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# Real-Time Monitoring and Analysis of Chemical Wet Etching of III-V Compound Semiconductors

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ABSTRACT

We report the use of optical interferometry for *in situ* real-time monitoring and analysis of chemical wet etching processes in AlGaAs/GaAs and InGaAsP/InP structures with or without masked regions. Interference between reflections from the etching surface and buried heterointerfaces in the wafer allows determination of the etch depth and any degradation of surface quality, identification of heterointerfaces, and detection of changes in etch rate and end point in real-time. We have used a sensor consisting of a 0.95-μm light-emitting diode and four detectors enclosed in a 12.5-mm-diameter transparent Teflon tube which is kept in front of the substrate being etched. The etching process can be stopped as soon as the desired etched depth is achieved. The technique can be useful particularly in regrowth requiring etching of base epitaxial structures, fabrication of lasers, and many other devices, and development of an etching process.

Chemical wet etching plays a prominent role in fabricating III-V semiconductor devices. The success of a device manufacturing process depends critically on the high controllability, wafer uniformity, stability, and reproducibility of the etching process. This demands a detailed knowledge of the etching properties of the various epi layers, both individually and in combination. Despite numerous excellent books and review articles on chemical wet etching, most chemical etches currently in use have been developed on an empirical basis and neither the formulation of chemical reaction equations nor the kinetics is well understood. The etch rates and the etching profiles can be sensitive to the nature and density of dopants, surface orientation, crystallographic defects, alloy composition, surface condition, and the method of preparation of the material being etched. Often similar semiconductor films prepared by different methods or even by the same method but in different growth conditions etch at different rates. The etching behavior also has a complex dependence on the composition, temperature, agitation, and aging of the etching solution. These factors make the etching process less controllable and less reproducible than is usually desired. An *in situ* real-time measurement technique for etch rate, etch depth and end point determination, and heterointerface identification therefore, would be a great asset in device fabrication.

## Principle and Experimental Details

Here we report the use of interferometry for *in situ* real-time monitoring and end point detection of chemical wet etch process of III-V semiconductors. The technique is simple to implement and interpret. While it provides no chemical information, it is a fast, accurate, and sensitive technique for studying an etching process. The technique requires that the film has a smooth surface and is at least partly transparent. It has been in use[1-5] for several years as an *in situ* process diagnostic for dry etching, deposition of materials, and many other commercial processes. However, we are not aware of its use in monitoring the chemical wet etch process. We have used a commercially available (Luxtron Corp.) end point controller, Model 2200, which is used primarily in the Si industry to monitor etching of metallic and dielectric layers and developing photoresist to eliminate process variations.[6] The equipment uses a sensor with a light-emitting diode (LED) emitting at 0.95 μm and four photodetectors, two on each side of the LED, all contained in a transparent pen-like Teflon tube which is 1.25 cm in diameter and 15 cm long. The substrate can be placed horizontally or vertically parallel to the sensor head. When the sensor is in close proximity of the sample being etched, the light from the LED strikes the wafer surface and is reflected back to the detectors. The light is reflected from the etching surface and from the buried AlGaAs/GaAs or InGaAsP/InP heterointerfaces or the polished rear surface of the wafer. The constructive and destructive interference between the two beams cause the intensity of the reflected light to oscillate between maximum and minimum levels, with a period ($\Delta d$) given by[2]

$$\Delta d = \lambda/2\sqrt{n_2^2 - n_1^2 \sin^2 \theta_i} \quad [1]$$

where $\lambda$ is the wavelength, $n_1$ and $n_2$ are the refractive indexes of the etching solution and the etched material, respectively, and $\theta_i$ is the angle of incidence with respect to the normal. Equation 1 can be dervied using the laws of geometrical optics. Maximum depth resolution is obtained at normal incidence. The average intensity of the reflected beam depends on its polarization, the angle of incidence, the refractive index of the top layer, and most importantly on the quality of the etch surface. The amplitudes of oscillation depend on the light absorption, refractive indexes, and the interference condition from underlying layers. Thus, the technique also allows qualitative determination of the surface quality during etching. Experimentally, we find that if the etching process degrades the surface, average reflected beam intensity goes down and the oscillations in reflected beam intensity are either not present or decrease in amplitude continuously. Since we are not using a collimated beam, the light strikes the surface at various angles, and a composite reflected signal is detected which is sensitive to any change in reflectivity and the film thickness. At the inflection points, a 1% change in reflectivity

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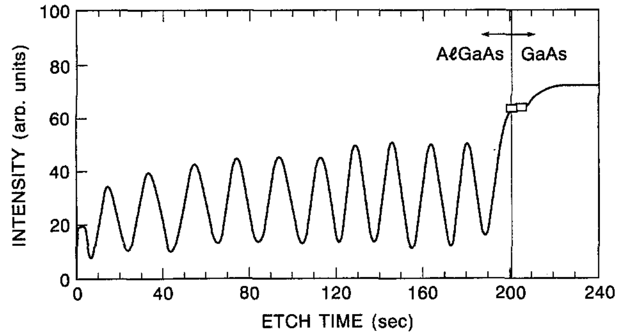


**Fig. 1. An interferogram (λ = 0.95 μm) taken during etching of a 1.55 μm-$Al_{0.28}Ga_{0.72}As$/GaAs structure in $3NH_4OH$:$1H_2O_2$:50 $H_2O$ solution.**

can be measured and corresponds to the removal of roughly 5 Å material. To use Eq. 1, accurate values of $n_1$, $n_2$, and $\theta_i$ are required, which can be determined for a given system from the oscillations while etching to a known depth. The desired information, particularly the end point, can be determined without the knowledge of these parameters, as is demonstrated here.

We have used a LED at 0.95 μm, but a shorter wavelength can be used to enhance the resolution for studying optically thin layers, or a longer wavelength to minimize absorption. The 0.95-μm radiation was partly absorbed in both GaAs and InP which prevented reflections from the rear surface of wafers. When the reflectance is monitored over a region of the wafer containing both etched and unetched areas (photoresist, oxide, or any other mask), the results can be affected by lateral interference, which is the interference between the light reflected from the different surface regions. Heimann's study[7] shows that the lateral interference has only a minor effect when less than 30% of the surface area being monitored is covered by an etch-resistant layer, which is usually the case when etching mesas for laser diodes. Indeed, we observe no noticeable effect of 7-μm wide oxide stripes on 250-μm centers on the measured interferogram. Serious distortions can occur, however, when a larger fraction of the region is covered by the mask. Furthermore, when the light is incident on and reflected from a multilayered structure with multiple reflections, the waveform can be complicated and a full understanding of the wave shape and how it changes with thickness may require computer simulation.

## Results and Discussion

Figure 1 shows an interferogram obtained during etching of the 1.55-μm $Al_{0.28}Ga_{0.72}As$ on a GaAs substrate in $3NH_4OH$:$1H_2O_2$:50 $H_2O$ solution. Interference occurred between the light reflected from the eroding surface and AlGaAs/GaAs heterointerface. Figure 2 shows an interferogram obtained during etching of a 0.4-μm-$Al_{0.3}Ga_{0.7}As$/0.35-μm-GaAs/70 Å $In_{0.2}Ga_{0.8}As$/1.55-μm-GaAs/AlAs/4.24-μm GaAs/AlAs/GaAs heterostructures in $3NH_4OH$:$1H_2O_2$:50 $H_2O$. In the inset, we show an enlarged view of the interferogram obtained during the first two minutes of etching to illustrate the sensitivity of the technique to detect AlGaAs/GaAs and InGaAs/GaAs heterointerfaces. The interferogram in Fig. 3 was obtained during etching of a 0.98-μm triple $In_{0.2}Ga_{0.8}As$ strained quantum wells (QWs) graded index separate confinement (GRINSCH) laser structure in $3NH_4OH$:$1H_2O_2$:$15H_2O$. In this structure, InGaAs QWs were separated from each other and from AlGaAs GRIN regions by 100 Å-thick GaAs spacer layers. Furthermore, between the GaAs buffer and $n^+$-AlGaAs cladding layers, 10 periods 100 Å GaAs/200 Å $Al_{0.6}Ga_{0.6}As$ superlattices were used. The thicknesses of p-AlGaAs and n-AlGaAs cladding layers were 1.79 μm and 1.85 μm, respectively. In Fig. 4, we show the interferogram obtained while etching an InGaAsP/InP buried double heterostructure base structure.

The signal-to-noise ratio in all cases is so high that all the features of the structures are clearly discernible. They can be seen in Fig. 1-4 by changes in slope, period, amplitude, and average intensity of oscillations. Figures 1 through 3 deal with GaAs/AlGaAs/strained InGaAs QW heterosystems. Due to the lower refractive index of AlAs and hence of AlGaAs as compared to that of GaAs, the average light intensity is significantly lower during the etching of AlGaAs layers in all cases. In Fig. 1, the oscillations are observed during the etching of AlGaAs layer and a plateau is reached as soon as the AlGaAs is etched away because there is no other buried heterointerface to cause reflection. Thus, the method can be employed to etch the top AlGaAs layer in a device structure like the emitter in AlGaAs/GaAs

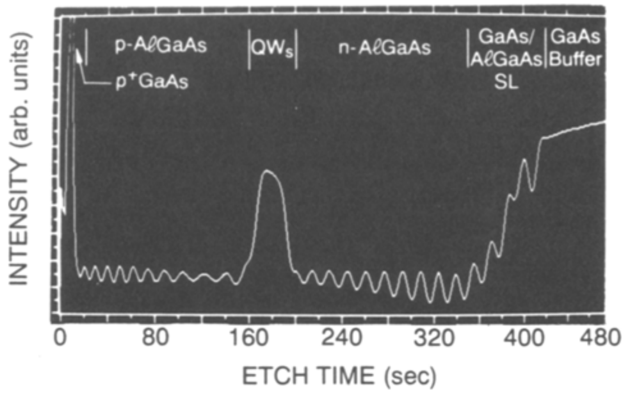


**Fig. 3. An interferogram (λ = 0.95 μm) taken during etching of an AlGaAs/GaAs/strained $In_{0.2}Ga_{0.8}As$ quantum well laser structure in $3NH_4OH$:$1H_2O_2$:$15H_2O$.**

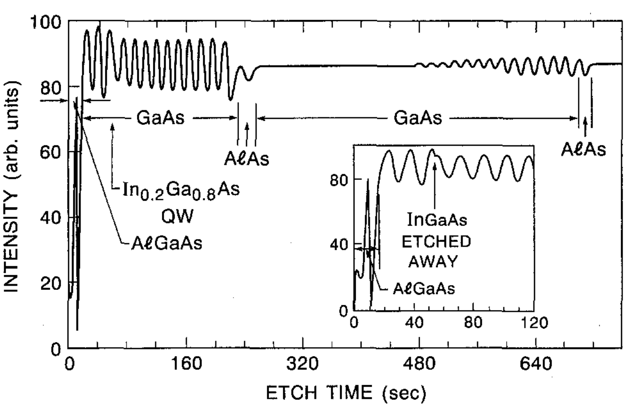


**Fig. 2. An interferogram (λ = 0.95 μm) taken during etching of a 0.4-μm $Al_{0.3}Ga_{0.7}As$/0.35-μm GaAs/70Å $In_{0.2}Ga_{0.8}As$/1.55-μm-GaAs/AlAs/4.25-μm GaAs/AlAs/GaAs heterostructure in $3NH_4OH$:$1H_2O_2$:50 $H_2O$ solution.**

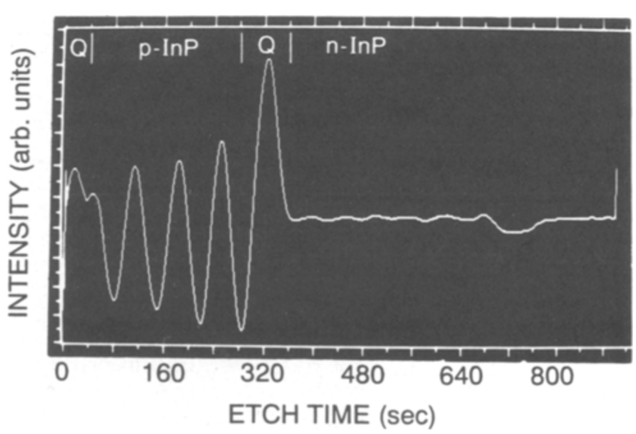


**Fig. 4. An interferogram (λ = 0.95 μm) taken during chemical wet etching of an InGaAsP/InP laser base structure.**

heterojunction bipolar transistors. Futhrermore in Fig. 1, the average intensity and the amplitude of oscillations increase due to reduced light absorption as the AlGaAs thickness decreases. Interestingly, the decrease of the period of oscillation toward the substrate suggests the increase of etch rate with time. From the total thickness and the etched time, the average etch rate is 0.465 μm/min. The distance between the two consecutive maxima is 0.14 μm. From this, we determine the value of $\sqrt{n_2^2 - n_1^2 \sin^2 \theta_i}$ as 3.3712.

The structure in Fig. 2 was grown for growth calibration proposes. Except for the top AlGaAs layer which was etched in the first 20 s, the entire structure is mostly GaAs with very thin InGaAs and AlAs layers used as marker layers. These thin marker layers have worked as good reflecting planes, and their positions are well revealed during the etching process. As soon as the top AlGaAs layer is etched away, the average light intensity increases significantly due to the higher refractive index of GaAs. Remarkably, the average intensity remains constant throughout the 13-min etching of GaAs suggesting no change in the surface quality of GaAs throughout the etching process. The variations in the amplitude of oscillations in GaAs are again due to light absorption. In GaAs, when the reflection is from a shallow InGaAs/GaAs heterointerface, the amplitude is higher. As soon as the InGaAs is etched away, there is a change of slope which is clearly visible in the inset of Fig. 2. During the etching of the AlAs marker layer, the oscillations amplitude goes down. After the etching of AlAs, the oscillations disappear because the other reflecting AlAs/GaAs heterointerface is 4.25-μm away from the surface and there is a total absorption of the weak reflecting signal. As the etching proceeds towards the AlAs/GaAs heterointerface, the oscillations reappear with increasing amplitude due to decreasing light absorption. These results suggest that thin layers of differing refractive indices can be used as the marker layers in device structures to monitor the etching process and determine the end points in realtime. These thin reflecting marker layers do not require any selective etch and are thus conceptually different from the so called "sacrificial stop etch" layers. When a selective etching is used, the overetching, which is usually a problem, now can be prevented using the above technique.

In Fig. 3, the oscillations from each region of the laser structure are distinguished clearly. Since the active region consists of GaAs/InGaAs QWs which have higher refractive indexes as compared to AlGaAs cladding layers, there is a marked increase in the peak intensity during etching of this region. A similar interferogram was obtained during etching of AlGaAs/GaAs QW laser structures. For the fabrication of ridge wavequide lasers or for the etching of cladding layers in laser base structures for regrowth purposes, the technique can be used to detect the end point.

The interferogram in Fig. 4 is almost identical to that obtained by Hayes and co-workers[2] using a 1.15-μm laser source during the reactive ion etching of a similar laser structure. The difference is that with a 1.15-μm source, these workers were able to see reflection from the rear face of the wafer. In our case, however, the 0.95-μm radiations are absorbed in the $n^+$-InP substrate and the oscillating signal is detected during the etching of the epi-structure alone. The weak oscillations in the n-InP cladding layer in Fig. 4 are due to reflections from the n-InP (buffer)/n-InP (substrate) interface due to Ga contamination of the buffer layer. The peak intensity is higher when etching through the InGaAsP active quaternary layer due to the higher refractive index of InGaAsP. A change in slope occurs at the InGaAsP (cap layer)/p-InP interface. By using a longer wavelengjth source, we expect to observe reflection from the rear surface of the substrate and hence the oscillations during the etching of the n-InP substrate as well.

## Conclusion

We have shown that the optical interference in the infrared is a powerful tool for controlling a chemical wet etching process. Both etch depth monitoring and end point detection can be performed with a high degree of precision. While we have used the method for AlGaAs/GaAs and InGaAsP/InP heterostructures, the technique is applicable for any combination of semiconductor materials with smooth surfaces.

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