELO film suspended, re-adhesion to the substrate is avoided and the ELO film is prevented from touching any part of the teflon beaker or settling down at the bottom of the beaker where damage to the thin epitaxial film may occur.

Manipulation of the thin ELO films after they have been released from their substrates involves first the complete dilution of the 10% HF and the subsequent use of a pen vacuum probe with a rubber or teflon cup. The dome shape of the wax carrier makes it easily handled by the pen vacuum probe. Forceps or tweezers have

been used and found to cause cracks in the ELO film, even when the edges of the wax carrier are held. The pen vacuum probe is tied to a water trap, thus allowing underwater manipulation of the ELO film.

3.5 Issues Regarding Bonding of ELO films

Whereas lifting off the ELO films from the growth substrates is a relatively straightforward and easy task, bonding the ELO film to alternative substrates without any cracks, bubbles, or particulate contamination can be very difficult. The process depends not only on the cleanliness in the process, but also on the type of adhesives or bonds that are used as well as the properties of the thin film and the substrate. Adhesion can be defined as the holding together of two surfaces by valence forces, mechanical anchoring, or by a combination of both. At the interface between a film and a substrate, a thermodynamic expression can be used to describe the work required to separate the film and the substrate [Ohr92]:

$$W = \gamma_f + \gamma_s - \gamma_{fs} \quad (3.5)$$

where γ_f is the specific surface energy of the thin film, γ_s is the specific surface energy of the substrate, and γ_{fs} is the specific interfacial energy. Thus, the properties of the film and the substrate play an important role in the adhesion between the two. For $W > 0$, there is adhesion and for $W < 0$, there is repulsion. Since metals have a high surface free energy, when they come into contact with each other, they have a tendency to adhere. This property of metals is the reason why the eutectic-metal-bonding (EMB) method, mentioned in section 3.11, has utilized a bond between a metallized substrate and a metallized thin film. The adhesion also depends on the chemical interaction and interdiffusion that occurs between the two films. In the following sections, 3.6 and 3.7, a discussion of the various possible bonds used for the ELO films will be presented.

3.6 Van der Waals (VDW) Bonding of ELO Films

Once the ELO films, which are as thin as 100\AA , have been separated from their original substrates, the black wax is used to act as the carrier and to provide

mechanical support. The ELO GaAs films can be bonded to various smooth substrates through the use of weak, long range electrostatic van der Waals (VDW) forces [YaH90]. The various substrates that can be used are any dielectric (i.e. glass, quartz, sapphire, Al₂O₃, etc.) and semiconductor (GaAs, Si, InP and diamond). Van der Waals bonds cannot be made to metals since their electrostatic charges overlap. To achieve a reliable van der Waals bond, a couple of conditions must be met. First, the environment under which the bonding takes place must be absolutely clean. Dust particles not only prevent a good mechanical contact to the substrate, but may also outgas, form bubbles and damage the film. Etching the substrates and using an O₂ plasma to remove any organic residues is useful in improving the van der Waals bonds. In addition, if there is no cleanroom environment in which to perform the bond, bonding in DI-H₂O is very useful [TsI92] [YaS91]. Second, the total bond strength is area dependent and the use of larger area films may help increase the bond strength between the substrate and the film. Films on the order of 1 in² have been lifted off and VDW bonded to other substrates. Furthermore, the combination of surface tension forces from the DI-H₂O, pressure as the sample is being bonded, and the temperature of the sample during the bond, all contribute to a reliable VDW bond. It has been noted that with DI-H₂O in between the epitaxial film and substrate, an inequality between the surface free energies of the substrate/film γ_{sf} , the water-substrate γ_{ws} , and the water/film γ_{wf} exists such that the film and substrate have a tendency to come together, forcing the DI-H₂O outward [YaH90].

$$\gamma_{sf} < \gamma_{ws} + \gamma_{wf} \quad (3.6)$$

With the subsequent application of pressure, the majority of the water is pressed out from underneath the film. The sample is usually left under pressure until all the water has been driven out from underneath the ELO film through extremely small diffusion paths. Raising the temperature of the sample to about 50°C to 60°C aids in the removal of water and the formation of a reliable VDW bond to the substrate.

An improved method to obtain VDW bonds to substrates is through the use of a vacuum bag [TsR91]. The vacuum bag can be inserted in the DI-H₂O environment

where the sample and substrate are placed. In this DI-H₂O environment, a Kim-wipe can be placed on top of the sample/substrate combination. With the vacuum line tied to a water trap, the vacuum can be used to draw all the water out of the bag and into the water trap.

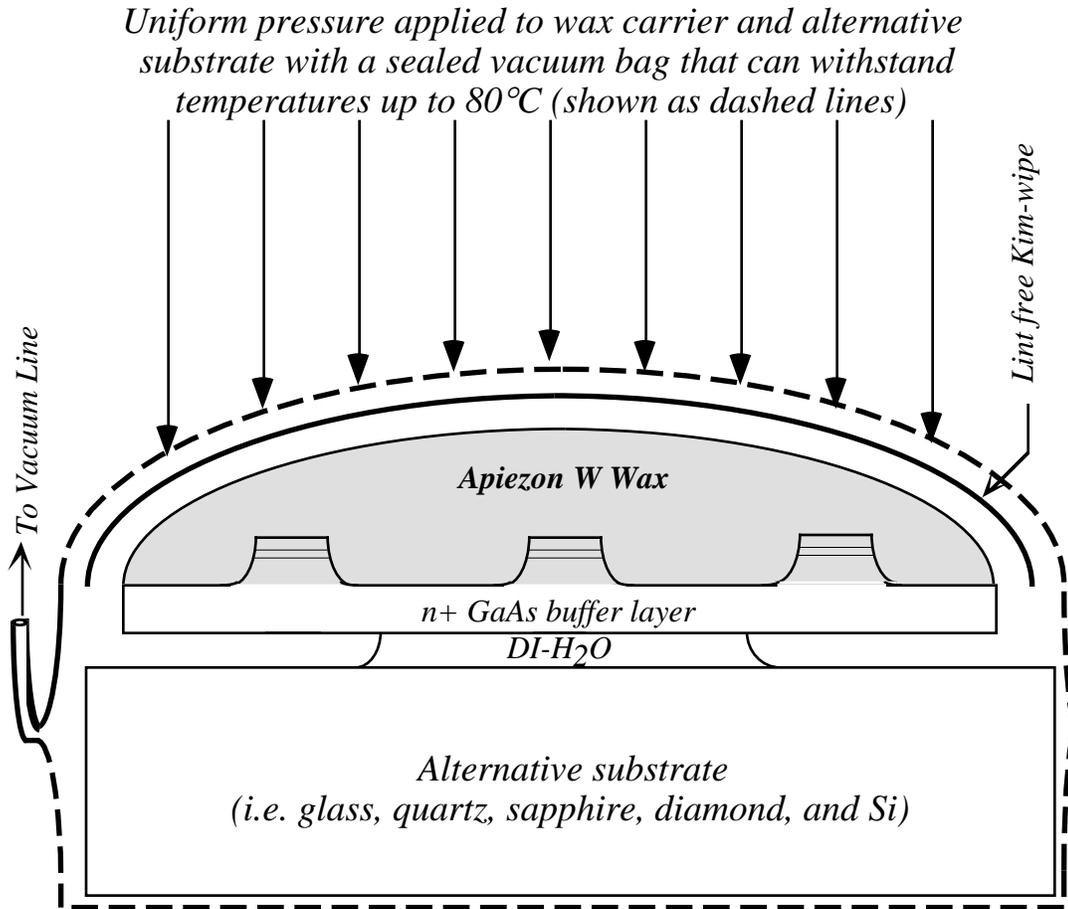


Figure 3.7: Illustration of uniform pressure applied by vacuum bag to wax carrier as ELO film is pressed together with an alternative substrate for a VDW bond. A lint free Kim-wipe is placed between the wax carrier and vacuum bag to prevent pinching off of the vacuum. This process can also be used on the metal/alloy based bonds.

Once the water is removed, the sample and substrate are pressed together with uniform and even pressure on top of the black wax carrier, as shown in Figure 3.7.

With the vacuum line touching the Kim-wipe, one is guaranteed in preventing the vacuum bag from pinching off the vacuum and therefore the uniform pressure over any portion of the ELO sample. The vacuum speeds removal of moisture from underneath the ELO film. In addition, the vacuum bag can be placed in an oven for any raised temperature bonding cycles that may be required. The pressure/temperature variables are very important parameters for a good VDW bond. Pressure as high as 15 lb/in² have been used without any breakage or cracking in the epitaxial film [TsR91]. The temperature should be kept below the softening point of the wax carrier unless specifically testing the elasticity of the ELO film. In this work, pressures between 7-15 lb/in² and temperatures from 30°C - 60°C have been used. This technique is an improvement over the simple placement of a weight on top of the black wax carrier. In addition, the pressure can be adjusted with a pressure regulator.

Avoiding bubbles in the ELO films bonded to other substrates is very difficult even when using a clean room environment or DI-H₂O environment. If there are any asperities or defects which may trap moisture or gas, bubbles may form after bonding and degrade the overall bond to the substrate. In addition, when patterning and mesa isolating these ELO films down to the alternate substrates, the overall reliability of the VDW bond is degraded because the total surface area has been reduced. If the patterned ELO films are very small, on the order of 100 μm², it has been observed that their bonds to the surrogate substrate do not survive. Demeester and co-workers report similar findings, but also indicate that the bubble size is proportional to the thickness of the epitaxial film thickness [DeP90]. Therefore, the VDW bonds of thinner films that are mesa isolated all the way down to the surrogate substrate have a higher percentage survival rate over thicker films. This author also observes the same trends for thinner and thicker films, but feels the trend may be more a function of the etch time required to completely mesa isolate these films; thinner films require less time in the etchant than the thicker films. The etchant, usually peroxide based, can be quite exothermic as well as turbulent and can separate the ELO films from their hybrid substrates. Combined with a reduced mesa size, the films ability to adhere to the substrate is degraded. Bubble size and bubble density in the ELO films seem to be more a function of the actual bonding technique, substrate surface, and particulate contamination.

3.7 Adhesive and Metal/Alloy Based Bonds to Alternative Substrates

With the limitations of the VDW bonds, other bonding methods have been investigated. The two primary methods are adhesive based bonds and alloy based bonds. The type of bond is basically determined by the type of device and alternative substrate one is working with. For a two-terminal device structure with one contact on top and the backside used as another contact, both a mechanical and electrical contact must be made to the backside of the ELO layer. For a planarized microwave/millemeter wave device, where all the electrical contacts are made on the top, it may be more appropriate to place this type of ELO device on an insulating substrate where the dielectric constant is lower than that of the original GaAs substrate. For optical studies of quantum wells and bulk material, it is sometimes desired to obtain complete transmission through the film which can only be accomplished if it is put on a transparent substrate. Furthermore, for devices which generate significant amounts of heat, such as semiconductor lasers and impact ionization avalanche transit time (IMPATT) diodes, bonds to substrates with very high thermal conductivity are important. Here, the adhesive or metal/alloy must, itself, have a high thermal conductivity.

Adhesive bonds to transparent substrates were initially investigated by Yablonovitch and co-workers [YaK89]. Here they used Norland Optical Adhesive 61 which is a clear, transparent, optically curable liquid photo-polymer. In addition, cyanoacrylates have been used primarily in this work when bonding ELO films to transparent substrates. Whereas the VDW and metal/alloy bonds are performed in a DI-H₂O environment, the adhesive bonds usually involve immediate transfer of the ELO film from the completely diluted etch environment to the alternative substrate. The main benefit of these optically transparent adhesives is that they allow optical contact to the back of the ELO device layers. Furthermore, these optically transparent bonds have the ability to survive complete mesa isolation to the substrate whereas the VDW had a tendency to fail when the ELO film was mesa isolated. In Figure 3.8, $\approx 16\mu\text{m}^2$ blocks of GaAs were mesa isolated to the quartz substrate. Here the epitaxial GaAs film was 3 μm thick. When using cyanoacrylates as an adhesive, care must be taken not to get the cyanoacrylate on the black wax carrier since the cyanoacrylate does not dissolve in TCA and makes subsequent removal of the wax carrier difficult.

Care must be taken not to allow the acetone to attack the cyanoacrylate bond between film and surrogate substrate. Quick rinses in acetone are usually not a problem.

The use of conductive silver-based epoxies (Epo-Tek H20E, H20S, and H20D) and polyimides (Epo-Tek P10) was also investigated as a means of obtaining an electrical contact to the backside of the ELO layers. Since these epoxies and polyimides required thermal curing, the vacuum bag/oven combination was used. The results on all of the silver-based conductive adhesives were not adequate to provide a damage free mechanical bond to the ELO films. The failure is primarily attributed to the size of the silver flakes which are dispersed in the epoxies and polyimide. The thickness of these flakes appear to be as thick as the ELO films, causing significant cracking in the ELO films and poor mechanical contact to the substrate.

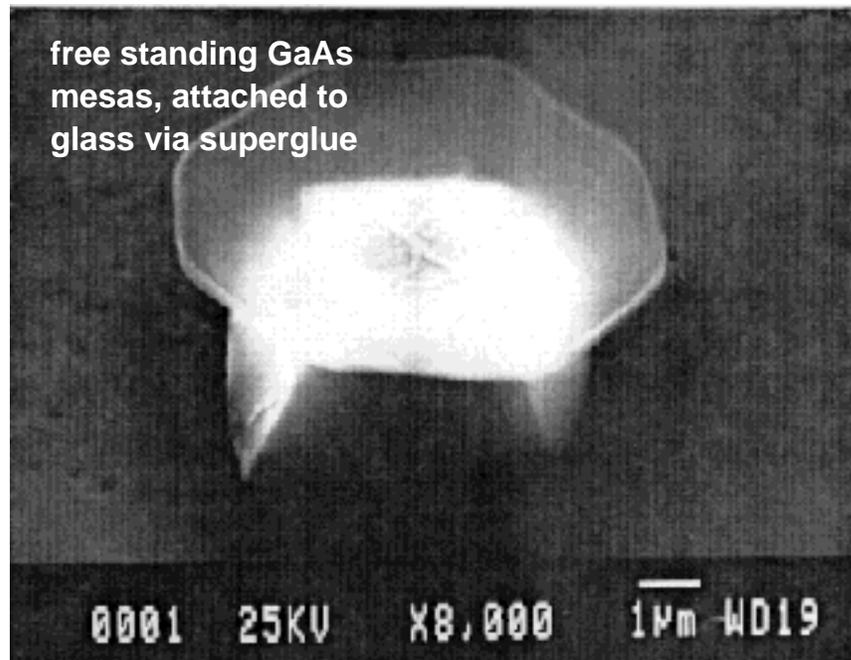


Figure 3.8: SEM micrograph showing the complete mesa isolation of an 3 μm thick ELO film into $\approx 4\mu\text{m}$ by $4\mu\text{m}$ islands. The "mushroom" cap on top of the GaAs island is the remaining photoresist after the mesa etch.

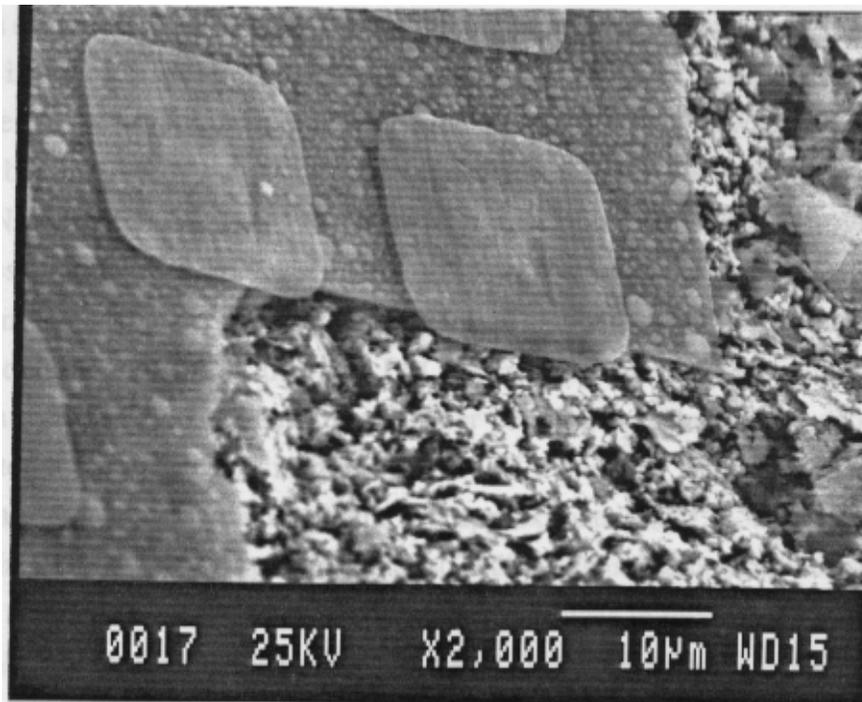


Figure 3.9: SEM micrograph showing an ELO film that has been bonded to P-20 silver polyimide. Note that the size of the silver flakes appear to be on the order of the ELO film thickness and does not provide a smooth flat bond line.

For electrical contacts, especially ohmic contacts, to the backside of ELO layers, the metal/alloy based bonds have been much more promising. Some of the earlier efforts include bonding of the ELO film directly onto Au-coated substrates [YoD91] [YoF91] and by the deposition of AuGe/Ni onto the backside of the ELO layer just after it had separated from the substrate [PoD90]. From the metal-semiconductor contact literature, Au can react with GaAs through a dissociative diffusion process at fairly low temperatures including room temperature, but can also result in severe pitting in the GaAs with pit dimensions larger than a micron once the sample is annealed [WeF88]. Furthermore, it is well known that Au contacts to GaAs result in nonuniform, spiking contacts. No discussion was given in these works about the specific contact resistivity of the backside contact. Evaporation of AuGe/Ni onto the backside of ELO films in this research has proven to be unsuccessful due to severe deformation of the wax carrier and cracking of the ELO film from the heat generated by the evaporation sources. Another method to obtain ohmic contacts to the backside of the ELO layers involves directly bonding the ELO layer onto In/AuCr coated substrates. After bonding the films to the substrates is complete, the wax carrier is removed in TCA and the ELO sample/substrate combination is alloyed at a temperature of approximately 350°C, which results in a graded InAs/InGaAs/GaAs ohmic contact [LaK84]. This method has been shown to provide adequate ohmic contacts to some of the devices in this work. Although the electrical ohmic contact to the backside of the ELO sample is adequate, the mechanical contact was rather poor and did not survive a simple "Scotch" tape test. A typical bond to a In/AuCr substrate is shown in Figure 3.10. In an effort to improve the mechanical bond to the In, flux was applied to an In coated substrate before an ELO sample was bonded to it [Cam91]. The surface morphology seemed to be slightly improved, but the bond still did not survive a tape test. Yablonovitch and co-workers were the first to find an improved bonding method to any type of substrate through the use of a palladium, Pd, based bond [YaS91]. In this study, bonds to substrates coated with Pd were found to occur at low temperatures ($\leq 200^\circ\text{C}$) including even room temperature, provided an ohmic contact to heavily p and n-type doped GaAs ELO films, and were quite reliable and strong even surviving "Scotch" tape tests. The basis for this improved bond is the low temperature solid phase reaction between

GaAs and Pd which forms Pd₄GaAs similar to the dissociative diffusion process between Au and GaAs mentioned earlier. The Pd displaces any native oxides and reacts with the GaAs. With Pd based contacts to GaAs, it is well known that they are non-spiking and therefore produce a much more uniform and smooth interface. Regardless of the topography, even if it is slightly nonplanar, the ELO film can elastically conform to the substrate and react with the Pd. Therefore, this reaction process has been referred to as a solid-phase "topotaxial" reaction [YaS91]. This process has been extremely useful in this research allowing a wide variety of bonds to many different substrates such Si, quartz, and copper. A typical SEM of an ELO bond to Pd is shown in Figure 3.11.

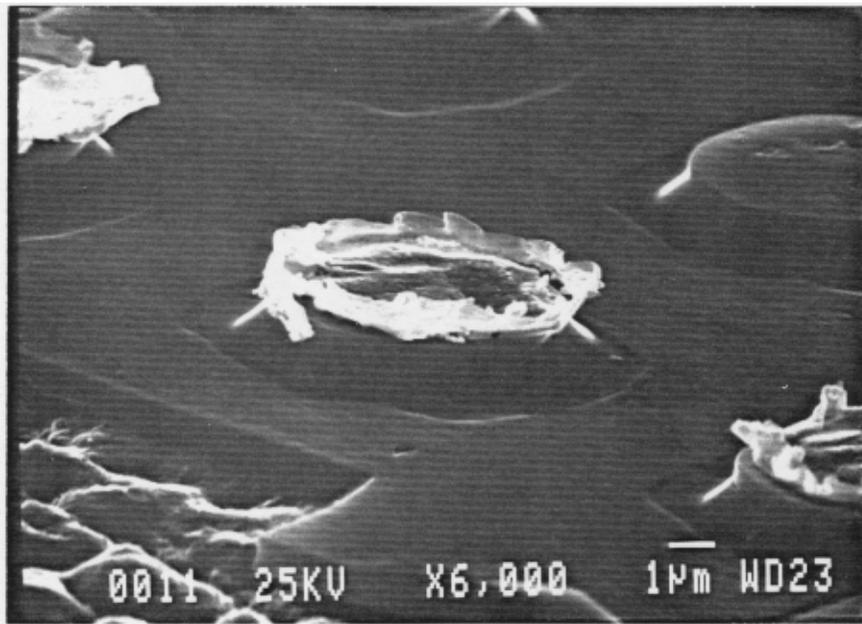


Figure 3.10: SEM micrograph of an ELO device bonded to an In/AuCr coated glass substrate. The specific contact resistivity of the ohmic contacts were indirectly extracted and found to be $8 \times 10^{-6} \Omega\text{-cm}^2$. The mechanical contact and bond strength were poor. Note the roughness of the In at the edges of the ELO film.

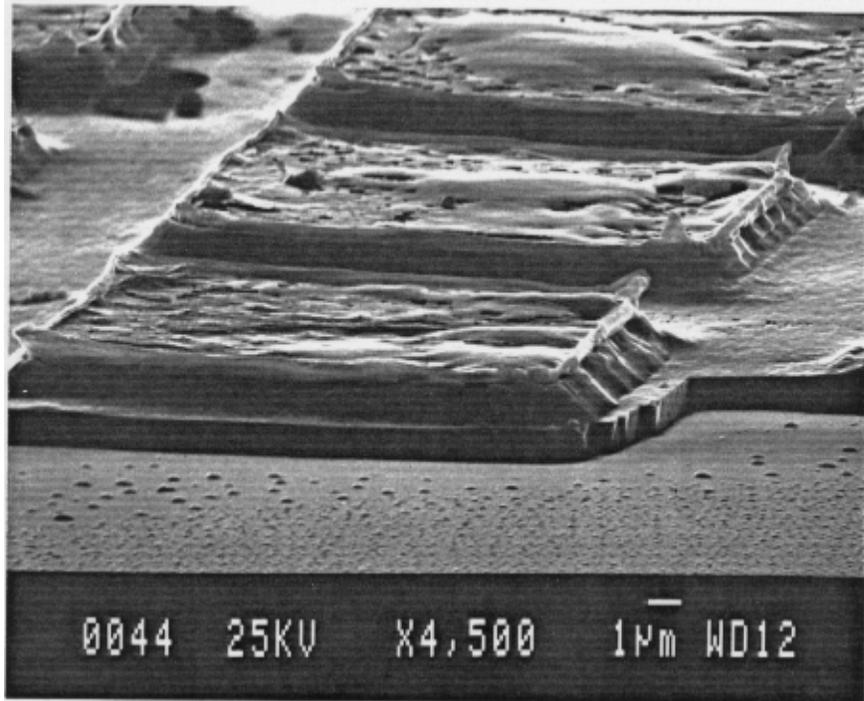


Figure 3.11: SEM micrograph of an ELO device bonded to a Pd/AuCr coated Si substrate. The electrical ohmic contacts were indirectly extracted and found to be $\approx 1 \times 10^{-5} \Omega\text{-cm}^2$. The mechanical contact and bond strength was very good. Note the smooth morphology of the Pd surface and the excellent bond at the edges of the film.

3.8 Hybrid substrates used in ELO

One of the optimal alternative substrates is silicon which provides the opportunity to integrate AlGaAs/GaAs devices with silicon devices. Furthermore, the surrogate silicon substrate acts as a better heat sink with ≈ 3 times the thermal conductivity than that of GaAs. In addition, as stated earlier in Chapter 2, the silicon substrates are more rugged and cost significantly less than GaAs substrates. Alternative quartz substrates also offer special advantages since they are transparent and have a lower dielectric constant than the GaAs substrate. Substrates with large electro-optic coefficients and low losses, such as lithium niobate (LiNbO_3), were found to be useful in coupling ELO metal-semiconductor-metal (MSM) detectors with the LiNbO_3 waveguides [YiC89]. Substrates of even higher thermal conductivity, such as copper, diamond IIa, and silicon carbide, have become viable candidates as hybrid substrates for an ELO device through the use of Pd based epitaxial bonds. A discussion of bonding double barrier resonant tunneling diodes to substrates of higher thermal conductivity will be presented in Chapter 5.

Pre-patterned or nonplanar substrates of any type, (copper, Si, glass, LiNbO_3), are of interest from the viewpoint that the ELO film can be made to conform to the shape of the substrate or bend over the substrate. Within the elastic limits of the ELO film, it can be made to conform to the shape of the substrate without damage to the ELO film. Studies have been performed with ELO GaAs/AlGaAs quantum wells placed over pre-patterned substrates for purposes of examining the effects of strain induced lateral variations in the bandgap of the semiconductor [ChR92]. Furthermore, beyond the elastic limits of the ELO film, pre-patterned substrates can be used to define the manner in which the film is cleaved. In other words, the pre-patterned substrate can be used to define the physical dimensions of a certain device. In this situation, alignment of the film to the substrate becomes a very important issue. Methods as simple as marking or scribing the wax carrier before ELO and using these scribes as alignment keys after ELO can provide reasonable alignments to substrates. More advanced methods include the use of transparent polyimide diaphragms as transfer mediums for the ELO films [CaH91].

3.9 Epitaxial Lift Off of Strained Layer Films

The insertion of an AlAs release layer in the layer structure of any common AlGaAs/GaAs heterostructure device results in minimal strain in the films since the lattice mismatch is only about 0.1%. For the InGaAs/InAlAs heterostructures latticed matched to InP substrates, the insertion of an AlAs release layer results in considerable strain with a lattice mismatch of about 3.5%. If the typical AlAs release layer thicknesses used on AlGaAs/GaAs device structures, as shown in the previous illustrations, were used in InGaAs/InAlAs layers, there would be a significant amount of dislocations and defects. The motivation for applying the ELO method to the InGaAs/InAlAs system is that it extends the range of devices that can be studied, such as longer wavelength photonic devices [ScG89] and higher current density, higher r.f. output power double barrier resonant tunneling diodes (DBRTDs). The strain induced from these layer structures can impact the overall radius of curvature of the film. The critical layer thickness for InGaAs on AlAs is around 50Å and based on the discussion in section 3.1, any ELO layer structure with an AlAs release layer of $\leq 50\text{\AA}$ should not face any significant difficulties in allowing the 10% HF to completely separate the ELO film from its InP substrate. In our work, we have been able to lift off InGaAs/AlAs DBRTDs from their InP substrates using 40Å AlAs release layers. Such a structure is shown in Figure 3.9, which was bonded to a conductive polyimide on Si.

The metallization of the epitaxial films can also contribute to the overall strain that the film is exposed to. ELO layers with metallizations as thick as 1.2 μm and covering $\approx 90\%$ of the sample surface area were successfully lifted off with no observed difficulties [IsT91]. In fact, it is believed that this thick metallization provides additional support for the fragile ELO film during sample manipulation and bonding. Pollentier and co-workers have shown that Pd/Ge metallizations induced the least amount of stress compared to AuGe/Ni, AuZn, and TiW/Au metallizations [PoZ91].

Although ELO has been proposed as an alternative for GaAs on Si growth, there has been work performed that combines the two technologies in an effort to relieve the strain in the heteroepitaxial GaAs on Si [DeV91] [BuF92A] [BuF92B]. This method, referred to as mesa release and deposition, MRD, by some, involves

the insertion of an AlAs release layer on heteroepitaxially grown GaAs on Si. The device layers are subsequently processed and mesa isolated down past the AlAs release layer. Then through the use of a "photoresist clamp", rather than the usual Apiezon W black wax carrier, the AlAs release layer is etched out, thus relieving the strain in the GaAs film, and subsequently rebonding the layer back onto the Si. This method has the advantage of placing strain free GaAs films on Si, just like in the standard ELO method, but also has the advantage of not having to deal with the difficulties involved with sample transfer and manipulation of very thin and fragile epitaxial films. The MRD method also avoids having to use solvents such as TCA to remove a black wax carrier and instead rely on the simple removal of the photoresist with acetone. This method does not address issues concerning ohmic contacts to the backside of the lifted off layers and apparently requires a planarized process. The MRD method solely addresses the issue of strain relief in GaAs on Si films and does not allow the benefits that are intrinsic to the ELO process, such as bonding to alternative substrates that are transparent or of higher thermal conductivity.

3.10 Epitaxial Lift Off of Silicon on Insulator (SOI)

As a proof of principle, the ELO method was performed on some silicon on insulator (SOI) material obtained from IBM [Kes91]. A typical cross-section of the SOI material used is shown in Figure 3.12.

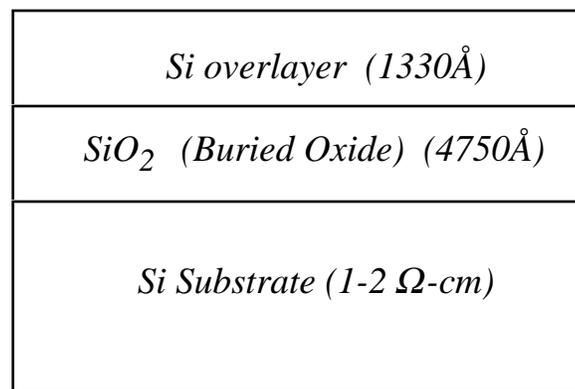


Figure 3.12: Cross-sectional schematic of a silicon-on-insulator (SOI) sample used in the ELO process. A standard 49% HF solution was used to etch out the oxide as the Si overlayer was held to a Apiezon W black wax carrier.

The application of the Apiezon W black wax and ELO process used on this SOI sample were very similar to the AlAs/AlGaAs/GaAs structures described in the previous sections. The only major difference here was the use of a standard 49% HF solution to etch out the oxide from underneath the Si overlayer. The SiO₂ (oxide) underneath the Si overlayer is not thermally grown oxide but created through a three-stage oxygen implant and anneal process [Kes91]. Although no data could be collected on the etch rates of this type of oxide in 49% HF, a figure of merit that can be used is the 300Å/second etch rate of thermally grown oxides at 25°C [WoT86] . Note that this etch rate of thermally grown SiO₂ is much slower than the quoted etch rates of AlAs, which are about 50-60 μm/minute [DeP90]. With these much slower etch rates, the complete separation of the Si overlayer from its substrate required about 2 days for a 1cm² sample. Upon complete separation from the substrate, the sample was VDW bonded to a quartz slide. Once the Apiezon W black wax was removed from the ELO Si film, it was observed that the film was multi-colored, which was probably due thickness variations in the original Si overlayer that could be observed before ELO. The motivation for this experiment was more a test of principle rather than using ELO as a tool for fabricating thin Si film devices. In the silicon area, the technologies are much more mature including those used in developing extremely thin film devices such as the Bond and Etchback SOI (BESOI) method [Las86] [Hun88] and the use of a high energy boron implant that can be used as an etch stop combined with BESOI [MaP91] . Using the BESOI method, four inch wafers have been thinned to 1000Å ± 100Å for very thin, single crystalline substrates used for fabricating CMOS devices [Mas91] . The current level of development of the ELO process and the fact that the etch rate of the oxide underneath the Si overlay is ≈ 30 times slower than that of AlAs does not provide a viable alternative to the more developed BESOI technologies.

3.11 Alternative Processes other than the ELO Process

Other thin film monolithic/hybrid integration methods include GaAs on Si MBE growth, bonding by atomically rearrangement (BAR) where currently InP/InGaAsP structures and possibly GaAs/AlGaAs structures are bonded to silicon

[LoB91] and bonding by eutectic-metal-bonding (EMB) where GaAs/AlGaAs epitaxial films are grown on lattice matched and nearly thermal matched Ge substrates [VeT92]. The disadvantages associated with GaAs on Si MBE growth have already been previously mentioned in Section 2.6 of Chapter 2. Both the BAR and EMB methods have the disadvantage of having to completely etch back the substrate, generating a significant amount of GaAs waste by-products. In addition, both methods use a fairly high ($\geq 400^\circ\text{C}$) temperature cycle for bonding GaAs to Si. Although all the methods described above are capable of integrating extremely thin layers of GaAs with Si, the ELO technique has proven to be the most versatile in allowing GaAs integration with almost any substrate, including nonplanar and transparent substrates. Anodic bonding of GaAs to glass has been investigated for purposes of attaching optoelectronic devices to optical fibers [HöC83], but the process is rather involved requiring temperatures of 360°C and applied voltages of 800V. The ELO method allows bonds to a wide variety of transparent substrates such as glass, quartz, sapphire, and diamond.

For InGaAs/InAlAs films grown lattice matched to InP substrates, a highly selective HCl substrate back-etch provides a useful alternative to the ELO method [YiC90]. Since the ELO method requires the insertion of an AlAs release layer, which induces strain in the InGaAs/InAlAs films, an alternative method may involve etching back the InP substrate with HCl which has a high etch selectivity over InGaAs. Furthermore, for device structures which may already have strain from the intrinsic device structure itself, such as AlAs/InGaAs structures, the additional strain from the AlAs release layer may not be desired. Thin film, substrateless AlAs/InGaAs double barrier resonant tunneling diodes have been fabricated using this substrate back-etch technique and were topside bonded to alternative substrates using silver epoxy. Selective back-etching of GaAs substrates that have AlGaAs etch stops have also been performed using $\text{NH}_4\text{OH}:\text{H}_2\text{O}_2$, but the etch selectivities are not as high and not as controllable [AnE75] [LeP80]. Again, the drawback to these substrate back-etch methods is the amount of waste by-products produced in selectively etching away the whole substrate to get the desired thin films whereas the ELO method etches away an extremely thin AlAs release layer and leaves behind a reuseable substrate.