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Measuring surface cleanliness of n-GaAs by ellipsometry

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IN THE fabrication of Gunn and LSA devices surfaces of GaAs must be carefully cleaned before contacts are alloyed. This letter describes an investigation using the method of ellipsometry, to evaluate the relative effectiveness of some of the cleaning and etching procedures presently used for this purpose.

Ellipsometry is an optical technique used to measure the optical constants of a surface (n,k) and the thickness of thin surface films much less than one wave-length thick. The method and technique of ellipsometry are described elsewhere, $^{(1,2)}$ and have been shown to be very sensitive to surface cleanliness. $^{(3)}$ Thus, the presence of a spurious film on the surface of a crystal alters the apparent optical constants of the crystal.

The ellipsometer consists of two polarizers, an optical compensator $\lambda/4$ plate, a reflecting sample (GaAs), and a photo-detecting system. Two quantities are measured: Δ and ψ . The quantity Δ is the change in the relative phase between the parallel (P) and perpendicular (S) component of the polarized light on reflection, and $\tan \psi$, the factor by which the amplitude ratio of the P and S components changes on reflection. The parameters Δ and ψ are determined by measuring the polarizer and analyser angles which give a null in the reflected light signal. (2)

It is well known that whenever a thin transparent film is present on the surface, the value of Δ decreases, and ψ is approximately constant. An oxide surface layer of the order of 20Å thick on aluminum or on silicon leads to a change in Δ by about 10°. A change in Δ by 1° can be readily measured. Thus, a procedure to find which of a series of etching and cleaning processes gives the 'cleanest' surface is to measure Δ after treatment of the surface by each of the procedures of interest. The procedure which gives the maximum value of Δ is the so-called 'best' process considered. This technique has been used to obtain the best optical constant of InSb. (4)

We have measured values of Δ for a slice of n-GaAs ($n = 6 \times 10^{14}$ cm⁻², $\mu = 6400$ cm²/V-sec, $\rho = 1.8$ Ω -cm, $\langle 100 \rangle$) suitable for fabrication of Gunn devices. This material was subjected to several cleaning and etching treatments commonly used just before contacts are applied. The results are shown in Table 1. Steps 1-4 are commonly employed before contacts are alloyed. As shown in the table, this sequence of treatments does improve surface cleanliness. Correlated observation⁽⁶⁾ indicates that increasing Δ by proper cleaning and etching techniques increases the reproducibility and uniformity of the ohmic contacts. The sequence of treatments 7 through 9 suggests that the sodium dichromate solution is mainly responsible for the improvement in surface cleanliness and that it in fact leaves a cleaner surface than the HF treatment. No correlation in improvement of the contacts was attempted by reversing the order of sodium dichromate and HF etch. The results of aging steps 3, 5, 6 indicate that the surface character of GaAs is relatively stable in air.

This study has shown that the above method of ellipsometry is useful in evaluating the cleanliness

Table 1. Ellipsometer measurements to determine surface cleanliness at $\lambda = 5460 \, \text{\AA}$

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Ch	emical and cleaning treatment of GaAs		Δ±1
(1)	Mechanically polished; clean with tri- chlorethylene, acetone and freen; ex- posed to room air for 24 hr		155.0
(2)	Etched with $3H_2SO_2+H_2O_2+H_2O$ for \sim 5sec; rinse with distilled water; blown dry with a freon duster		157.2
(3)	24 hr later in air		156.0
(4)	Etched with sodium dichromate solution (15 ml sodium dichromate and 75 ml of $\rm H_2SO_2$) and 48% HF for \sim 5 sec each; rinse in water; blown dry with freon duster	;	164.6
(5)	4 hr later in air		164.0
(6)	24 hr later in air		163.2
(7)	Repeat $3H_2SO_4+H_2O_2+H_2O$ for ~ 5 sec; rinse in water; blown dry, with freon duster		165.8
(8)	Sodium dichromate solution for \sim 5 sec; rinsed in distilled water; blown dry with a freon duster		172.0
(9)	Etch with HF for \sim 5 sec; rinse in water; blown dry with a freon duster	:	165.8

of the surface before the fabrication of Gunn, LSA, and other devices. This technique can be used for metals and most semiconductors (Ge, Si, InSb, InAs, etc.).

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