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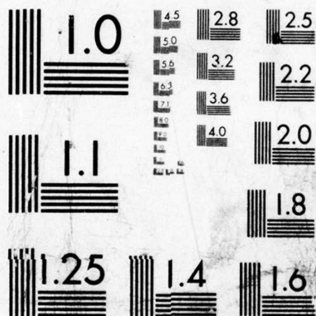
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ION IMPLANTATION OF WIDE BANDGAP SEMICONDUCTORS

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The principal dopant studied under this contract is Si (n-type). Topics covered in this report include (1) encapsulation technology, (2) transferability of Si implantation technology using plasma-deposited "silicon nitride" encapsulation, and (3) problems associated with performing channeling into [110] crystal axes of a (100)-oriented GaAs wafer.

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TABLE OF CONTENTS

Section		Page
1	INTRODUCTION AND SUMMARY	3
2	ENCAPSULATION STUDIES	5
3	Si TRANSFER TEST	8
4	CHANNELED Be IMPLANTS	16
5	DIAGNOSTIC IMPROVEMENTS	18
	REFERENCES	20

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SECTION 1

INTRODUCTION AND SUMMARY

The primary objectives of this program are to improve our understanding of the basic processes that control the electrical and physical properties of ion-implanted layers in GaAs and to exploit this understanding to develop a transferable technology base for reliable ion-implantation doping of GaAs. During this one-year contract, the primary effort was to study, through duplicate processing and evaluation projects at Hughes Research Laboratories (HRL) and the Naval Research Laboratory (NRL), the factors that affect the transferability and reproducibility of implanting Si into GaAs using "silicon nitride" as an annealing encapsulant.

The primary encapsulant deposition process being used for this program is plasma-enhanced deposition (PED) to react silane with nitrogen in the presence of argon. Both laboratories are using essentially identical commercial plasma reactors (LFE Corporation Model PND-301). The films deposited in these reactors are not pure Si_3N_4 but generally are silicon oxynitrides containing several atomic percent oxygen plus other contaminants such as hydrogen and traces of argon. Therefore, the term "silicon nitride," which denotes Si_3N_4, is not appropriate. The term "plasma nitride" will be used to denote the films actually prepared by PED.

Early in this report period, a substantial effort was made to establish gas flow conditions that would yield plasma nitride films with good encapsulation properties. Section 2 briefly describes the methods used and the results obtained. These preliminary studies yielded promising results, but reproducibility of the encapsulant properties was lacking. Section 2 also describes a new PED system to be constructed from Hughes internal funding. This system will have high vacuum capabilities, and it is hoped that the reproducibility problems can be overcome with this system.

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When both laboratories had developed their plasma nitride technology to the point that a duplicate processing experiment was warranted, a standard Si implant was performed into several samples at each laboratory. These samples were then independently encapsulated and annealed. All the samples were electrically evaluated at each laboratory. The results of this test of implant transferability are discussed in Section 3.

Recent Hall-effect measurements of the depth distribution of the electrical activity of Be-ion implants into GaAs performed by Code 5212, NRL, have indicated that channeling effects may cause significant departures of supposedly "random equivalent" implants from the LSS range distribution. A set of well-channeled <110> implants into well-characterized <100> GaAs was requested by Code 5212, NRL, for use as reference samples. The techniques used to prepare these samples are briefly described in Section 4.

The primary electrical evaluation technique used on this contract has been room-temperature Hall-effect analysis. This technique generally has been adequate to determine the success with which electrical activation of impurities has been achieved. As the plasma nitride encapsulants used improve, additional techniques will be required to assess electrical and atomic profiles, the degree of damage reduction achieved during annealing, and the generation-recombination mechanisms active within the implanted region. Section 5 describes improvements in analytic capability being instituted at HRL and suggests additional diagnostic tasks that would be useful for improving our understanding of ion implantation in GaAs in the near future.

During the period covered by this report, Mr. C.L. Ramiller left the Hughes Aircraft Company to assume a position with Intel Magnetics, Santa Clara, California. Mr. Ramiller had been responsible for the development of pyrolytic encapsulants for the annealing of implanted GaAs. Dr. K.V. Vaidyanathan joined Hughes Aircraft Company in August and will be primarily responsible for the encapsulation, implantation, and annealing studies.

SECTION 2

ENCAPSULATION STUDIES

Work on the development of pyrolysis and of the PED of silicon nitride layers for the annealing of ion-implanted GaAs continued during the past year. The pyrolytic films deposited at 600°C this year were excellent barriers to Ga diffusion but had mechanical problems that caused cracks to form on the GaAs surface during annealing. Three factors seem to influence this cracking: inherent stresses in the nitride films; the difference between the thermal expansion coefficients of the Si_3N_4 films and of GaAs; and the lack of plasticity in dense, stoichiometric Si_3N_4 films. These problems apparently can be partially overcome by depositing the film at high deposition rates.

Plasma-deposited nitride films were evaluated extensively. The films were deposited using our modified LFE Corporation Model PND-301 system. The HRL system has been extensively modified to render it "leak-tight." Also, the needle valve flow controllers have been replaced by Tylan Corporation mass flow controllers. The system pressure in the HRL system is monitored by an MKS Instruments Model 222 "Baratron" capacitance manometer.

As discussed in the earlier progress report, films deposited with the following parameters exhibited the best mechanical adhesion to the sample: nitrogen flow rate of 4.00 sccm, dilute silane (1.5% in Ar) flow rate of 35 sccm, sample temperature of 350°C, rf power of 65 W, and pressure during deposition of 0.460 mm Hg (61 Pa). SEM examination revealed that films deposited at silane flow rates higher than 35 sccm tended to blister and that those deposited at silane flow rates lower than 35 sccm tended to craze. The GaAs surfaces below films that blistered were heavily pitted, while the surface of samples annealed with silicon-deficient films showed ridges, presumably resulting from the formation of Ga_2O_3 in the area beneath the cracks.

Rutherford backscattering (RBS) and Auger analysis have been used to analyze the composition of the nitride films. In all cases, the deposited film is silicon oxynitride and contains typically 3 to 15 at.%

of oxygen. The chemical composition of the film varies somewhat from run to run. No correlation between the oxygen content and the silane flow rate was observed.

It is generally believed that the oxygen content in the deposited films can be reduced by maintaining a nitrogen discharge in the system prior to deposition. A series of runs were made to investigate the effectiveness of this nitrogen "preburn." The basic process consisted of cleaning the system prior to deposition and then predepositing silicon oxynitride for 60 to 90 min. The samples were then baked out at 350°C under vacuum for 30 to 60 min, and a nitrogen "preburn" for 5 min was performed. The rf power was then switched off and the silane flow begun and stabilized. The rf power was then switched on and the film deposited for 10 min. Films were also deposited without the prebake, preburn cycle. The electrical evaluation of these samples is illustrated in Table 1. The samples were implanted with Si^+ at 100 keV to a dose of 1×10^{13} cm^{-2} and were annealed at 800°C for 30 min.

Table 1. Comparison of PED Films

Deposition Condition	Sheet Resistivity, Ω/□	Mobility, cm^2 V^{-1} sec^{-1}	Sheet Carrier Concentration, cm^{-2}
Clean, predeposit, prebake, preburn	2600 ± 1100	1720 ± 280	$(1.57 \pm 0.57) \times 10^{12}$
Clean, predeposit, no preburn	1460 ± 530	1910 ± 230	$(2.39 \pm 0.51) \times 10^{12}$
No preburn	1530 ± 230	2003 ± 77	$(2.07 \pm 0.27) \times 10^{12}$
No preburn	a	a	a
Clean, predeposit the day before, prebake, no preburn	1580 ± 240	2410 ± 180	$(0.66 \pm 0.12) \times 10^{12}$

[a]No activation

6424

Since the data show no consistent trend, it is not clear whether the "preburn" step is really effective. As mentioned before, RBS and Auger analysis show 3 to 15 at.% of oxygen in the films. No dependence of oxygen content on deposition parameters has been observed. Recently, B.G. Streetman, K.V. Vaidyanathan, and co-workers at the University of Illinois have shown that the oxygen content in the films can be reduced to less than 1 at.% by using a high vacuum system with a diffusion pump and preburning with nitrogen.[1] A similar system capable of depositing uniform films over four 2-in. wafers simultaneously is being designed under Hughes internal funding. The system will include a shutter to ensure that the sample surface remains covered until the actual deposition takes place. All the parts necessary for constructing the system have been ordered.

SECTION 3

Si TRANSFER TEST

In all, four tests of the transferability and reproducibility of ion-implantation processes using GaAs substrates have been carried out by HRL and NRL. The results of the first three transfer tests, involving Be, S, and Se, have been described in detail in previous progress reports[2,3] and will not be repeated here.

During this reporting period, the fourth transfer test, "Si 1," was performed. This test represents the first transfer test performed using encapsulant films prepared independently at each facility. Both HRL and NRL used plasma nitride as their principal encapsulant. Seven samples were prepared at NRL. Thirty samples, including 10 unimplanted control samples, were prepared at HRL. In addition, 12 samples, including 4 control samples, were prepared at HRL using each of the three alternative encapsulants: pyrolytic SiO_2 prepared by the "silox" process (silane plus oxygen) at $400^{\circ}C$, pyrolytic Si_3N_4 prepared at $700^{\circ}C$, and pyrolytic SiO_2 prepared by the "Sperry-Rand" process (silane plus nitrous oxide) at $600^{\circ}C$.

The substrate material was Cr-doped, semi-insulating GaAs from Crystal Specialties (ingot 2988). The samples were in the form of parallelpiped dice roughly 7 mm x 7 mm x 0.5 mm. All samples were provided with implanted corner contacts using a 5×10^{13} cm^{-2} 100 keV silicon implant. All samples except the controls were given an overall implant of $10^{13}/cm^2$ 150 keV $^{29}Si^+$ using SiF_4 fuel. The implantations were performed at room temperature and "random" incidence. The mass-29 isotope of Si was used to prevent possible contamination of the ion beam with $^{28}N_2^+$. (A mass-29 nitrogen molecule $^{14}N^{15}N$ exists but is only 0.8% of the total nitrogen concentration and, therefore, is not expected to contaminate the Si beam significantly.) All samples were annealed at $850^{\circ}C$ for 30 min in flowing forming gas. This anneal cycle is considerably more severe than the $800^{\circ}C$ anneal that gave good results in sulfur test S 1. It is, however, more in keeping with current GaAs device technology. Annealing in the 850 to $860^{\circ}C$ range generally yields better

electrical activity and doping profile than 800°C annealing if the encapsulant film is reliable.

Only the NRL samples have been measured at both facilities. The electrical results for these samples are presented in Table 2. One sample, number 462, yielded measurements inconsistent with the others and was not delivered to HRL. Electrical measurements performed on the remaining six samples at the two facilities are in good agreement. These results appear to indicate that very good electrical properties have been achieved at NRL: 81% electrical activity, good values of mobility, and standard deviations of mobility and sheet resistivity of only 8% of the mean value.

Table 2. Si Transfer Test Si 1
(NRL Samples, Plasma Nitride Cap, Measurements at NRL and HRL)

Sample Number	Sheet Resistivity, Ω/□		Hall Electron Mobility, $cm^2/Vsec$		Resistance Ratio
	NRL	HRL	NRL	HRL	
455	271	267	3060	3100	2.1
462	310	-	2310	-	-
466	250	265	3110	2970	1.4
468	229	237	3400	3300	3.2
715	243	245	3600	3620	1.0
743	219	221	3230	3220	3.0
747	229	223	2930	3070	1.7
Mean and Standard Deviation					
7 samples	250 ± 31		3090 ± 410		
6 samples	240 ± 19	243 ± 20	3220 ± 250	3210 ± 230	
81% electrical activity.					

6424

The HRL samples with plasma nitride encapsulation were prepared in two batches of 15. Nominally identical deposition parameters were used for each batch. The results for the first batch are presented in Table 3. Electrical results for 9 of the 10 test samples (eliminating one sample that apparently had not been implanted or had been implanted on the wrong side) differ significantly from those of the NRL samples. These samples exhibit 57% apparent electrical activity and higher sheet resistivity than the NRL samples. The mobility values of the HRL "first batch" samples are statistically indistinguishable from those of the NRL samples. The control samples from the first batch remained semi-insulating.

The second batch of samples prepared at HRL using plasma nitride films gave quite different results. Of the 10 test samples, 2 exhibited resistance assymmetries far from those expected on the basis of the geometric features of the dice and 1 exhibited excessive drift during the measurements. Electrical measurements on the remaining 7 samples, shown in Table 4, gave results comparable to those of the NRL samples: 79% apparent electrical activity and statistically equivalent values of sheet resistivity and mobility. In this case, however, the control samples failed to remain semi-insulating. Thus, it is likely that the high electrical activity and low sheet resistivity of the second batch of samples probably resulted in part from carriers not produced by activation of the implanted Si ions. Since no control samples were run at NRL, there is no way to know whether the NRL results are partially influenced by substrate conversion during annealing.

Of the three batches of samples prepared at HRL using the alternative dielectric materials, only the samples provided with $600^{\circ}C$ SiO_2 encapsulant films gave interpretable electrical results. Samples coated with both $400^{\circ}C$ silox and $700^{\circ}C$ Si_3N_4 gave erratic results. This is the first time in at least a year that the silox encapsulant has failed. Failure of the $700^{\circ}C$ nitride was not surprising since we have had only limited experience with the $700^{\circ}C$ deposition process.

Table 3. Si Transfer Test Si 1

(HRL Samples (First Batch), Plasma Nitride Cap, HRL Measurements)

Sample Number	Sheet Resistivity, Ω/□	Hall Electron Mobility, $cm^2/Vsec$	Resistance Ratio
Test samples[a]			
552	326	2960	1.6
553	347	3460	1.0
554	342	3350	2.0
555	282	3760	1.2
556	428	2640	1.2
558	317	3480	1.1
559	311	3360	1.7
560	398	3020	1.3
561	301	3330	1.5
Mean and Standard Deviation	339 ± 47	3260 ± 330	
Control Samples			
603	1.3×10^6	460	2.6
604	1.3×10^5	480	2.6
605[b]	4.6×10^9	240	1.3
606[c]	1.1×10^7	8	1.1
607[b]	3.8×10^9	360	3.5

[a] 57% electrical activity.

[b] Current source saturated.

[c] Much drift.

6424

Table 4. Si Transfer Test Si 1

(HRL Samples (Second Batch), Plasma Nitride Cap HRL Measurements)

Sample Number	Sheet Resistivity, Ω/□	Hall Electron Mobility, $cm^2/Vsec$	Resistance Ratio
Test Samples[a]			
564	224	3320	1.0
565	207	3630	1.0
567	249	3510	1.4
570	252	3190	1.0
571	230	3190	1.1
572[b]	209	3490	4.4
573	273	3560	1.6
Mean and Standard Deviation	235 ± 24	3410 ± 180	
Control Samples			
611	346	3920	1.1
612	388	3650	1.1
613	483	4340	1.6
614	349	3770	3.3
Mean and Standard Deviation	392 ± 64	3920 ± 300	

[a] 79% electrical activity.

[b] Sample not square (broken).

6424

The results from the samples given 600°C SiO_2 caps are presented in Table 5. Although the mobility values of these samples are quite good, the sheet resistance and apparent electrical activity of the samples are poor. Since our earlier work showed that 600°C SiO_2 is quite a good barrier to gallium out-diffusion, we doubt that surface decomposition is responsible for the poor electrical results achieved. A more likely explanation is that the mechanical properties of the encapsulant inhibit the complete annealing of implant-induced lattice damage. Alternatively, this encapsulant may introduce a degree of damage into the crystal during annealing. The 600°C SiO_2 is denser and apparently more "vitreous" (less plastic) than 400°C silox SiO_2 and may suffer some of the mechanical problems encountered with our 600°C Si_3N_4 films.

Following these evaluations, a second attempt at achieving transferability of Si implantation was attempted using pyrolytic SiO_2 (silox) encapsulants. A set of 30 dice were sent to code 5212, NRL, for implantation and annealing. A set of 19 samples were implanted at HRL, encapsulated with 2500 Å of silox, and annealed at 850°C for 30 min. The results are summarized in Table 6. These data clearly show that the silox oxide provides an extremely reproducible anneal cap. The samples from Code 5212 have not been evaluated.

Table 5. Si Transfer Test Si 1

(HRL Samples, $600^{\circ}C$ SiO_2 Cap)

Sample Number	Sheet Resistivity, Ω/□	Hall Electron Mobility, $cm^2/Vsec$	Resistance Ratio
Test Samples[a]			
524	804	3550	1.7
525	815	3230	2.1
526	677	3790	1.4
527	1870	2700	1.3
528	1110	3470	2.0
529	896	3550	1.2
530	1130	2440	1.0
531	1190	3970	1.1
Mean and Standard Deviation	1060 ± 370	3340 ± 530	
Control Samples			
590[b]	1.1×10^7	4	622
591[b]	1.3×10^7	16	10
592[b]	1.2×10^7	- 3	12
593[b]	3.6×10^6	-28	10

[a]19% electrical activity.

[b]Much drift.

6424

Table 6. Si Transfer Test

(HRL Samples, 850°C Anneal, Silox Cap)

Sample Number	Sheet Resistivity, Ω/□	Hall Electron Mobility, $cm^2/Vsec$
830	273	3208
831	325	3001
832	316	3259
833	316	3014
834	317	3289
835	332	2977
836	292	3241
838	272	3232
839	357	3077
840	334	3091
841	330	3236
842	283	3228
843	305	3207
844	285	3178
845	280	3196
846	288	3210
847	373	2490
848	324	3204
849	315	3239
Mean and Standard Deviation (18 Samples)[a]	311 28	3135 182
Electrical Activity (65 ± 4)%		

[a]The data from sample 847 were deleted because they were excessively far from the mean.

6424

SECTION 4

CHANNELED Be IMPLANTS

Hall/stripping measurements performed by Code 5212, NRL, on Be-implanted GaAs samples have indicated that channeling effects may be responsible for some of the deviations of the electrical profile of the samples from that predicted for "random-equivalent" implants of Be into GaAs. Accordingly, Code 5212, NRL, requested that we prepare a set of GaAs samples from a well-characterized Cr-doped ingot implanted with the beam incident parallel to a <110> axis of the crystal. (The <110> axis is the optimal channeling axis for zincblende-type lattices.) The experiment would have been quite straightforward if well-characterized (110) wafers had been available. Unfortunately, our supply of reliable Cr-doped substrates consisted entirely of (100) wafers.

On a (100) GaAs wafer, the surface normal is nominally a [100] axis. There are four [110] axes that project out from this surface at a 45° angle to the surface normal: [110], [101], $[\bar{1}10]$, and $[10\bar{1}]$. There are also four <110> axes that are at 90° to the surface normal: $[0\bar{1}1]$, $[0\bar{1}\bar{1}]$, $[01\bar{1}]$, and [011]. The last four axes are the surface normals to the {011} cleavage planes that form the edge faces of a typical cleaved die used for implant studies in this contract. If such a die were perfectly square, the plane which contains both the [100] surface normal to the wafer and any one of the <110> axes that project at 45° to the surface normal would pass through two corners of the surface of the die.

To produce a channeled implant, it is necessary to position the sample so that a single crystal axis is collinear with the impinging ion beam. Thus, to perform a <110> channeled implant into a (100) GaAs wafer, the wafer must be oriented at a 45° angle relative to the beam and must be rotated so that one of the four <110> channeling axes is collinear with the beam.

The ion channeling system at HRL is equipped with a sample goniometer that permits aligning sample axes to within 0.02° of the direction of the ion beam. Unfortunately, this goniometer can tilt the sample a maximum of 35°. Thus, to perform a <110> implant into a (100) wafer, an

additional tilt must be supplied by mounting the sample on a wedge. Accordingly, we prepared a wedge with a 45° apex angle to tilt the sample at 45° to the ion beam when no tilt was indicated on the goniometer controls.

To properly rotate the samples, the samples were provided with cleaved edges. A well-cleaved sample has quite straight edges. Scribed lines were provided on the mounting wedge to help in aligning the sample. These lines formed a 90° angle that was bisected by a line running directly up the slope of the wedge. By positioning two edges of the sample accurately along a pair of these guidelines, the sample was properly rotated so that alignment of the crystal could be performed using our standard alignment procedure.

The actual implant was performed after the sample had been aligned to within 0.02^{o} of a <110> axis using proton backscattering. In our channeling system, the deflection plates that raster-scan the ion beam are located roughly 3 m from the sample. Using this deflection system, a 1 cm x 1 cm area normal to the beam can be scanned while beam alignment is maintained within $\pm 0.12^{o}$ of a channeling axis. With the sample mounted on the 45° wedge, the actual implanted area is 1 cm x 1.4 cm, and the ion fluence measured in cm^{-2} must be reduced by a factor of 1.4 to account for the oblique incidence of the beam.

Using these techniques, 4 samples from Crystal Specialties ingot 2988 were implanted with 10^{13} cm^{-2} 150 keV Be^{+} and were delivered to Code 5212, NRL, for evaluation. The penetration of this implant will be roughly equivalent to that of a 110 keV implant into a (110) wafer because of the foreshortening that results from the oblique incidence of the implant. More channeled Be implants are presently being performed for delivery to Code 5212, NRL, for evaluation.

SECTION 5

DIAGNOSTIC IMPROVEMENTS

Hall-effect analysis is the primary technique being used for electrical evaluation of the implanted samples for this program. For this purpose, we use a computer-controlled Hall-effect system that controls all the variables of the Hall effect and sheet resistivity measurements except for the absolute value of the magnetic field. All lead switching and magnetic field reversals are performed under computer control. With this system, roughly 80 independent measurements are performed within 6 to 10 min to establish the sheet resistivity and Hall coefficient of a single sample. Very complete information concerning the electrical asymmetry of the sample and the drift of the measured values are provided on the computer output.

More complete diagnostics of the properties of the implanted layers are clearly needed in view of the significant improvements in electrical activation of implanted layers recently achieved at HRL and NRL using plasma nitrides. (Section 3 describes the latest results.) In particular, electrical profile measurements will be very important in determining if redistribution of the implanted species is occurring (or if the electrical and atomic profiles differ). Hall/stripping measurements are one method of doing this, but they are very time-consuming and require great care if an accurate depth scale is to be maintained. Capacitance-voltage (C-V) analysis is less time-consuming in the measurement stage but generally requires lightly doped epitaxial material on heavily doped substrates to be used if really critical profile analysis is to be performed.

HRL has a commercial analog C-V profiling system that is used for routine diagnostic work. Units of this type generally are not particularly suitable for critical work on rapidly varying profiles (such as Gaussian implants) because the test voltages applied to determine dC/dV may degrade the resolution. (Ref. 4 discusses the problem of determining dC/dV in an analog system.) A digital data-acquisition system has been acquired for determining impurity profiles by computer-controlled C-V

measurements. Using this system, low test levels (10 mV rms) will be applied at all times, and the computer will control the bias voltage steps applied to ensure that optimal depth resolution is maintained.

In addition, we are presently establishing the capability to perform deep level transient spectroscopy (DLTS) and low-temperature photoluminescence (PL). The combined use of DLTS and PL will provide a very powerful capability for identifying impurity energy levels over a large fraction of the bandgap region. DLTS can provide quantitative information on impurity energy level, capture cross-section, and concentration. PL provides quantitative energy level information and useful concentration information. Attempts to quantify PL concentration measurements are being made at several establishments. PL intensities provide a very useful measure of the degree of lattice disorder in implanted regions by providing a direct indication of the relative radiative lifetime. The use of DLTS and PL on this program in the near future should provide very useful information concerning the defect levels introduced during ion implantation and annealing procedures. This information will then provide a basis for developing optimized techniques that will permit controlling the defect-generation processes.

Finally, a Cambridge Stereoscan S-150 SEM was recently installed in our laboratory. This unit will be used extensively for studies of the topography of the GaAs surface after annealing. The information gained will be used to fine-tune our encapsulation procedures to ensure that a minimum of texturing of the GaAs surface occurs during implant annealing.

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