	1b. RESTRICTIVE	PAGE		
		MARKINGS		
	3 DISTRIBUTION	I AVAILABILITY	OF REPOR	<del>ر</del>
	Approve	d for publi	.c rele	ease;
7 0 I 1900	distrib	ution unlim	ited.	
	AR	0 25534.1-E	L-SBI	AOMBEK(2)
65 OFFICE SYMBOL	a. NAME OF M	ONITORING ORG	ANIZATIO	N .
(If applicable)	U. S. /	Army Resear	ch Off	ice
	7b. ADDRESS (Ci	ty, State, and Zil	P Code)	
	P. 0. 1	Box 12211		
	Researd	ch Triangle	Park,	NC 27709-2211
8b. OFFICE SYMBOL	9. PROCUREMEN	T INSTRUMENT I	DENTIFICA	ATION NUMBER
	DAALO3-87	′-C-0024		
	10. SOURCE OF	FUNDING NUMBE	RS	······································
709-2211	PROGRAM ELEMENT NO	PROJECT NO:	NO.	WORK UNIT
1/87 TO 3/31/88	6/6/88	ontained in	thic	report are these
be construed a	is an officia	al Department	nt of	the Army position
18. SUBJECT TERMS (	Continue on revers	e if necessary ar	nd identif	y by block number)
	number)	····		
ase I program,	Microwave Mc	nolithics I	Incorpo	orated (MMInc.)
a new low tempe	rature activ	vation metho	d for	ion implanted
takan by MMInc	t processing	g. The work	c perfe	rmed during
taken by MMInc. tegrated circui	- F	•		Jimed during
taken by MMInc. tegrated circui efect restricti	on to re-cry	stallation	of the	e amorphous
taken by MMInc. tegrated circui efect restricti the application step. Althoug	on to re-cry of this app h this topic	vstallation proach in Ga has high r	of the As app isk it	e amorphous pears to occur t has extremely
taken by MMInc. tegrated circui efect restricti the application step. Althoug essing technolo	on to re-cry of this app h this topic gy needed fo	vstallation proach in Ga has high r pr high freq	of the As app isk in quency	e amorphous pears to occur t has extremely mm-wave devices
taken by MMInc. tegrated circui efect restricti the application step. Althoug essing technolo	on to re-cry of this app h this topic gy needed fo	vstallation proach in Ga has high r pr high free	of the AAs app isk in quency	e amorphous pears to occur t has extremely mm-wave devices
taken by MMInc. tegrated circui efect restricti the application step. Althoug essing technolo	on to re-cry of this app h this topic gy needed fo	stallation broach in Ga has high r br high free	of the As app risk it quency	e amorphous pears to occur t has extremely mm-wave devices
taken by MMInc. tegrated circui efect restricti the application step. Althoug essing technolo	on to re-cry of this app h this topic ogy needed fo	vstallation proach in Ga has high r pr high free	of the As app risk in quency	e amorphous pears to occur t has extremely mm-wave devices
taken by MMInc. tegrated circui efect restricti the application step. Althoug essing technolo	on to re-cry of this app h this topic gy needed fo	ecurity classifi	of the AS app risk in quency	e amorphous pears to occur t has extremely mm-wave devices
taken by MMInc. tegrated circui efect restricti the application step. Althoug essing technolo	on to re-cry of this app h this topic gy needed for 21. ABSTRACT SI UT 225. TELEPHONE	stallation proach in Ga has high r or high free CURITY CLASSIFI classified (Include Area Coc	of the aAs app isk it quency (CATION	office Symbol
	6b OFFICE SYMBOL (If applicable) 8b. OFFICE SYMBOL (If applicable) 709-2211 709-2211 10n Implanted 1/87 TO 3/31/88 opinions and/or be construed a designated by construed by construed a designated by construed by c	AR         6b OfFICE SYMBOL (If applicable)       Ja. NAME OF M U. S. J U. S. J D. ADDRESS (CI P. 0. 1 Researd J. ADDRESS (CI P. 0. 1 Researd DAAL03-87         8b. OFFICE SYMBOL (If applicable)       9. PROCUREMEN DAAL03-87         709-2211       10. SOURCE OF PROGRAM ELEMENT NO         709-2211       14. DATE OF REPO 6/6/88         Opinions and/or findings co be construed as an officia designated by other document 18. SUBJECT TERMS (Continue on reverse and identify by block number) ase I program, Microwave Mc	ARO 25534.1-E ARO 25 ARO	ARO 25534.1-EL-SBI ARO 25534.1-EL-SBI ARO 25534.1-EL-SBI ARO 25534.1-EL-SBI Ja. NAME OF MONITORING ORGANIZATION U. S. Army Research Off 7b. ADDRESS (City, State, and ZIP Code) P. O. Box 12211 Research Triangle Park, 8b. OFFICE SYMBOL (If applicable) 9. PROCUREMENT INSTRUMENT IDENTIFICA (If applicable) 9. PROCUREMENT INSTRUMENT IDENTIFICA 10. SOURCE OF FUNDING NUMBERS PROGRAM ELEMENT NO 10. TASK NO. 709-2211 10. Implanted Compound Semiconductors ACC State of the semiconductors 14. DATE OF REPORT (Year, Month, Day) 1/87 TO 3/31/88 14. DATE OF REPORT (Year, Month, Day) 1/87 TO 3/31/88 14. DATE OF REPORT (Year, Month, Day) 1/87 TO 3/31/88 14. DATE OF REPORT (Year, Month, Day) 1/87 TO 3/31/88 15. SUBJECT TERMS (Continue on reverse if mecessary and identify 18. SUBJECT TERMS (Continue on reverse if mecessary and identify ase I program, Microwave Monolithics Incorpored

# TABLE OF CONTENTS

	List of Figures	
1.0)	Introduction	1
2.0)	Low Temperature Activation of Compound Semiconductors	2
	2.1) Basic Concept	2
	2.2) Technical Approach	4
	2.3) GaAs Amorphization	5
	2.4) Dopant Implantation	5
	2.5) Low Temperature Regrowth	6
	2.6) Characterization of Amorphized Layers	8
	2.7) Electrical Activity	10
3.0)	Program Phase I Technical Progress	11
	3.1) Modified Approach	11
	3.2) Experimental Procedures - Wafer Preparation	14
	3.3) Si <sup>+</sup> Ion Implantation	14
	3.4) Low Temperature Amorphization	16
	3.5) Low Temperature Annealing	18
	3.6) Electrical Characterization of Low Temperature	22
	Regrowth	
	3.7) Characterization of Amorphized Layers	23
4.0)	Summary and Recommendations	25
5.0)	References	32

Accusion Fur	
NTIS CRAS	2
DT'S TAB	
le contration	1 G
	1
	ļ
	1, Pages
·- ·	· · · ·
H-/	

88 1028 DI4

i

(mat

# List of Figures

2.1-1)	Maximum Strain as a Function of Dose	3
2.6-1)	Damage Depth Profile by Ellipsometry	9
3.1-1)	Projected Amorphization Profile with 50KeV Si <sup>+</sup> Distribution	12
3.1-2)	Projected Amorphization Profile with 120KeV Si <sup>+</sup>	13
	Distribution	
3.4-1)	Activated <sup>29</sup> Si <sup>+</sup> Profiles for 50KeV and 120KeV Implants	15
3.5-1)	Sample Holder for Low Temperature Bombardment	17
3.5-2)	Hot Plate Anneal Apparatus	19
3.5-3)	Flash Anneal Apparatus	21
3.7-1)	Damage Depth Profile for 50KeV Si <sup>+</sup> /1MeV Ar <sup>+</sup>	24
3.7-2)	Electron Diffraction of Regrown 500KeV Ar <sup>+</sup> Samples	26
3.7-3)	Electron Diffraction of Regrown 1MeV Ar <sup>+</sup> Samples	27
3.7-4)	Rutherford Backscattering Profile - 500KeV Ar <sup>+</sup>	28
3.7-5)	Rutherford Backscattering Profile - 1MeV Ar <sup>+</sup>	29

#### 1.0) Introduction

Ion implantation is the only materials technology that permits the high degree of control necessary for cost effective production of advanced GaAs integrated circuits. Presently the only realistic method of removing the ion implantation damages in III-V compounds is by high temperature annealing. This high temperature method, is restrictive to certain device profiles and fabrication techniques, but even more serious it is detrimental to many advanced materials such as HEMT and Ge/GaAs heterojunctions. These advanced structures are severely damaged by the high temperature annealing methods and many possible device and integrated circuits cannot be fabricated as a result of these limitations.

In this program a new concept of annealing bombardment damage is undertaken that has promise of performing removal of ion bombardment damage at extraordinary low temperatures. The achievement of conditions needed to accomplish this amorphization/anneal method are complex and require detailed study of the separate variables. Although the risk of this project is high the benefits to SDIO are enormous. The application of this technology to III-V integrated circuit processing would revolutionize selective ion implantation for microwave, digital and opto-electronic circuits. Mm-wave IC's which are bound to be the most difficult to achieve would also benefit.

During phase I research on this low temperature activation of ion implanted compound semiconductors Microwave Monolithics adopted an approach that took advantage of prior work on GaAs and circumvented many of the earlier difficulties shown to be a problem with solid phase epitaxy in III-V semiconductors. The basic principles of this approach are based upon the successful work of solid phase epitaxy in silicon that has been developed to aid fabrication of advanced integrated circuits. Our initial work has identified a problem in the complex amorphization step that influences the successful application of this

method. The solution to the problem of defect generation in GaAs amorphization will be resolved in future fundamental studies where greater attention can be spent with basic regrowth mechanisms as the potential of this powerful technique has yet to be exploited with III-V semiconductors.

### 2.0) Background Discussion

### 2.1) Basic Concepts

Low temperature activation or solid phase epitaxy has been studied in silicon and reduced to practice in recent years for use in controlling abrupt distributions of diffusion prone impurities. Amorphons layer generation in silicon and germanium is feasible due to a favorable strain-dose relationship as shown in Figure 2.1-1. As the amorphizing species  $(Si^{\dagger})$  is implanted into the Si lattice the crystal undergoes a linear strain relationship with increased dose all the way to a point where a coalescence of point defects occurs producing an amorphous crystalline state whose depth depends upon the energy of the bombarding species and the threshold for amorphization on the dose in ions  $cm^{-2}$ . Following amorphization a dopant species may be implanted within the amorphous region in the same manner as conventional ion implantation. Upon annealing the amorphized layer it has been demonstrated that recrystalization will occur at approximately one half the temperature for conventional implant damaged layers. Electrical activation of low temperature annealed Si layers (550°C) is equivalent to that obtained at the more conventional  $(1100^{\circ}C)$  anneal temperature.

Substantial improvements have been obtained in control of doping distributions of rapid diffusing species using the pre-amorphized approach to ion implantation of Boron in Si.<sup>(1)</sup>Channeling, a common problem in the generation of abrupt profiles is also minimized by the use of a pre-amorphized substrate.



V.S. Speriousu et. al. Appl. Phy. Lett. 40, (7), (1982)



For GaAs, an amorphized low temperature regrowth method should greatly improve the processing of selective ion implantation for integrated circuit devices. At the present time a dielectric cap is used by most workers to offer surface protection and retard group V evaporation during the high temperature furnace anneal cycles. The diffusion of donor and especially acceptor impurities is very prominent in GaAs under the furnace anneal conditions. Impurities such as high dose Be, Mg and especially Zn experience high diffusion and broadening effects at high temperature making many device applications marginal to not feasible. Our interest in developing a low temperature activation technique for compound semiconductors has far reaching implications to making ion implantation feasible for temperature sensitive materials such as HEMT and multiple HEMT as well as strained layer materials that would not survive the normal post implant anneal cycle.

Other applications involve opto-electronic devices of ternary and guarternary III-V compounds grown and lattice matched to InP. The normal Zn and Cd acceptors diffuse very rapidly and have very limited annealing conditions for conventional ion implant processing.

### 2.2) <u>Technical Approach</u>

A similar general approach was taken for GaAs as was performed in silicon. The amorphizing implantation is done first at 77K and the dopant implantation is performed in situ with substrate temperatures held at 77K until all implants are complete, in this manner the heating effects from dopant bombardment are eliminated and no uncontrolled dynamic annealing should take place. For true epitaxy to take place, layer by layer, growth from the amorphous/crystalline interface should proceed upon heating to some critical temperature. In the section to follow a description of III-V amorphization, dopant implantation, and low temperature regrowth will be described along with characterization methods for measurement of thin crystalline layers near the surface. Electrical activity will also be addressed.

### 2.3) III-V Amorphization

In GaAs the maximum strain as a function of dose is complex and occurs between  $10^{13}$  and  $10^{14}$  cm<sup>-2</sup>. The yielding effects of extended defects and stacking faults appear to be an irreversible transition occuring with room temperature amorphization. The intent of this program will be to perform an amorphizaton at 77K for purposes of eliminating generation of micronuclei that would later propagate during re-crystallization into faults and microtwins.

Defects propagating upon re-crystallization can lead to compensation of the doped region. It is clear from the work of Sadana et al<sup>(2)</sup> that doses in the range of  $10^{15}$  cm<sup>-2</sup> are required to produce full amorphization in GaAs. It is not clear whether displacement effects will occur to interfere with an ordered layer by layer regrowth performed at low temperature. If As<sup>+</sup> is a bombarding species there is also possibility of precipitation effects in the lattice to produce nucleation centers for defects growth upon recrystallation. The compound region in the GaAs phase diagram becomes quite narrow at lower temperatures and solubility restraints could be a problem where self diffusion is also limited. The case for elemental semiconductors is most ideal where displacement effects are not detrimental and only solubility limits the extent of doping in the pre-amorphized region. <sup>(3)</sup>

# 2.4) Dopant Implantation

Introduction of a dopant species in GaAs may be performed following amorphization in situ at 77K, or before amorphization. The important self annealing problem should be avoided by holding the substrate always at 77K. Knock-on distributions have not proven to be a difficulty in GaAs for post-amorphization approaches. The desired dopant species for this work is  $^{29}$ Si<sup>+</sup>. The pre-amorphization damage could be broken up by a post-amorphization which could provide the final assurance for full amorphization. In any case the entire amorphization stage carries

limited information until annealing is achieved and then the data contains several variables.

The introduction of dopant into a pre-amorphized region has been pursued in GaAs as was performed in Si and Ge. The major difficulty in the dopant implant is the dynamic annealing effects that occur with room temperature implants. Later regrowth of the partially re-crystallized region leads to propagation of defects that produce compensating effects upon the activation of implanted carriers. For amorphization in silicon control of substrate temperature resolved the problem of dynamic annealing and low carrier activation. In GaAs problems related to dynamic annealing are present for all room temperature implants. In addition the low temperature amorphized layers in GaAs all tend to regrow with major defects. Re-crystallation of low temperature amorphized GaAs proceeds at low temperature, but not without propagation of stacking faults and extended defects.<sup>(2)</sup> Before the dopant implants can be activated the problem of severe defect generation needs to be resolved.

### 2.5) Low Temperature Regrowth

Si amorphous layers regrow on a hypothetical layer by layer basis and leave little trace of defect regions. The exception of course is solubility limitations and non-ideal a/c interfaces. Small clusters (>15Å) of point defects can propage to form small embryonic dislocation loops. Interstitial Si atoms can also coalesce to form loops. <sup>(3)</sup> In GaAs several other problems are possible:

- 1) Displacement effects
- 2) Stoichiometry related effects
- 3) Solubility limit effects
- 4) Interstitial problems
- 5) Ordering effects
- 6) Thermal activation threshold

<u>Displacement</u> effects from bombardment of a layered III-V semiconductor could result in long range disorder making crystalline reconstruction difficult and prone to effects of interstitial and antisite defect generation. The effects of displacement are very difficult to assess as the amorphized layer is not prone to characterization methods that will yield analytical data. Post regrowth analysis revealing interstitial and antisite defects need to be corollated with other factors such as stoichiometry and solubility.

<u>Stoichiometry</u> effects from atomic displacement could produce compensation or other dopant activation anomalies. Also precipitation of As would invoke a stoichiometry imbalance if excess As could not escape during the re-crystallation step.

<u>Solubility limit</u> problems in GaAs are normally not experienced due to the requirements for doping in existing device structures. As<sup>+</sup> bombardment could exceed solubility limits for the compound and as such could contribute to stoichiometry, antisite defects or precipitation problems leading to crystalline regrowth defects.

<u>Interstitial</u> effects can be produced by solubility limit difficulties as in the case of high  $As^+$  doses that remain in the lattice following annealing. Ordering effects experienced with lattice defects disturbing the A-B structure in GaAs can also produce interstitial defects of excess Ga or As.

<u>Ordering</u> effects as mentioned earlier can be the result of lattice defects (point defects or extended defects) due to the disruption of continuous growth. Interstitial atoms  $(As^+)$  cculd also induce ordering effects by straining the lattice. Precipation due to  $As^+$  clustering and solubility limit problems can also incite non-continuous crystal growth.

<u>Thermal Activation</u> threshold requirements are expected to be different to crystalline regrowth conditions. The lattice mobility necessary for high dopant activity has been demonstrated in non-

amorphized GaAs to be achieved at a higher temperature than that for recrystallation.

# 2.6) Characterization of Amorphized Layers

A

Measurement of amorphized layers can be accomplished before recrystallation to document amorphized depth and lack of crystallinity. Re-measurement following regrowth can be referrenced to show remanent damage effects. The following techniques have been used successfully to characterize both Si and GaAs amorphous layers.

Diffraction	1) <u>HRTEM</u> :	Images crystalline order and gives
Techniques:		excellent distinction of relative
		crystalline perfection

- <u>SEM</u>: Angular diffracted patterns can produce a qualitative indication of crystallinity on a low sensitivity scale.
- Scattering 1) <u>RBS:</u> Rutherford back scattered protons or Techniques: He<sup>+</sup> ions are used to measure thick layers penetrated by MeV accelerated ions. A well disordered layer will show major differences between one that has good crystal perfection. No arguments about crystal quality can be made, only relative comparisons.
  - 2) <u>Ellipsometry:</u> The extinction coefficient is extremely sensitive to damage effects and is useful as a profiling method when used in conjunction with a thinning etch. Figure 2.6-1 shows a damage profile produced with Si<sup>+</sup>, 320KeV, 6E12cm<sup>-2</sup> before and





after annealing. Even following recrystallization that produced >80% electrical activation the residual damage level is discernible.

- <u>IR Reflectance</u>: Infrared reflectance techniques can also be used to detect amorphous layer depth using optical interference methods.
- <u>Differential</u> Etch delineated bevel or groove <u>Etch Methods</u>: measurements can be made with a profilameter.
- 5) <u>Optical</u> Visable reflectance shows a <u>Refelctivity:</u> contrast change in the surface reflectance of damaged GaAs. An "as implanted" GaAs surface appears lighter than as polished surface. Heavily bombarded GaAs surfaces (500KeV, 1E15cm<sup>-2</sup>) look light yellow in appearance. The regrown or annealed surfaces return to original appearance of a polished wafer.

### 2.7) <u>Electrical Activity</u>

Electrical measurement of dopant activity in regrown GaAs layers can be performed with the customary tools for thin layer measurement. Electrical assessment using resistivity and Hall measurement are performed. The measured distribution of dopants by profiling is essential to show the corollation with LSS projections. Trap measurements can be used to identify deep damage centers effecting full carrier activation. Additional corollation with chemical distributions can be obtained with Secondary Ion Mass Spectrometry (SIMS) for Si implanted layers, where projected depth is  $>1000\text{\AA}$ .

3.0) Phase I Research and Results

### 3.1) Modified Approach

In order to obtain an amorphised layer deep enough for typical "device" dopant implants an energy in excess of 400KeV is required. Our earlier approach using commercial 200KeV ion implantation equipment and doubly charged As would give only a  $1500\text{\AA}$  separation between the 120KeV Si<sup>+</sup> implant and the amorphous-crystalline interface. In order to place a greater margin of safety a revised approach was taken where a higher energy source could be used. A research accelerator with MeV capability for inert ions was available through California State University - Los Angeles. In this apparatus  $\text{Ar}^+$  was used to provide both 500KeV and 1MeV at  $1E15\text{cm}^{-2}$  fluences. The calculated damage depth for  $\text{Ar}^+$  at these accelarations are as follows:

500KeV	Ar <sup>†</sup> :	<u>2400Å</u>
1MeV An	c <sup>+</sup> :	4200 <b>X</b>

The bombardment damage should extend in from the surface and not require a multiple energy approach as suggested earlier. The calculated amorphized envelope is shown with the projection for the  $\mathrm{Si}^+$  dopant implant in Figure 3.1-1 and 3.1-2. In order to assure the validity of the dopant implant it was performed first and portions of that sample evaluated by conventional means before the amorphization was performed. In this manner the critical amorphization step was last and would possibly have less complications from dynamic annealing. In the sections to follow a description of the full experimental procedure and results will be shown.



Figure 3.1-1) Projected Amorphization Profile with 50KeV Si<sup>+</sup> Distribution



Figure 3.1-2) Projected Amorphization Profile with 120KeV Si<sup>+</sup> Distribution

### 3.2) Experimental Procedures - Wafer Preparation

High quality semi insulating GaAs substrates were selected for this study that had the following properties:

<u>Resistivity:</u>	>10 <sup>7</sup>
<u>Mobility:</u>	>5000 cm <sup>2</sup> /v-sec
<u>Thermal Stability:</u>	$>10^{7} \Omega/\Box$ following anneal to $950^{\circ}$ C
<u>lon Implantation:</u>	>80% carrier activation with <sup>29</sup> Si <sup>+</sup> and 950°C anneal conditions

# 3.3) <sup>29</sup><sub>51</sub> Ion Implantation</sub>

Two separate  $^{29}$ Si<sup>+</sup> implants were used in this study to evaluate the low temperature amorphization and low temperature regrowth as follows.

1) 50KeV, 1.5E13cm<sup>-2</sup> 2) 120 KeV, 3.0E12cm<sup>-2</sup>

The above implants were performed by implanting  $^{29}$ Si<sup>+</sup> produced from an ionized beam of SiF<sub>4</sub>. The implants were made at room temperature with beam currents of approximately 10uA. To verify the Si<sup>+</sup> implants a small piece was annealed before amorphization by the flash anneal method and the resulting carrier profile evaluated by the C-V method. The carrier profiles from the activated 50KeV and 120KeV implants are shown in Figure 3.4-1.

> Use or disclosure of data contained on this shoat is subject to the restriction on the Mile page



Figure 3.4-1) Activated <sup>29</sup> <sup>+</sup> Profiles for 50KeV and 120KeV Implants

The carrier profiles are shown with LSS projections to show the corollation between the observed and theoretical distributions for  $^{29}$ Si<sup>+</sup>. The profiles are both of good activation and show abrupt profile corollation with the LSS data. The remaining protions of these  $^{29}$ Si<sup>+</sup> implanted slices were taken for low temperature amorphization bombardment with Ar<sup>+</sup>.

## 3.4) Low Temperature Amorphization

Samples implanted with 50KeV  ${}^{29}$ Si<sup>+</sup> and 120KeV  ${}^{29}$ Si<sup>+</sup> were mounted on a specially constructed heat sink for reduced temperature bombardment. A diagram of the holder is shown in Figure 3.5-1. The interior of the substrate holder was machined to allow a liquid  $N_2$  cooling channel for efficient transfer of heat from the GaAs undergoing bombardment. Small Be-Cu clips hold the GaAs securely against the cooled holder. The assembly is inclined  $7^{\circ}$  to the beam to reduce effects of channeling. For bombardment the sample holder is mounted in the end station of a High Voltage Engineering Accelerator and evacuated prior to bombardment. Sample cooling is initiated by flowing  $LN_2$  thru the sample holder held under vacuum. When the sample holder passes liquid  $N_2$  the bombardment is started using an ionized Ar<sup>+</sup> beam. The beam is magnetically raster scanned across the target area. A precision dose integrator is used to collect the charge from the sample. The cooling is maintained during the bombardment period and the beam is terminated by the dose integrator when the desired charge is achieved. Following bombardment the cooling is terminated and the sample is slowly warmed to room temperature by ambient temperature. The sample is removed and replaced with another pre-implanted slice and the cycle is resumed.



COLD FINGER APPARATUS FOR LOW TEMPERATURE BOMBARDMENT



Figure 3.5-1) Sample Holder for Low Temperature Bombardment

The schedules for Ar<sup>+</sup> amorphization are as follows:

<u>Sample</u>	<u>Si<sup>†</sup>Implant</u>	<u>Ar<sup>+</sup>Amorphization</u>	<u>Temperature</u>
2075-020	50KeV, 1.5E13cm <sup>-2</sup>	1MeV, 1E15cm <sup>-2</sup>	77K
2075-012	120KeV, 3.0E12cm <sup>-2</sup>	$^{2}$ 500KeV, 1E15cm <sup>-2</sup>	77K

### 3.5) Low Temperature Annealing

Amorphized samples of both the 50KeV Si<sup>+</sup> implant and the 120KeV Si<sup>+</sup> implant were annealed at temperatures between  $400-950^{\circ}C$  as follows:

1)  $400^{\circ}$ C: Low temperature annealing was performed in reducing atmosphere (Forming Gas) in a face down configuration as shown in Figure 3.5-2. The apparatus as shown utilized a petri dish and a domed cover to introduce the protective atmosphere. A hot plate was used for heating the assembly to temperature. In operation the hot plate was heated to temperature and the pre-purged assembly with wafer was transferred for rapid heating to  $400^{\circ}$ C. Following a 20 minute anneal the assembly was moved off the hot plate and allowed to cool before the sample was removed.

2)  $500^{\circ}$ C: Intermediate temperature annealing was performed in a reducing atmosphere (forming gas) using a flash anneal apparatus as shown in Figure 3.5-3. The heat source in Figure 3.5-3 was a 9 KW bank of quartz halogen lamps controlled by an SCR power supply. The saample was placed face down and purged with forming gas for 15 minutes prior to heating. The control temperature was reached in 2 minutes by slowly ramping the lamp power to the indicated  $500^{\circ}$ C displayed by a chromel-alumel thermocouple positioned near the sample. Following a 20 minute anneal the lamps were turned gradually off and the apparatus cooled to room temperature before removing the sample.

3) <u>600<sup>o</sup>C</u>: The highest gradual anneal was performed at  $600^{\circ}$ C in the flash anneal apparatus described in 2) as shown in Figure 3.5-3. The



Ç

P

Figure 3.5-2) Hot Plate Anneal Apparatus

heating was performed in the same manner as in 2) only heating to  $600^{\circ}$ C for 15 minutes before cooling to ambient temperature.

4)  $950^{\circ}C$ : Samples failing to activate at the lower temperatures were heated in the flash anneal apparatus to  $950^{\circ}C$  as a conventional room temperature implant would be processed. This mode of annealing was used to activate the Si<sup>+</sup> implants shown in Figure 3.5-3 for purposes of verifying the implants prior to Ar<sup>+</sup> bombardment.

The samples taken from annealing 1-4 were inspected by optical microscopy and taken to a C-V apparatus and contacted with a Hg probe for rapid measurement of electrical activation.

Table 3.5-1 shows a summary of the data produced in low temperature annealing experiments.

### Table 3.5-1

### Low Temperature Annealing of Amorphized GaAs

Sample	<u>Anneal Temperature</u>	Anneal Period	<u>Observations</u>
2075-012	400 <sup>0</sup> C	20 minutes	Yellow
2075-012	500 <sup>0</sup> C	20 minutes	Yellow
2075-012	600 <sup>0</sup> C	15 minutes	Grey
2075-012	950 <sup>0</sup> C	8 seconds	Grey



P

ł

ļ



Figure 3.5-3) Flash Anneal Apparatus

# 3.6) <u>Characterization of Low Temperature Regrowth - Electrical</u> <u>Measurements</u>

Following low temperature anneal the  $\text{Si}^+$  implanted-amorphized layers were measured on a C-V apparatus as used for Figure 3.4-1. The C-V measurement system used was a Material Development Corporation Analog profiler. To profile a layer the bias voltage is set to near breakdown voltage of the Hg Schottky barrier under reverse bias (-). The analog system ramps the bias to zero and a log-log plot of N(x) vs x is obtained. The system can also be operated manually at any bias point for point data as well. The verification data on the 50KeV and 120KeV Si<sup>+</sup> implants in Figure 3.4-1 was obtained using the above apparatus.

Data taken from annealed samples following low temperature regrowth are shown in Table 3.6-1.

### Table 3.6-1

### Capacitance Measurements of Low Temperature Regrown Layers

		Anneal		
<u>Sample</u>	<u>Si<sup>†</sup>Implant</u>	<u>Ar<sup>+</sup>Bombardment</u>	<u>Temperature</u>	$\underline{C}_{(0)} \underline{PF/cm}^2$
2075-012	120KeV, 3E12cm <sup>-2</sup>	500KeV,1E15cm <sup>-2</sup>	600 <sup>°</sup> C, 950 <sup>°</sup> C	<0.1pF
2075-020	50KeV,1.5E13cm <sup>-2</sup>	$1 \text{MeV}$ , $1 \text{E} 15 \text{cm}^{-2}$	600 <sup>0</sup> C, 950 <sup>0</sup> C	<0.1pF

Further measurement of samples not indicating a capacitance were made by using Indium contacts in a Van der Pauw configuration. The samples were approximately 7mm on a side and were biased to 25v, far from any critical breakdown fields. The sheet resistance data from these samples is shown in table 3.6-2.

# Table 3.6-2

## Sheet Resistance of Low Temperature Regrown Layers

			<u>Anneal</u>	Sheet
<u>Sample</u>	<u>Si<sup>†</sup>Implant</u>	<u>Ar<sup>+</sup>Bombardment</u>	Temperature	<u>Resistance</u>
2075-030	-0-	-0-	950 <sup>0</sup> C	6.2E9 <b>.0./D</b>
2075-020	50KeV, 1.5E13cm <sup>-2</sup>	1MeV, 1E15cm <sup>-2</sup>	600 <sup>0</sup> C, 950 <sup>0</sup>	1.9E8Ω/□
2075-012	$120 \text{KeV}$ . $3 \text{E} 12 \text{cm}^{-2}$	500KeV, 1E15cm <sup>-2</sup>	600°C, 950°C	4.7E7.0./□

# 3.7) <u>Characterization of Amorphized Layers -</u> <u>Crystalline/Amorphous Measurements</u>

Ł

Following bombardment of the  ${\rm Si}^+$  implanted samples the layers were measured by ellipsometry to determine amorphous layer depth. The extinction coefficient as described in 2.6 is sensitive to lattice damage and disorder.<sup>(4)</sup> Samples of amorphized GaAs were measured before and after annealing with a Rudolph Model EL-III ellipsometer. The 6328Å He-Ne laser line was used for sampling the optical properties of the near surface region. In order to obtain profiles the samples were etched in 500Å steps and remeasured at each step until the interface was reached. Figure 3.7-1 shows a 50KeV Si<sup>+</sup> implant with 1MeV amorphization before and after annealing to 600°C. The fact that the annealed values are only one-half the magnitude of the damaged ones is indicative of the remanent damage remaining in the layer following anneal.

Assessment of crystal quality following low temperature regrowth was made by electron diffraction and RBS measurement.

<u>Electron Diffraction</u>: Samples implanted and bombarded with Ar'were measured by imaging the angular reflected electrons from the top 2000Å nearest the surface with a 20KV electron beam. Samples were





selected from the 50KeV and 120KeV implant that were measured as bombarded, and after annealing to  $600^{\circ}$ C. For reference purposes a sample of undamaged GaAs from the same ingot was included for comparison. Figure 3.7-2 shows the undamaged (a), as damaged (b) and annealed (c) for the 120KeV,  $3.0E12cm^{-2}$  with 500KeV Ar<sup>+</sup> bombardment. Figure 3.7-3 shows a similar set for the 50KeV,  $1.5E13cm^{-2}$  with 1MeV Ar<sup>+</sup> bombardment. The reflected electron diffraction figures in 3.7-2 and 3.7-3 show reasonable diffracted images for the undamaged substrates, but no evidence of reconstruction for either the 50KeV or 120KeV implant. The extent of bombardment damage on the 500KeV and 1MeV samples show no discernable differences between as bombarded and annealed for annealing to  $600^{\circ}$ C for 10 minutes.

<u>Rutherford Back Scattering (RBS):</u> Samples were bombarded with 4MeV He<sup>+</sup> ions and backscattered He ions were measured at 55° for samples "as bombarded" with  $Ar^+$  and those after annealing at 600°C. Figure 3.7-4 shows a RBS plot of count vs channel number for the 120KeV Si<sup>+</sup> implant bombarded with 500KeV  $Ar^+$  at 1E15cm<sup>-3</sup>. Figure 3.7-5 shows the RBS measurement for 50KeV Si<sup>+</sup> bombarded with 1MeV  $Ar^+$  at 1E15cm<sup>-2</sup>. The before and after annealing measurement of the 500KeV  $Ar^+$  bombardment in Figure 3.7-3 shows some signs of damage removal after the 600°C anneal. In Figure 3.7-5 the before and after anneal data, for the 1MeV bombardment indicating minimal damage removal.

### 4.0) Summary and Recommendation

Bombardment of GaAs with high energy  $Ar^+$  ions produced a deep amorphized region in excess of the 50KeV and 120KeV Si<sup>+</sup> ion implants in the samples studied. However attempts to regrow the amorphous region by low temperature annealing were ineffective. The electrical activation of Si<sup>+</sup> implants in the  $Ar^+$  bombarded samples was obscured by the compensating effects of residual crystalline damage. The program phase I results can be summarized as shown below:



Figure 3.7-2) Electron Diffraction of Regrown 500KeV Ar<sup>+</sup> Samples







Figure 3.7-4) Rutherford Backscattering Profile - 500KeV Ar<sup>+</sup>



Figure 3.7-5) Rutherford Backscattering Profile - 1MeV Ar<sup>+</sup>

Tasks Performed	Evaluation Method	Results
Amorphization of GaAs with 500KeV, 1MeV Ar <sup>+</sup>	Ellipsometry	Deep damage >4000Å
Pre-implant <sup>29</sup> Si <sup>+</sup> 50KeV, 120KeV	Flash annealed, C-V profile data LSS projection for Si <sup>+</sup>	Carrier profiles in agreement with LSS projections
Low temperature annealing to 600 <sup>0</sup> C in reducing atmosphere	Ellipsometry, C-V, resistivity meas. RBS, electron diffraction	Incomplete re-cryatallation
Assessment of low temperature activation of Si <sup>+</sup>	C-V, Resistivity measurement	No apparent activation of Si <sup>+</sup> due to residual bombardment damage

A focus of the major difficulty experienced in phase I was the problem producing high quality regrown layers. The extent of damage in the  $\operatorname{Ar}^+$  bombardment appeared to be in excess of what could be removed by annealing. Anneal temperatures up to 950°C were not effective in regrowing the damaged region. We believe the major difficulty resides in the method of producing the amorphous layer and not the annealing method providing a sufficiently high temperature.

We therefore recommend that future research be focused primarily upon the basic amorphization step and use impurity implants only as a demonstration of electrical activity in high quality regrown layers. In our study the 77K variable was not the only factor in preventing a source of defects from forming that would later complicate low temperature epitaxy. We believe 77K bombardments will circumvent the generation of point defects and extended defects from dymamic annealing, but crystalline regrowth problems can also arise from the following effects:

Primary Effect	Result
1) Displacement Effects	Long range disorder of compound semiconductor lattice, anti site defects, interstitials, precipatates
<pre>2) Solubility Limit Effects (As<sup>+</sup>)</pre>	Anti site defects, stoichiometry defects, interstitials and precipitates
3) Inert Gas Solubility Effects	Interstitials leading to deep electrical defects with compensating effects on shallow impurities

Resolution of the above three effects will require definitive HRTEM measurements in a study of amorphizing <u>species</u> and <u>dose</u> for energies >500KeV.

A recommendation of  $As^+$  as the desired amorphizing species is also important.

Unless severe displacement effects prevail the low temperature epitaxy method should work and provide an effective means of activating ion implanted impurities in sensitive materials.