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ANNUAL REPORT

Crystal Growth of Device Quality GaAs in Space

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I. SUMMARY

The present program was initiated in 1977, and it has been aimed at solving the fundamental and technological problems associated with "Crystal Growth of Device Quality in Space". The initial stage of the program covering the period 1977-84 was devoted strictly to ground-based research. We felt that before a successful growth process could be developed and optimized for near-zero gravity conditions, we should first identify the unsolved problems associated with the growth of bulk GaAs in the presence of gravitational forces. We felt further that it was equally important to assess, and if necessary develop reliable chemical, structural and electronic characterization methods which would permit the direct relation of the salient materials parameters (particularly those affected by zero gravity conditions) to the electronic characteristics of single crystal GaAs, in turn to device performance. These relationships are essential for the development of optimum approaches and techniques.

The results of the ground-based research have clearly surpassed our expectations: we established that the findings on elemental semiconductors Ge and Si regarding crystal growth, segregation, chemical composition, defect interactions, and materials properties-electronic properties relationships are not necessarily applicable to GaAs (and to other semiconductor compounds). In fact, in many instances we found totally unexpected relationships to prevail. We further established that in compound semiconductors with a volatile constituent, control of stoichiometry is far more critical than any other crystal growth parameter. Detailed results of our ground studies and their discussions have appeared in about sixty publications which have provided a fundamental back-up for technological advancement in GaAs. We have also shown that, due to suppression of nonstoichiometric fluctuations, the advantages of space for growth of semiconductor compounds extend far beyond those observed in elemental semiconductors.

Having developed the necessary characterization techniques, having identified the immense importance of stoichiometry, and having assessed the potential benefits of processing GaAs in space, we proceeded recently with the search for a suitable configuration for the growth of GaAs from the melt in space. We have developed a novel configuration for "partial confinement of GaAs in space" which overcomes the two major problems associated with growth of semiconductors in total confinement: volume expansion during solidification and control of pressure of the volatile constituent (details discussed below). Development of this configuration for space experimentation has been approved by the Office of Materials Processing in Space and will be funded in the near future.

We should point out at this time that our experimental arguments and discussions on the potential benefits of space processing of GaAs led to an already-signed Agreement between the National Aeronautics and Space Administration and Microgravity Research Associates, Inc., for a Joint Endeavor in the Area of Materials Processing in Space. This endeavor involves the growth of GaAs and other compound semiconductors employing liquid phase electroepitaxy. For about the last two years the development of a breadboard configuration for space growth of GaAs with this method has been sponsored in our laboratory by Microgravity Research Associates and is distinctly separate from our work supported by NASA.

In the proposal of our space experiment, its development and assessment, we assumed that our present program will evolve into its logical stage whereby the ground-based research is carried out simultaneously and in direct correlation with, and in support of, the space growth experiments. This ground-based research is designed to provide a fundamental guidance and back-up for GaAs space growth.

It combines three elements: crystal growth, device-related properties, and characterization on a micro- and macro-scale. We believe, based on our several years of experience, that the research along these lines is now critical for ensuring a successful growth of device quality GaAs in space.

During the last three years significant improvements in GaAs crystal quality have been made on earth having an immediate impact on GaAs electronics and opto-electronics devices for governmental and commercial applications. We believe that significant improvements will continue to be made on earth. However, we are convinced that the quality and findings potentially attainable in space can under no circumstances be obtained on earth, as discussed later on in this renewal.

II. PROGRESS TO DATE

II.1. Introduction

Since the initiation of this program in 1977 our ground-based research has led to discoveries and significant developments in three areas which are fundamental for engineering of semiconductor materials: (a) crystal growth, (b) macro- and microscale characterization, and (c) phenomena and processes relevant to device applications. The scientific results of our study have been reported to NASA annually in seven consecutive Annual Progress Reports starting in April 1978. The major developments are summarized in Table I. Detailed discussions of the results are also given in about sixty scientific publications. (The list is attached.) Thus, in the "Progress to Date" section of this proposal only selected results which bear directly on GaAs growth in space will be presented, so that the forthcoming stage of our research correlating ground-based and space experiments can be viewed in a better perspective.

TABLE 1

PROGRESS TO DATE - SUMMARY OF MAJOR DEVELOPMENTS

(Shaded areas indicate the most important achievements in the last three years)

	Development	Comments	Representative References
LPEE-Liquid Phase Electroepitaxy	1. Growth Kinetics Model 2. Dopant Segregation Model 3. Model of Multicomponent Systems 4. Interface Stability Model	Theoretical model was developed which explains experimental LPEE characteristics. The model is based on a mass transport process with two driving forces: solute electromigration and the Peltier Effect.	1-3 14,15,21,33
	5. In-situ Monitoring of Growth	Successful in-situ monitoring of the growth velocity was realized for the first time in LPE using the contactless LPEE configuration.	
	6. Growth of Bulk Crystals	LPEE process was successfully extended to a growth of bulk crystals of the thickness of the order of 1 mm.	
	7. Growth in Space Environment	LPEE process was selected in 1983 in order to realize the growth of GaAs in Space. Further R&D effort in this area is realized under a sponsorship of Microgravity Research Associates.	
MELT GROWTH	1. Construction of Advanced GaAs Melt-Growth System	Advanced system has been designed & constructed for horizontal and/or vertical growth of GaAs. The system provides unique feasibility for controlling and monitoring growth parameters.	34,35
	2. Growth of n-type Dislocation-Free GaAs	Utilizing precise control of As pressure above the melt we have achieved reproducible growth of dislocation-free GaAs in a horizontal Bridgman configuration.	43,58
	3. Growth of Electron Trap-Free GaAs	Growth conditions were discovered which lead to melt-grown GaAs of superior structural & electronic properties. For the first time electron trap-free bulk GaAs was achieved.	38
	4. Identification of the Role of Oxygen in Melt Growth of GaAs	Oxygen has been identified as a constituent of growth system which affects electronic and stuctual properties of GaAs.	23,60

TABLE 1

PROGRESS TO DATE - SUMMARY OF MAJOR DEVELOPMENTS

(Shaded areas indicate the most important achievements in the last three years)

	Development	Comments	Representative References
MELT GROWTH (cont.)	5. Role of Stoichiometry	Stoichiometry was identified as a fundamental factor controlling structural & electronic properties of GaAs.	59,34,43
PROPERTIES AND PHENOMENA	1. Relationships between Electronic Properties & Melt-Growth Conditions on Micro-Scale	Microprofiles of electron & ionized impurity concentrations in melt-grown GaAs were obtained for the first time.	17
	2. Stoichiometry controlled segregation;	It was shown that impurity segregation in melt-grown GaAs is governed by amphoteric doping and deviation from stoichiometry.	17,43
	3. Interaction between Epitaxial Layer & Substrate	It was demonstrated that outdiffusion of recombination centers from the substrate into LEP layers during growth process takes place. Growth conditions were formulated to minimize outdiffusion	6
	4. Growth-Property Relationships in Epitaxial Growth	It was found that growth rate variations have significant effect on the formation of recombination centers in GaAs.	7,25
	5. Stoichiometry controlled deep levels	A direct relationship was established between As atom fraction in the melt and the concentration of electron traps in GaAs.	36,59
	6. Stoichiometry controlled dislocation density	It was found that an optimum stoichiometry defined by arsenic source temperature 617-618^{o}C corresponds to a minimum dislocation density	59
	7. Oxygen related midgap level	We have unambiguously identified the oxygen related deep level ELO at 0.825 eV below the conduction band.	53,57,60
	8. Origin and properties of Major Electron Trap in GaAs	0.82 eV electron trap in GaAs has been identified as native defect complex involving the antisite As_{Ga}	36,59

PROGRESS TO DATE - SUMMARY OF MAJOR DEVELOPMENTS

(Shaded areas indicate the most important achievements in the last three years)

	Development	Comments	Representative References
PROPERTIES AND PHENOMENA (cont.)	9. Passivation of Deep Levels by Hydrogen	It was found that a concentration of the major deep level in GaAs can be effectively controlled by atomic hydrogen introduced by a standard plasma treatment.	38
	10. Optoelectronic Properties of InP	Cathodoluminescence studies of InP were completed	20
	11. Interface States	Surface states on GaAs-anodic oxide interface were determined with modified DLTS	18
	12. GaAs-Anodic Oxide Interface	A gigantic photoionization effect on GaAs-oxide interfaces was discovered. Utilizing this phenomenon it was shown, for the first time, that both deep & shallow interface states originate from Ga and As vacancies.	28
	13. Fermi Energy Control of Point Defect Formation	Electron Traps in GaAs can be controlled by changing the Fermi Energy during postsolidification cooling of crystals.	59
	14. Fermi Energy Control of Dislocations	Dislocation density in GaAs was found to vary over 5 orders of magnitude when the Fermi Energy was shifted by about 0.3 eV. This discovery was explained in terms of vancancy coalescence which is controlled by the charge state of vacancies.	54
	15. Fundamental Limitations of High Mobility Transistors	A theoretical model was formulated for electron scattering in a two-dimensional electron gas. Absolute and inherent mobility limits were calibrated for GaAlAs-GaAs heterostructures.	52, 55
FUNDAMENTAL ASPECTS OF SPACE PROCESSING	1. Advantages of Space for the Growth of GaAs	We have identified for the first time potential advantages of zero-gravity for the growth of GaAs which stem from recently discovered nonstoichiometric defects affected by thermal and solutal convection.	61

TABLE I

PROFRESS TO DATE - SUMMARY OF MAJOR DEVELOPMENTS

(Shaded areas indicate the most important achievements in the last three years)

	Development	Comments	Representative References
FUNDAMENTAL ASPECTS OF SPACE PROPERTIES (cont.)	2. Novel configurations for semiconductor growth in space	We have proposed a new configuration which is based on a partial confinement of the growth melt by a prism of the triangular cross section. This configuration permits a control of the melt composition during the growth and accomodates volume expression of GaAs upon solidification.	61,62
CHARACTERIZATION	1. Characterization methods based on electron mobility and free carrier absorption	Quantitative methods were developed for determination of compensation ratio in GaAs and InP	12,17,19
	2. IR Scanning Absorption	Quantitative method was developed for microprofiling of carrier concentration & compensation ratio through free carrier absorption	17
	3. Derivative Surface Photovoltage and Photocapacitance Spectroscopies	New Approach was developed for determination of deep levels, band structure and shallow impurites	19,20
	4. Characterization of Semi-Insulating GaAs	A rigorous procedure was developed for the determination of ionized impurity concentration from transport measurements in SI material	
	5. SEM-Cathodoluminescence	Advanced variable temperature system was set up for cathodoluminescence microprofiling of defects, impurities & carrier concentration	41

TABLE 1

PROGRESS TO DATE - SUMMARY OF MAJOR DEVELOPMENTS

(Shaded areas indicate the most important achievements in the last three years)

	Development	Comments	Representative References
CHARACTERIZATION (cont.)	6. SEM-Electron Beam-Induced Current	Variable temperature system was set up for instantaneous profiling of diffusion length	
	7. Laser Scanning Photovoltage	Photovoltage microprofiling was developed for studying homogeneity of semi-insulating GaAs	
INTERACTION WITH INDUSTRIAL ORGANIZATIONS	1. Workshops, 1977 1981	Workshops were held with representatives of leading industrial & educational institutions devoted to the assessment of present status, major problems & future prospects for GaAs growth & applications	5
	2. Literature Survey, 1977 1982	The literature survey on GaAs was updated identifying the leading organization & most important trends in GaAs research and development	
	3. Exposure of the Program to Scientific Community	The present program and its major developments were exposed to the scientific community through a series of seminars given in industrial organizations (RCA, Texas Instruments, Hewlett-Packard, Hughes Int'l., Xerox, Eastman Kodak, Fujitsu Laboratories, NTT, etc.), presentations at scientific meetings and/or direct contacts with individual scientists	
	4. Working Contacts	Contacts were established with industrial organizations in the area of GaAs characterization, growth & device applications. Material supplied by industrial organizations has been characterized on many occasions Interaction with Microgravity Research Associates was established and aimed at the growth of GaAs in a space environment.	

II.2. Relationships between Stoichiometry and Properties of GaAs Crystals

We have discovered that the stoichiometry variations in the GaAs melt during growth constitute the most critical parameter regarding defect formations and their interactions. This defect structure determines all of the relevant characteristics of GaAs. Thus the control of stoichiometric variations caused by thermal and solutal convection in the melt is the key problem for improvement of the quality of GaAs crystals.

Stoichiometry and Semi-Insulating Behavior. Semi-insulating GaAs of enhanced thermal stability is commonly used for production of low and medium scale integrated circuits for governmental and commercial applications.[63-65] Control of the melt stoichiometry is the key to reproducible growth of "undoped" semi-insulating GaAs. The very high resistivity of this material is achieved due to the balance between ionized acceptors and deep donors commonly referred to as the "EL2 family".[66] Our experimental studies have shown that growth from arsenic-rich melt increases the concentration of the antisite defects which act as deep native donors. The results of the Rockwell (ref. 68) and Westinghouse (ref. 69) research groups reinterpreted using our theoretical analysis of transport phenomena are shown in Fig. 1. Indeed, high resistivity ($\rho \gtrsim 10^8$ cm) is obtained only for a narrow arsenic atom fraction range 0.47<[As]<0.52 (Fig. 1a). The major segment of this range yields low Hall effect mobilities (Fig. 1b) which correspond to mixed conductivity or to p-type conductivity. High quality crystals with high mobilities (>5000 cm^2/Vs) are obtainable only from melts slightly enriched with arsenic [As]$\gtrsim$0.505.

Stoichiometry-Controlled Deep Levels and Dislocations. Results of our research on horizontal Bridgman (HB) growth of GaAs where the melt stoichiometry is varied by changing the temperature of the arsenic source, T_{As}, have shown

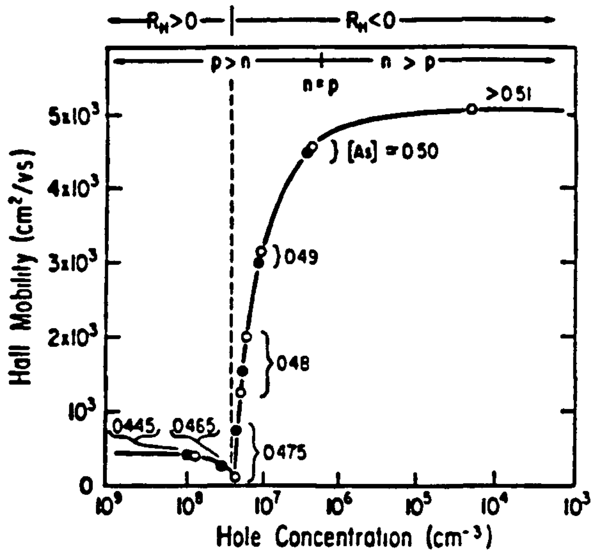


Figure 1. The results in this figure confirm the importance of our discovery of stochiometric effects. Hall mobility data for LEC-grown GaAs with p to n transition induced by enrichment of the melt with arsenic.

o - results of Rockwell group (ref. 68)

● - results of Westinghouse group (ref. 69) (see Fig. 2) that the concentration of the dominant midgap level EL2 decreases with decreasing arsenic atom fraction in the growth melt (Ref. 36). This dependence is consistent with the assignment of EL2 to the arsenic antisite As_{Ga}, and it explains the stoichiometry-induced n- to p-type transitions shown in Fig. 1.

Our group has also demonstrated the relationship between stoichiometry and density of dislocations (Ref. 43). As shown in Fig. 3a and 3b, the dislocation density in HB GaAs crystals responds to the changes in melt composition in a way which is very similar to the behavior of nonstoichiometry, ρ, defined as the difference between the concentration of arsenic and gallium atoms (Ref. 70).

Stoichiometry-Controlled Inhomogeneities. The first indication of the effects of stoichiometry on the properties of GaAs on a microscale was provided by our analysis of the carrier concentration variation in melt-grown crystals (Ref. 17). In elemental semiconductors electrical inhomogeneities are caused by variations of the growth velocity. In GaAs, however, carrier inhomogeneities can develop even when impurities are distributed homogeneously throughout the crystal. Experimental results illustrating such behavior are shown in Fig. 4a and b. It is seen in Fig. 4a that the total concentration of impurities [Ge] remains constant, whereas the concentration of free electrons exhibits dramatic fluctuations. This effect, which cannot be explained in terms of standard segregation kinetics, stems from stoichiometry-induced amphoteric behavior, i.e., impurity incorporation into Ga or on As sites, which leads to donor or acceptor behavior, respectively. Indeed, we have proven that effects similar to those of Fig. 4a are produced by intentional stoichiometry changes during crystal growth, as shown in Fig. 4b.

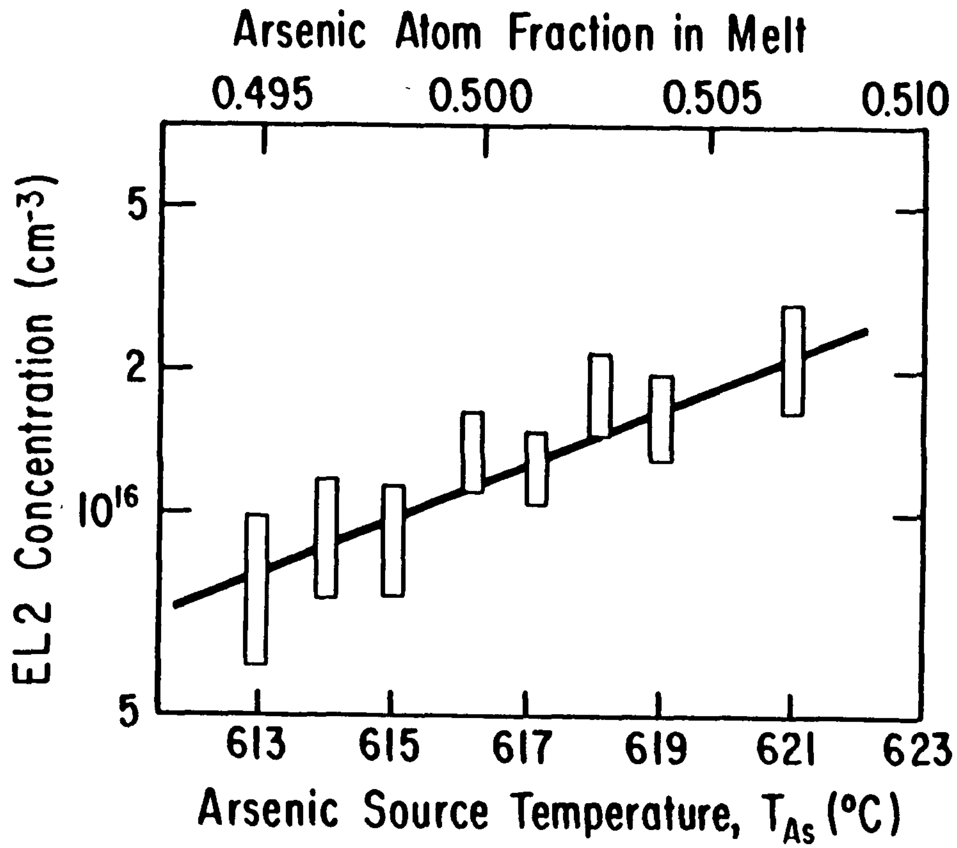


Figure 2. See text

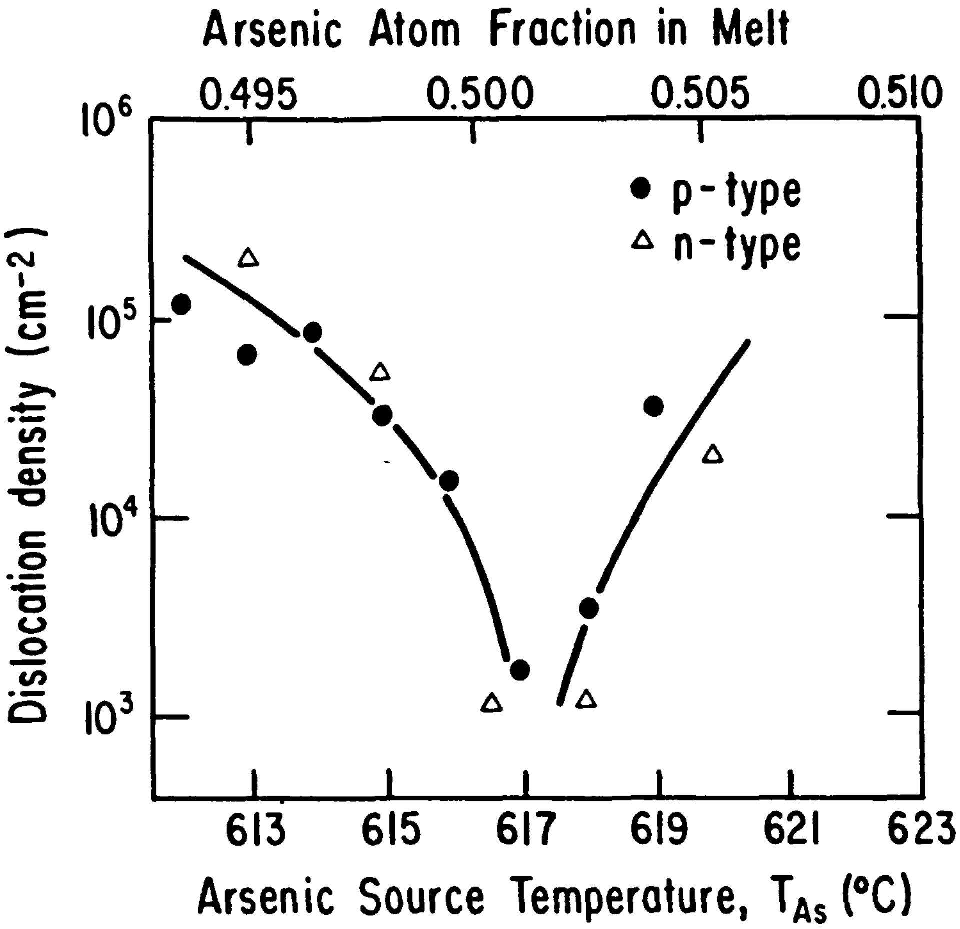


Figure 3a. Dislocation density dependence on the melt stoichiometry.

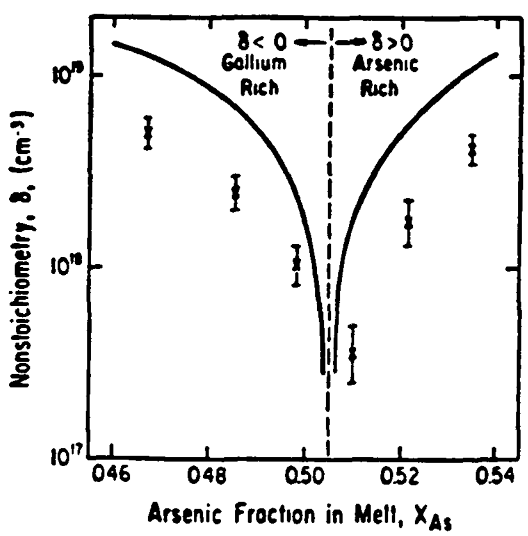
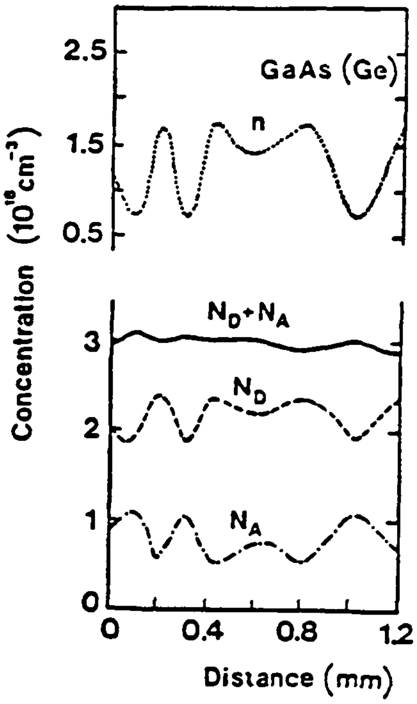


Figure 3b. Theoretical line and experimental points on GaAs non-stoichiometry.

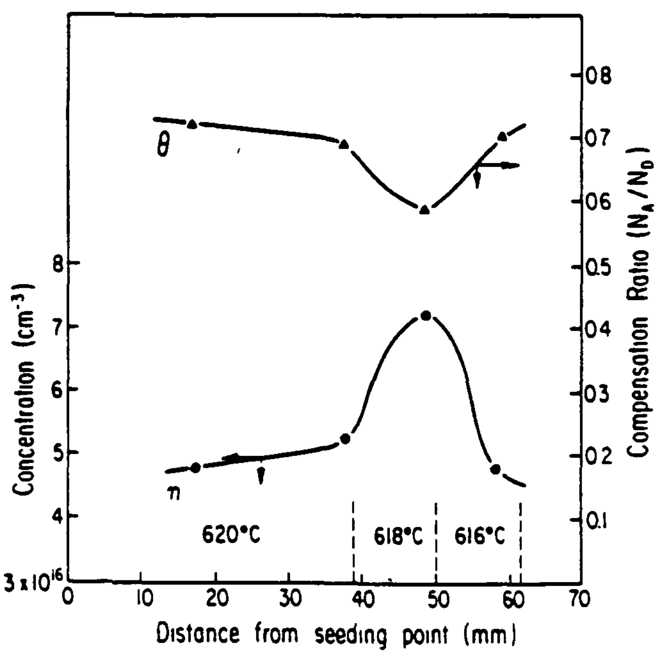


(a)

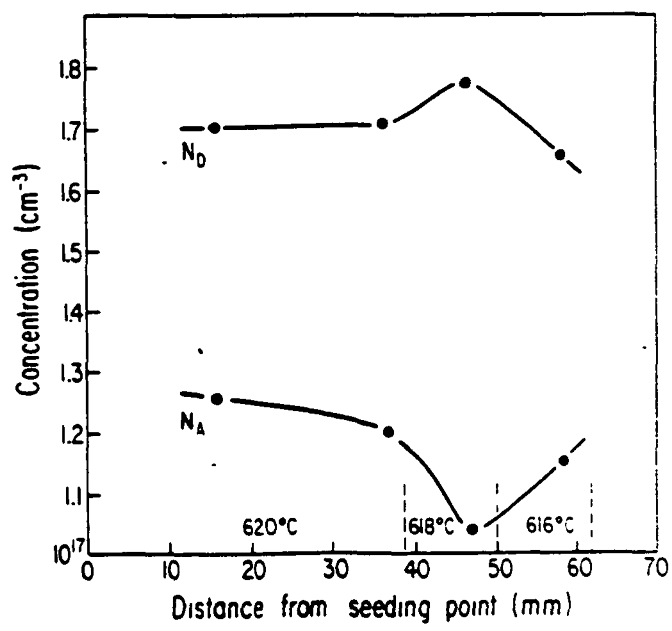
Figure 4.

Electron concentration and ionized impurity microprofiles of·Ge-doped melt-grown GaAs obtained with scanning IR absorption spectroscopy. Note corresponding variations in the concentration of donor and acceptor impurities showing that amphoteric doping does not obey standard segregation kinetics.

(b)



Dependence of the compensation ratio and free carrier concentration on the arsenic source temperature (pressure).



Response of donor (N_D) and acceptor (N_A) concentrations to arsenic source temperature as determined from the compensation ratio Total ionized impurity content is essentially constant.

II.3. Advantages of GaAs Growth in Space

The advantages of zero-gravity conditions in solidification in general, and semiconductor crystal growth in particular, stem primarily from the suppression (or virtual elimination) of thermal and solutal convection in the melt (Ref. 30). Furthermore, growth in space is a promising means for overcoming constitutional supercooling, which on the ground limits the yield of crystal growth of alloys and heavily doped semiconductors. As demonstrated in early experiments, elimination of thermal convection causes impurity segregation to proceed under ideal diffusion-controlled conditions and leads to a uniform dopant distribution and enhanced homogeneity of the crystals (Ref. 30).

Regarding the growth of compound semiconductors, and especially GaAs, the potential advantages of space extend far beyond the effects of impurity segregation and constitutional supercooling.[61] They relate to the profound role of the melt stoichiometry and stoichiometry fluctuations discussed above. The effects of stoichiometry were discovered only recently, and thus are not yet fully appreciated by many researchers.

The potential advantages of space for growth of GaAs crystals predicted from our ground-based research are summarized below in Table II. Stoichiometry and its control are the overwhelmingly important factors involved, as clearly indicated by our ground-based studies. For comparison, the advantages of space for the growth of elemental semiconductors, as deduced from previous space experiments, are included.

II.4. Direct Impact on Space Growth Programs

Our ground-based research has had a direct impact on crystal growth in space. Our developments in liquid phase electroepitaxy and in melt growth have evolved into two unique programs on GaAs growth in space which are currently being carried out under sponsorship of Microgravity Research

Table II

SUMMARY OF ADVANTAGES OF SPACE FOR GROWTH
OF SEMICONDUCTOR CRYSTALS FROM THE MELT

	Elemental Semiconductors (Ge;Si)	Semiconductor Compounds (GaAs & Related)
Effects of Microgravity on Crystal Growth Parameters	o Elimination of growth velocity fluctuations caused by thermal convection	o Elimination of growth velocity fluctuations caused by thermal convection in the melt
		o Elimination of growth velocity fluctuations caused by solutal convection
		o Elimination of stoichiometry variations caused by solutal convection
		o Elimination of stoichiometry variations caused by thermal convection in the melt
		o Elimination of stoichiometry variations caused by thermal convection in the vapor phase
	o Reduced effects of constitutional supercooling	o Reduced effects of constitutional supercooling
Effects of Microgravity on Crystal Properties		
Impurity	o Homogeneous distribution (due to diffusion-controlled growth	o Elimination of variations in amphoteric behavior (due to constant stoichiometry)
Native Point Defects		o Elimination of variations in deep compensating centers (resulting from stoichiometry variations)
		o Elimination of variations in recombination centers (resulting from stoichiometry variations)
		o Elimination of resistivity variations (resulting from stoichiometry variations)
Dislocations		o Suppression of dislocations due to enhanced control of stoichiometry

Associates and NASA, respectively. These two programs are briefly outlined below.

Electroepitaxial Growth of Bulk GaAs in Space. **Liquid phase** electroepitaxy (LPEE) is the only known growth process which has yielded **thick GaAs** crystals of ultra-high structural and electronic perfection. We have been developing this method and have established that implementation of this technique on the ground is impeded by the detrimental effects of thermal convection and of solutal convection in the liquid pahse.

In 1983 under the sponsorship of Microgravity Research Associates, we initiated an extensive study on the fundamental and practical problems related to the adaptation of the LPEE process to the microgravity space environment. The long-term goals of this work include: conceptual development of electro-epitaxy apparatus compatible with space environment; analysis of processes and phenomena limiting the quality of material grown in space by the LPEE process; optimization of hardware design and related interaction with the hardware manufacturer. Electroepitaxy growth experiments carried out under MRA sponsorship have shown that this technique indeed makes it possible to achieve thick GaAs crystals of outstanding electronic and structural characteristics. Thus, we have grown GaAs up to 3 mm thick with free electron concentration of about $2 \times 10^{14} cm^{-3}$ and electron mobility $\mu_{300} = 7000\ cm^2/Vs$. Furthermore, we have discovered that dislocation density decreases during prolonged LPEE growth, which opens the possibility of achieving virtually defect-free GaAs.

These developments we consider striking. It is now evident that the successful realization of the LPEE process in space carries the **promise** of a major breakthrough in GaAs and related compounds.

Growth of GaAs Crystals from the Melt in a Partially Confined Configuration. As pointed out earlier, our ground-based research has demonstrated that stoichiometry is the single most important factor in the melt growth of GaAs (see Table II). It is, thus, imperative that in order to benefit fully from the space environment a new growth configuration must be developed which permits the control of melt stoichiometry during the growth process and accommodates volume expansion during solidification.

Under zero gravity, melts acquire a shape corresponding to the minimum surface energy. Unconfined melts, generally preferred to avoid contamination, would acquire a spherical shape which is not suitable for directional single crystal growth. In previous space growth experiments cylindrical containers were employed. This confinement cannot accommodate the volume expansion upon solidification, and furthermore, it leads to major problems in controlling the melt stoichiometry during growth.

In 1984 we proposed to NASA's Office of Materials Processing in Space that we undertake the development of GaAs growth from the melt in a novel "partially confined configuration"[61,62] which we believe offers a unique solution to the problems outlined above. In this novel growth configuration a triangular prism is employed to contain the growth melt (see Fig. 5).

Under zero gravity the melt in a triangular prism acquires a cylindrical shape with a circular cross section of Fig. 5a, which corresponds to a minimum in surface energy. It can be readily shown, on theoretical grounds, that in the absence of wetting, the circular cross section is energetically more stable than the cross section of Fig. 5b. The empty spaces between the cylindrical melt and the edges of the prism provide the necessary room to accommodate expansion during solidification. Furthermore, they constitute three channels through which a vapor phase of controlled pressure can be in contact with the melt during the growth process. In Fig. 5c GaAs crystal growth is considered in a

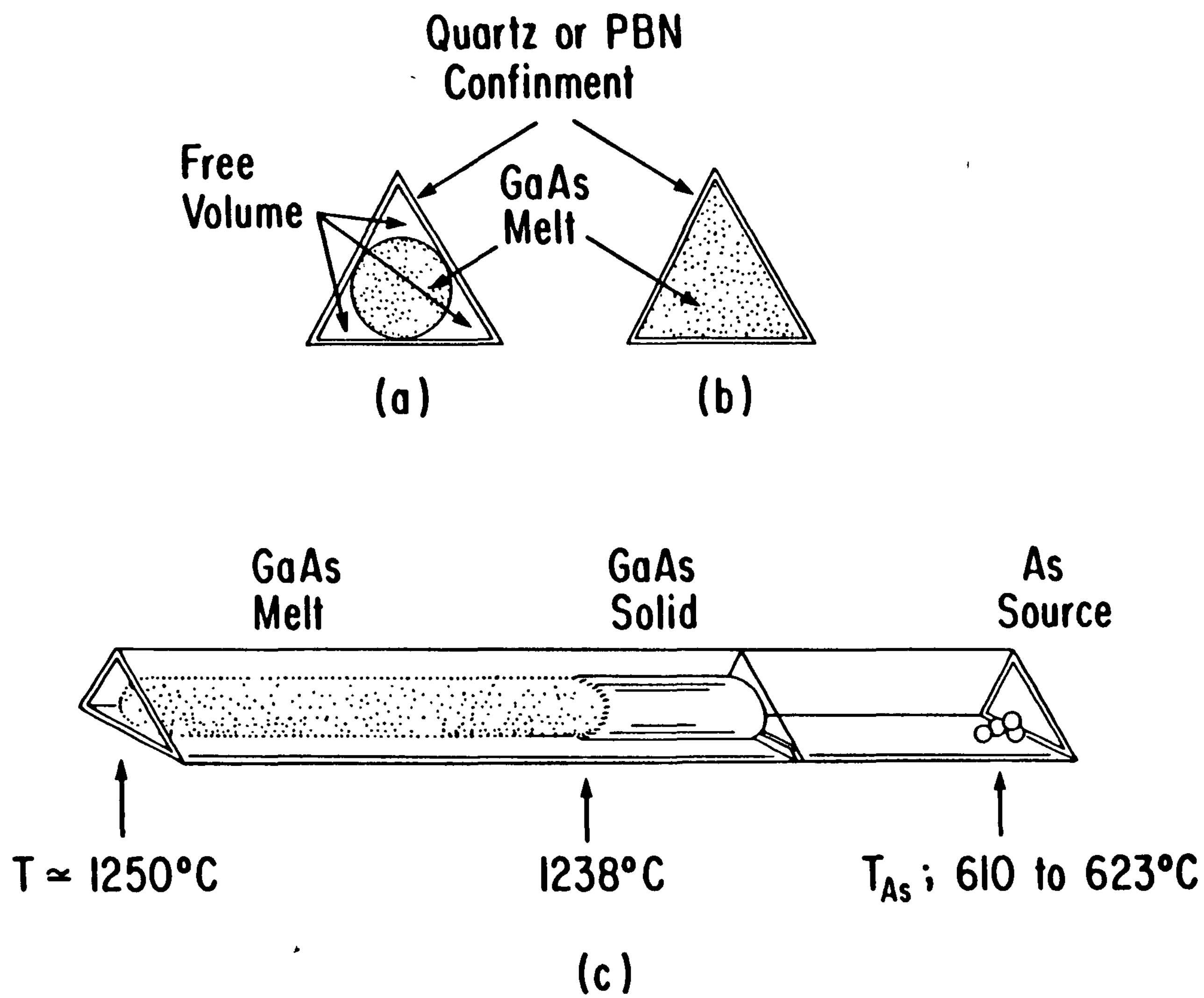

Quartz or PBN Confinment
Free Volume
GaAs Melt
(a)
(b)
GaAs Melt
GaAs Solid
As Source
T ≃ 1250°C
1238°C
T_{As} ; 610 to 623°C
(c)

Figure 5.

Bridgman-type configuration. An arsenic source is used to provide the arsenic vapor pressure desired to control themelt composition during growth by means of an arsenic source temperature. This aspect is of fundamental importance for the reasons pointed out above.

III. PROPOSED RESEARCH

III.1. Motivation and Objectives

As pointed out earlier, considerable progress has been made in recent years in the growth of GaAs single crystals for IC applications.[42, 63-65] In situ synthesis of the compound combined with the use of BN crucibles increased the purity of single crystals to the extent that semi-insulating "undoped" GaAs became available in contrast to Cr-doped material previously employed. Undoped SI GaAs exhibits higher electron mobility and enhanced thermal stability. Only recently, isoelectronic impurities have been found to reduce dislocation density making it possible to achieve dislocation-free SI GaAs (see e.g., ref. 58). These features are, of course, very important for device applications and have stimulated a rapidly growing interest in understanding the growth-property relationships relevant to device applications.

Our research has led to the identification of two factors of key importance: the stoichiometry and the post-solidification interactions of defects. We have also concluded that under zero gravity the stoichiometry effects can gain a high degree of controllability unattainable on earth due to interfering effects of the solutal and thermal convection. From our ground-based research we have deduced specific potential advantages of the space growth of GaAs crystals relevant to improved device quality. These advantages (see Table II) extend far beyond those projected for elemental semiconductors which are associated primarily with impurity segregation phenomena. It is for that reason that we now believe GaAs growth in space has to offer to our fundamental and applied knowledge on electronic materials much more than has been realized.

Our research and development ground-based program has been thus far designed to develop a crystal growth process compatible with space environment experimentation and to establish a facility for reliable structural, compositional, and electronic characterization of GaAs. These tasks have been largely completed. We have designed two space growth configurations for electroepitaxial growth and for melt growth. We have also set up a comprehensive characterization facility.

From now on, our ground-based research program will be polarized towards the optimization of the space growth experiments. It will, thus, evolve into the next logical stage aimed at the fundamental guidance and back-up for the space growth experiments which will be pursued in parallel. This stage will still combine three elements pursued in the past, i.e., crystal growth, device-related properties, and characterization on a micro- and macro-scale. However, emphasis will be shifted from establishing the fundamental relationships to answering crystal growth and characterization problems directly related to space engineering of GaAs crystals of improved device quality.

We believe that significant improvement in GaAs quality will continue to be made on earth. However, we also believe that the quality and findings potentially attainable in space can under no circumstances be obtained on earth.

III.2. Scope

A timetable of the proposed research is given in Table III. This timetable includes important new elements which reflect transition of our program into the next logical stage. Consistent with the previous study our program involves extensive GaAs crystal growth from the melt. The proposed research is subordinated to various aspects of engineering of electronic materials in space. Thus, the crystal growth includes GaAs growth by Horizontal Bridgman and by Liquid

Encapsulated Czochralski techniques, however, both experimental systems will be equipped with magnetic field generators[67] in order to achieve the control of convection in our ground-based crystal growth experiments. We consider the growth in a magnetic field extremely important for studying the fundamental aspects of space processing. Growth-property relationships are focussed on control of point defects and dislocations, and they include phenomena taking place during the solidification and post-solidification defect interactions. Defects in GaAs can be either detrimental or beneficial to GaAs IC technology.[59] The main objective of our study is to achieve GaAs crystals with improved device quality, i.e., crystals in which detrimental effects of defects are suppressed or virtually eliminated, and the beneficial effects are enhanced. These objectives are explicitly defined under "property-device relationships" of Table III.

Our characterization techniques are designed to determine the salient materials parameters and to relate them to the electronic characteristics of single crystal GaAs, and in turn to device performance. We propose to enlarge our micro-scale characterization, which we consider of key importance for GaAs processing in space, by setting up a photoluminescence scanning facility. This technique is being introduced in industrial organizations as a standard in the assessment of the homogeneity of GaAs wafers.

III.3. Ground-Based Research Guiding and Backing Up Space Growth Programs

III.3.1. Crystal Growth

Approaches and techniques employed in the growth of GaAs from the melt are summarized in Table IV. They include Bridgman growth system, which provides a high precision means for controlling the melt stoichiometry during the crystal growth, and two LEC systems with provisions for in-situ systems of GaAs. One LEC system is equipped with a generator and a magnetic field. The study is in

GROUND PROGRAM

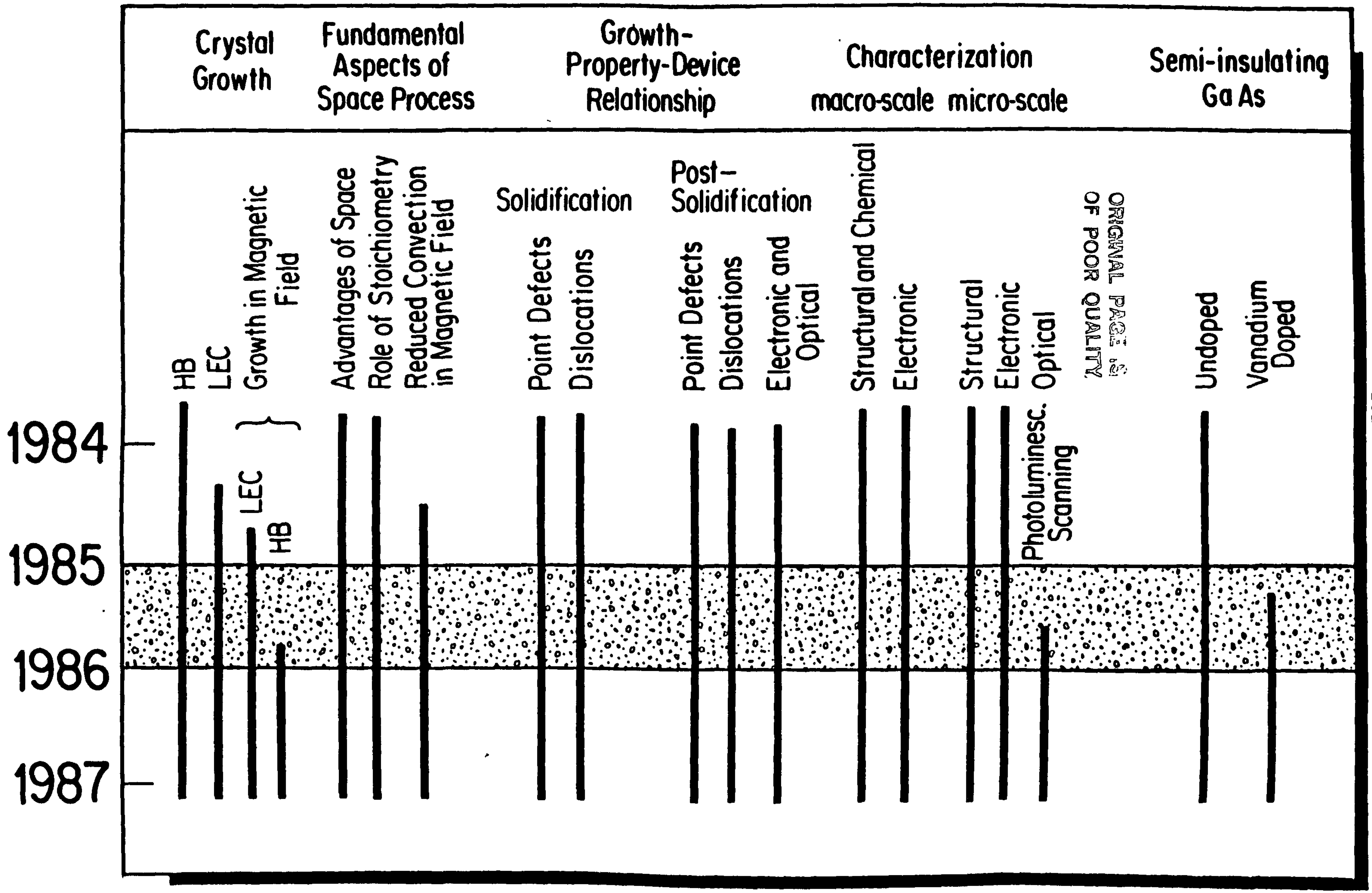


TABLE IV

Approaches and Techniques Employed in the Growth of GaAs

from the Melt

Method	Description	Comments
Horizontal-Vertical Bridgman System	Unique apparatus with ultra-high precision control of growth conditions & feasibility of operation in a horizontal or vertical mode. It proves possibility for studying growth-property relationships & for the growth of ultra-high quality GaAs.	Available in our Electronic Materials Group. Temperature control system will be modified in 1985.
Horizontal-Bridgman System with Magnetic Field	Magnetic Field up to 3000 Oe perpendicular to growth direction will be generated by a moveable electromagnet.	Apparatus should be completed in 1985; Electromagnet will be donated by Varian Associates.
Liquid Encapsulated Czochralski System	High pressure apparatus with provision of in situ synthesis of GaAs.	Apparatus available in our Electronic Materials Group.
Liquid Encapsulated Czochralski System with Magnetic Field	Prototype apparatus equipped with a generator of a magnetic field up to 3000 Oe parallel to the crystal growth direction.	Apparatus constructed, tested and currently used in our Electronic Materials Group.

progress on a design of the electromagnet for the Horizontal Bridgman apparatus. We expect this study to be completed in early 1985. It should be emphasized that the HB system with a magnetic field would be the very first apparatus permitting the in-situ control of the stoichiometry and of the convectional flow. We consider this apparatus extremely promising for studying the fundamental aspects of crystal growth in space.

Bridgman Growth of GaAs. Our Bridgman-type apparatus (see Fig. 6) with unique control features has already proven to be most effective for the study of growth-property relationships. Utilizing this system, we have established the critical role of As pressure in the structure and electronic properties of GaAs. Furthermore, we defined the optimum As pressure conditions which reproducibly yield low dislocation density crystals. These optimum melt-growth conditions have already been adopted by commercial producers of GaAs. We have identified and explained the effects of oxygen doping on GaAs properties. We have found that the major electron trap EL2 (0.82 eV below the conduction band) is not solely responsible for the semi-insulating behavior of the so-called "undoped" GaAs. An oxygen-related deep level, ELO, is also present in such material. We have also found (see Fig. 7) that Ga-vacancy coalescence controlled by the Fermi energy is the key factor in formation of deep levels and dislocations in GaAs.

These scientifically and technologically important results have been achieved through the close coordination of growth studies and extensive characterization. We propose to continue our crystal growth-materials property relationships studies with emphasis on problems important for obtaining dislocation-free semi-insulating material of improved homogeneity and thermal stability. This study, initiated only in 1983, has already led to the discovery of a new fundamental process controlling dislocation density. Further growth-property relationship study in p-type GaAs is needed in order to understand post-solidificatin defect interactions

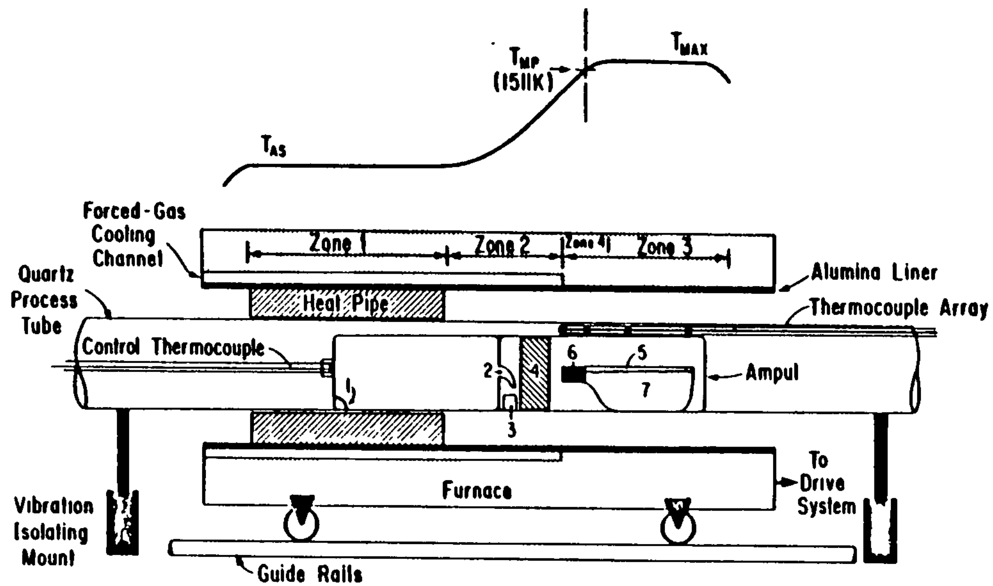


Fig. 6. Schematic representation of the growth apparatus and thermal profile (top). The quartz ampul contains the As source, 1; a breakable seal, 2; and seal breaking weight, 3; the quartz diffusion barrier, 4; a quartz boat, 5; the GaAs seed crystal, 6; and polycrystalline GaAs, 7.

Figure 6.

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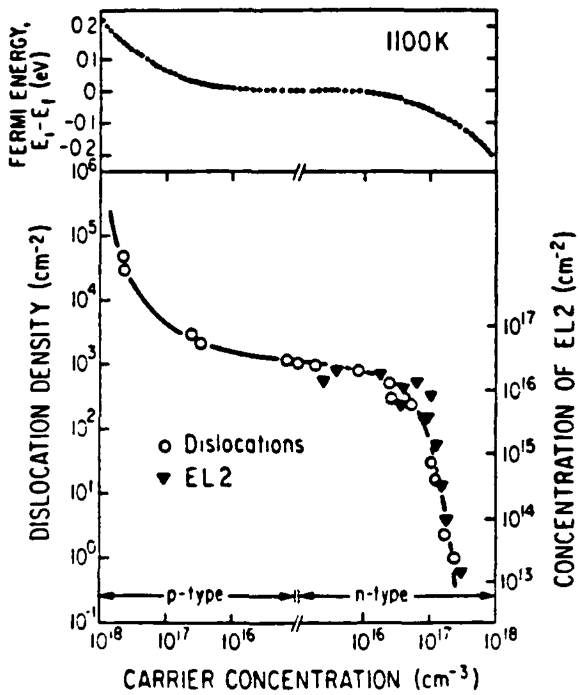


Figure 7. Fermi energy-controlled dislocation density and the concentration of midgap levels EL2

and their role in creating dislocations and electronically active complexes.

We have completed the technical analysis of the feasibility of incorporating an electro-magnet into our Bridgman growth system in such a way that the thermal characteristics of the furnace are not disturbed. A construction of the magnet will be initiated by Varian Associates in early 1985. We also plan to modify the temperature controlling system of the HB apparatus in order to realize magnetic field-controlled Bridgman growth of GaAs.

Czochralski Growth of GaAs. In 1983 we completed the construction of our first liquid encapsulated Czochralski system for in situ compounding and growth of GaAs. In 1984 we completed construction of a second LEC system equipped with a DC magnetic field generator. The system shown in Fig. 8 has been assembled, tested, and is currently used for the growth of GaAs under conditions of "suppressed convection" in the melt.

We propose to utilize the LEC growth system in 1985 for the growth of dislocation-free GaAs doped with isoelectronic impurities and for the growth of semi-insulating GaAs doped with vanadium.

Fundamental Aspects of GaAs Growth in Space. In our ground-based work towards space experiments we plan to utilize magnetic fields in order to decrease the magnitude of convection instabilities in the growth melt (magnetic field increases the kinematic viscosity of electrically conductive melts), and thus, to determine relationships between convection and crystal properties. In Fig. 9 we show microscopic inhomogeneities in an undoped crystal grown in our laboratory by the Liquid Encapsulated Czochralski method in a magnetic field, H. It is seen that in the portion of the crystal grown without magnetic field, very pronounced striations are revealed by "depth profiling" of differentially etched crystals and by cathodoluminescence scanning. The magnitude of inhomogeneities decreases with increasing magnetic field, and the microscopic

Figure 8. L. Pawlowicz (graduate student) checking the LEC system for GaAs growth in a magnetic field.

inhomogeneities are reduced by as much as one order of magnitude at H = 1500 Oe. it should be emphasized that inhomogeneities in undoped GaAs crystals are most likely due to native defects (lifetime killing recombination centers) and not to standard segregation of chemical impurities. Accordingly, the results of Fig. 9 provide experimental evidence that suppression of convection during GaAs growth has highly beneficial effects on the native defects and on their spatial distribution.

We propose to continue GaAs growth in a magnetic field using originally (in 1985) our LEC system and later on (in 1986) our HB system with a magnetic field which should permit us to assess simultaneously the effects and relationships between convection and nonstoichiometry.

III.3.2. Properties and Phenomena Related to Device Application

Studies of device-related properties and phenomena constitute an integral part of our research. In previous stages of the present program we have successfully investigated such problems as, for example:

- solar cell limitations provided by Auger recombination
- defect formation due to acceleration of the growth
- elimination of deep traps by hydrogen in plasma treatment
- electrical properties of GaAs-oxide interfaces
- fundamental limitations of high electron mobility transistors

We plan to pursue this device-related research, however, we also plan to focus our investigation on materials-related aspects in order to define GaAs material with defect structure optimized for IC processing.

It has recently been recognized that native defects can be not only detrimental, but also beneficial to GaAs IC technology. Detrimental effects originate in the high dislocation densities, while beneficial effects are encountered in the compensation mechanism in undoped semi-insulating GaAs. In order to establish the means for controlling native defects, we plan to

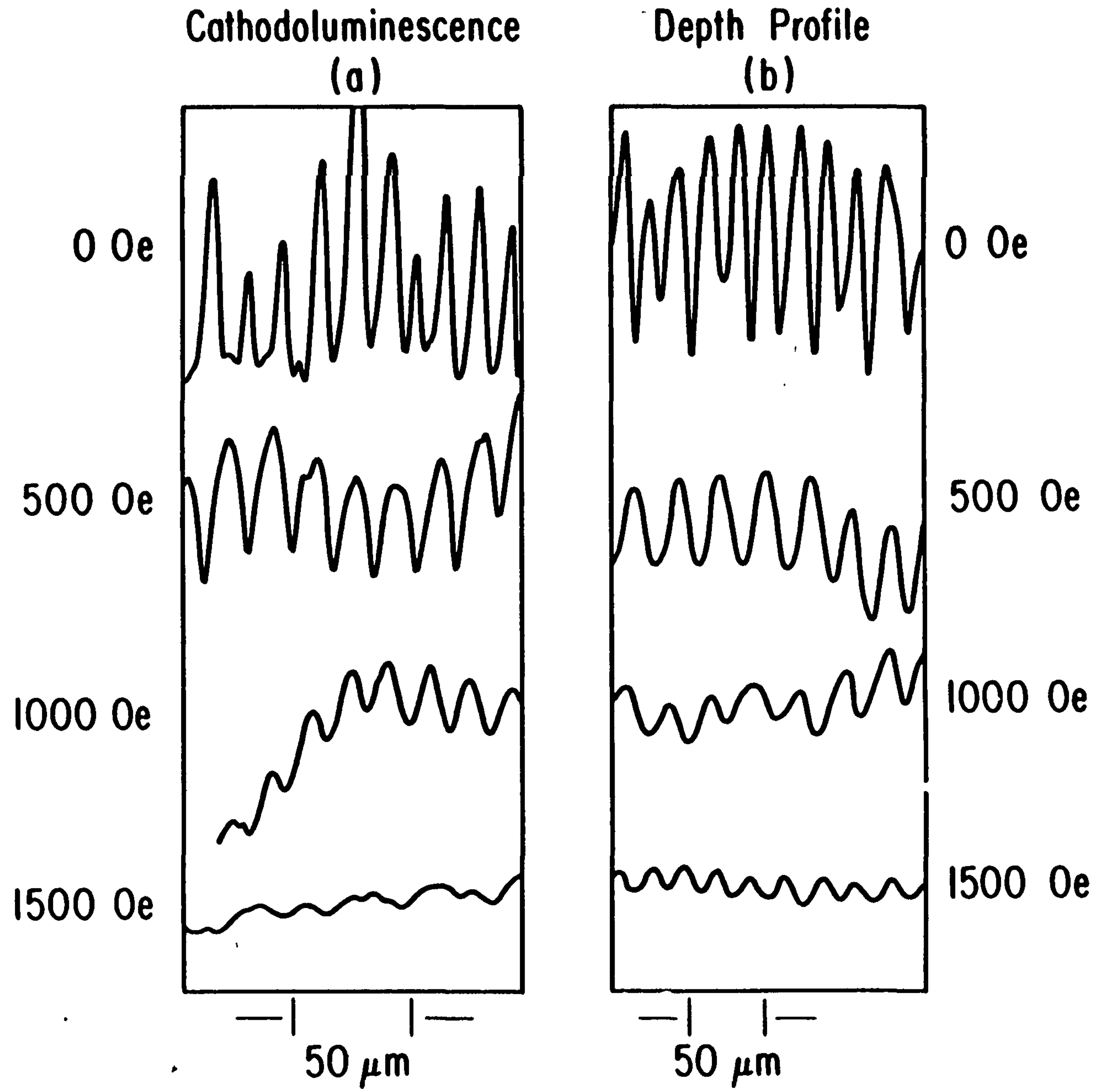


Figure 9. Suppression of microscopic inhomogeneities in GaAs by the magnetic field.

investigate defect evolution during crystal growth. As shown in Fig. 10, for LEC growth of GaAs the formation of defects and their state in GaAs crystals is governed by disorder generated during solidification (1238°C) and by post-solidification processes taking place at temperatures below 1000°C. Post-solidification processes can be identified and distinguished from processes concurrent with solidification due to their dependence on the Fermi energy. We propose to use this approach in order to explain the mechanisms of dislocation generation, the effects of impurities (see Fig. 11), and especially the role of isoelectronic impurities in achieving dislocation-free semi-insulating material.

III.3.3. Characterization

The first stage of our electronic characterization facility was essentially completed in 1980. Since then it has been used for evaluation of grown crystals on a macro- and micro-scale, for the study of growth-property relationships and for quantitative investigation of device-related phenomena and properties of GaAs. Our characterization approaches are of course being continuously upgraded experimentally and theoretically in accord with the state-of-the-art knowledge. Most recently we have refined electronic transport techniques for electrical and optical characterization of semi-insulating GaAs. In 1984 we have completed a new DLTS system for computerized measurements of deep levels. In accord with recent trends in GaAs characterization we propose to set up in 1985 a photoluminescence scanning system.

A brief summary of our characterization facility is given in Table V. It combines standard techniques with novel methods or approaches which we have developed in order to enhance the reliability of our studies. Our techniques listed in Table V make possible a comprehensive characterization

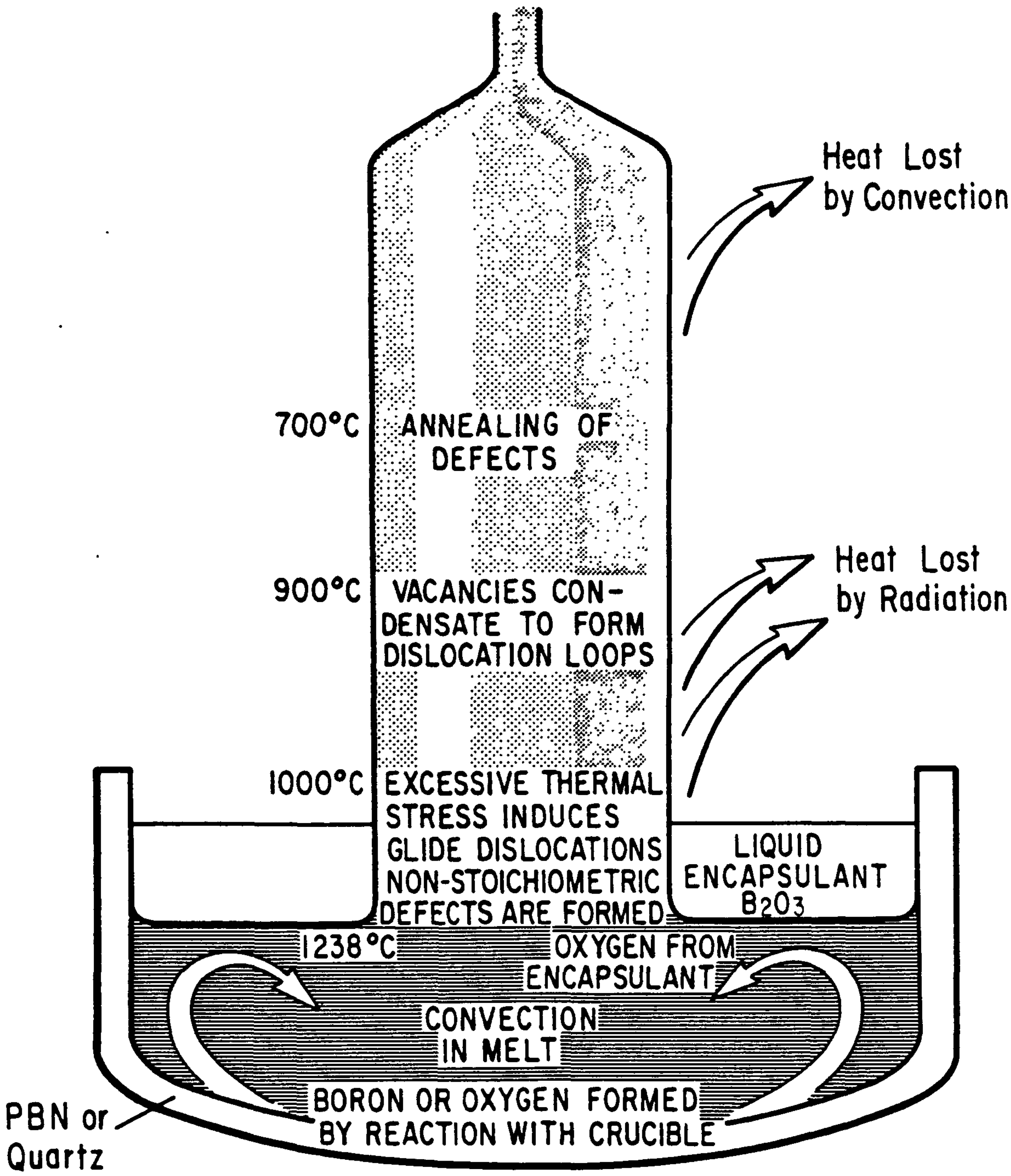


Figure 10. Defect evolution during LEC growth of GaAs.

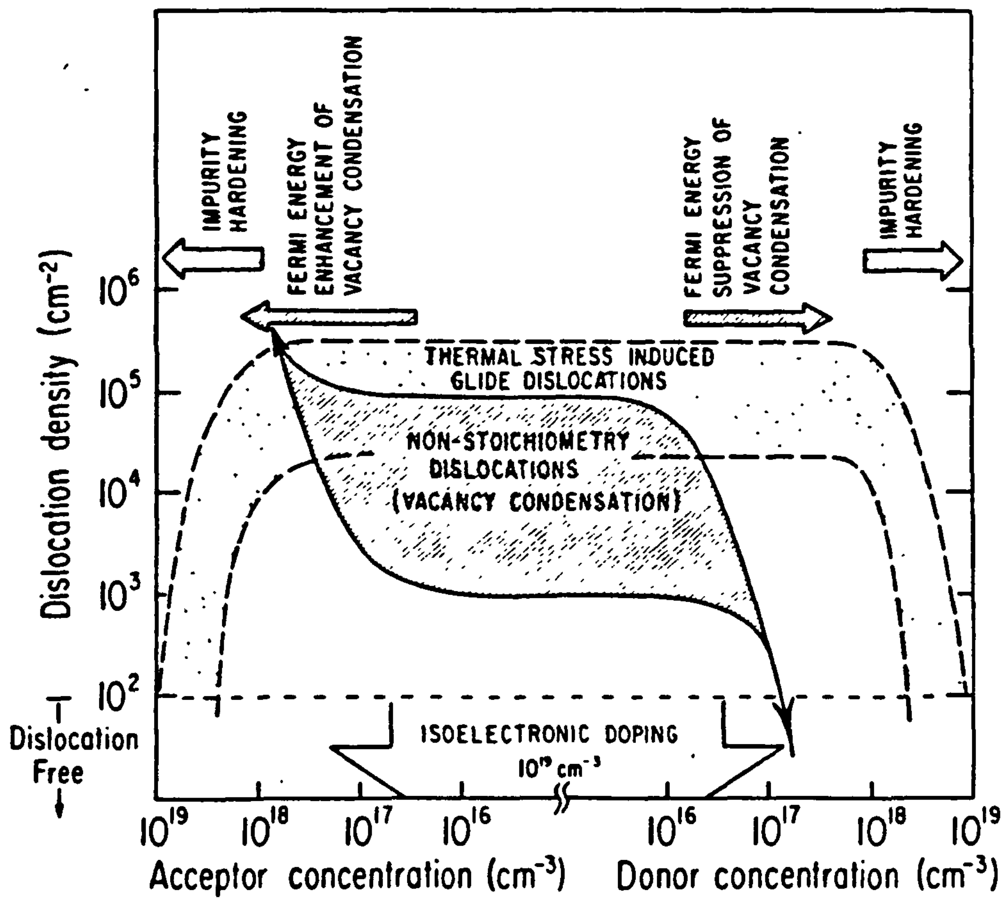


Fig. 11. Schematic diagram of the effects of impurities on the dislocation density in GaAs.

TABLE V

Approaches and Techniques Employed in the Characterization of GaAs

Method	Characterization	Comments
Standard		
Differential Etching and Interference Contrast Microscopy	Determination of microscopic growth velocities & layer thickness analysis of dislocations	Available in our Electronic Materials Group
Molten KOH Etching	Reliable determination of dislocation density on GaAs	Available in our Electronic Materials Group
Electron Probe	Determination of compensation of ternary compounds on macro- and microscale	Available at M.I.T. as Central Facility
Auger Analysis & Ion Milling	Compositional depth profiling	Available in our Electronic Materials Group
X-Ray Topography	Analysis of defect structure	Available in our Electronic Materials Group
Hall Effect	Determination of carrier concentration and mobility	Available in our Electronic Materials Group; high magnetic field arrangement available at National Magnet Lab (M.I.T.)
SEM-Cathodoluminescence (77-800K)	Profiling of radiative recombination centers	Available in our Electronic Materials Group
Current Transient Spectroscopy	Deep level characteristics	Under construction in our Group
Deep Level Transient Capacitance Spectroscopy (10-500K)	Deep level characteristics	Available in our Electronic Materials Group
Surface Photovoltage Spectroscopy	Surface state parameters	Available in our Electronic Materials Group
Novel		
Scanning IR Absorption	Carrier concentration and compensation ratio profiling	Available in our Electronic Materials Group
Laser Scanning Deep Level Spectroscopy	Deep level characteristics in microscale	Improved Version Will Be Available in our Electronic Materials Group

TABLE V (continued)

Method	Characterization	Comments
SEM-Electron Beam-Induced Current	Instantaneous profiling of diffusion length, lifetime, and surface recombination velocity	Available in our Electronic Materials Group
Derivative Surface Photovoltage Spectroscopy	Determination of optically active transitions, high precision absolute determination of composition of ternary compounds	Available in our Electronic Materials Group
Derivative Photocapacitance Spectroscopy	Optically active deep level characteristics	Available in our Electronic Materials Group
Transient Photocapacitance Spectroscopy	Optical determination of electron and hole traps	Available in our Electronic Materials Group
Laser Scanning Photovoltage	Microprofiling of deep levels	Available in our Electronic Materials Group

of the essential electronic properties of compound semiconductors on a macro- and micro-scale, and the study of the properties and phenomena related to device applications.

We propose to pursue further our research on the refinement of our characterization techniques with respect to sensitivity, spatial resolution, and specialized applications involving high-resistivity materials.

Photoluminescence Scanning Apparatus. The proposed photoluminescence (PL) apparatus will be based on the computer-controlled mapping of photoluminescence intensity developed only recently by Sumitomo Electric.[71] This system is shown schematically in Fig. 12.

Light emitted by an excitation source travels along the X axis and passes through a small hole in a mirror (R_1). It is then reflected by two mirrors (R_2 and R_3) and focused on the surface of the sample by a condenser lens (C_1). PL light is collected by the same lens, transmitted as parallel beams long the Y_2 axis, and reflected three times by the mirrors R_3, R_2 and R_1. It is finally focused by a condenser lens (C_2) on the entrance slit of a monochromator. When stage M_1 is moved horizontally by driving unit D_1, the excitation light can be scanned horizontally on the surface of the sample, and when stage M_2 is moved vertically by driving unit D_2, the excitation light can be scanned vertically on the surface. If both driving units are controlled propertly, the excitation light can be moved over the sample's surface in any pattern with no change in the collection efficiency of the PL light.

The PL measurements are performed at 4.2 K with the sample immersed in liquid helium in a metal Dewar. Luminescence is excited by 5145 Å emission from an Ar laser. One- and two-dimensional distribution maps of 1.49 eV photoluminescence obtained with this technique are shown in Fig. 13 a and b for a SI-GaAs wafer prior to and after the "whole ingot annealing" at 800°C

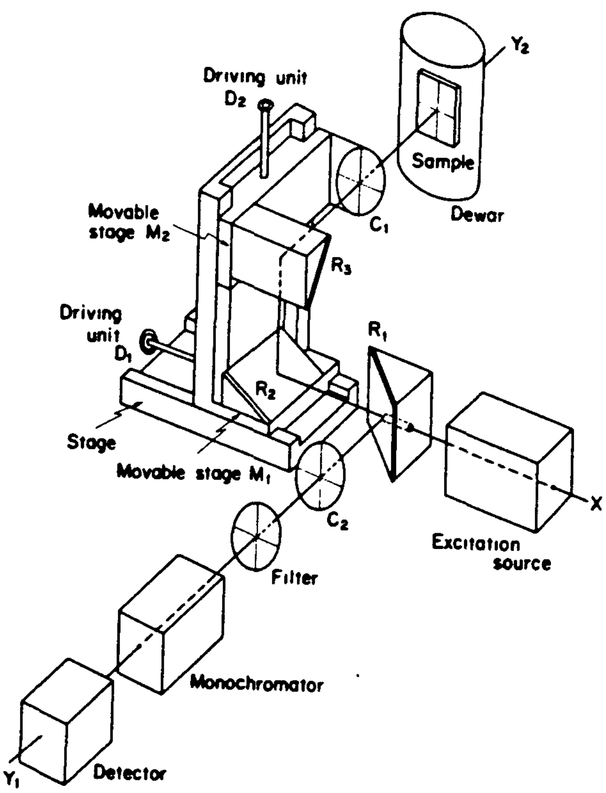
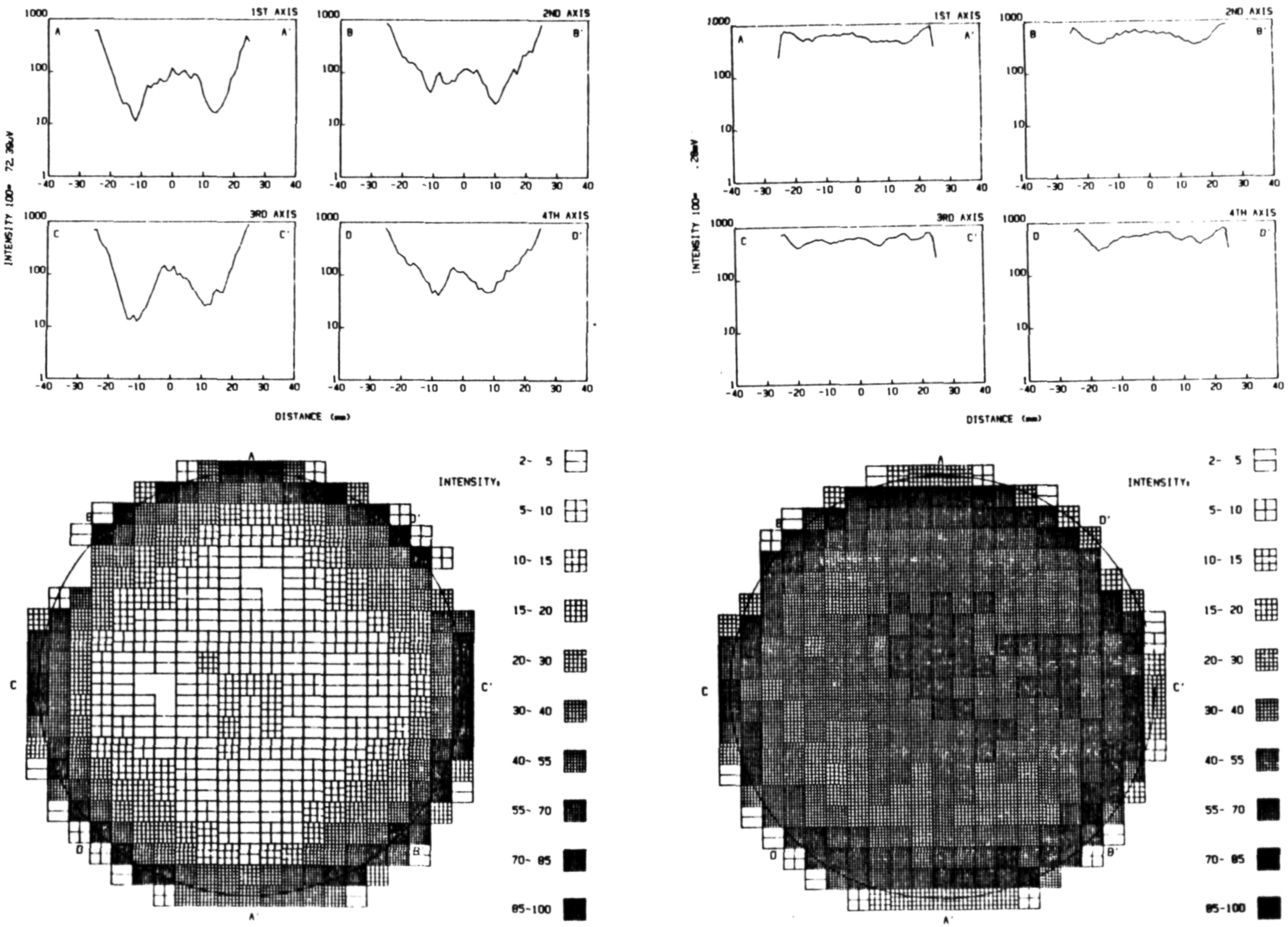


Figure 12. Schematic representation of photoluminescence scanning system developed by Sumitomo Electric Corporation.



(a)

(b)

Figure 13. Photoluminescence maps of GaAs wafers: (a) as grown, (b) homogenized by annealing at 800°C for 6^h (after ref. 72).

for 6^h. The efficiency of the method in assessing inhomogeneities is evident. It is also evident from Fig. 13 that annealing decreased the magnitude of spatial variations by as much as one order of magnitude. This remarkable homogenization of GaAs upon "whole ingot annealing" has attracted a great deal of interest. The photoluminescence scanning apparatus will be employed for detailed investigation of this phenomenon in GaAs crystals grown under precisely controlled stoichiometry and microscopic inhomogeneities.

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APPENDIX

Reprints and preprints of papers which appeared in the literature or were submitted for publication since our last annual report are attached. They provide a more detailed account of some of the work discussed in the test of the present report.