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FINAL REPORT

for the period

December 1, 1985 to November 30, 1988

CRYSTAL GROWTH OF DEVICE QUALITY GaAs IN SPACE

(NSG 7331)

February 1989

Submitted by

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I. SUMMARY

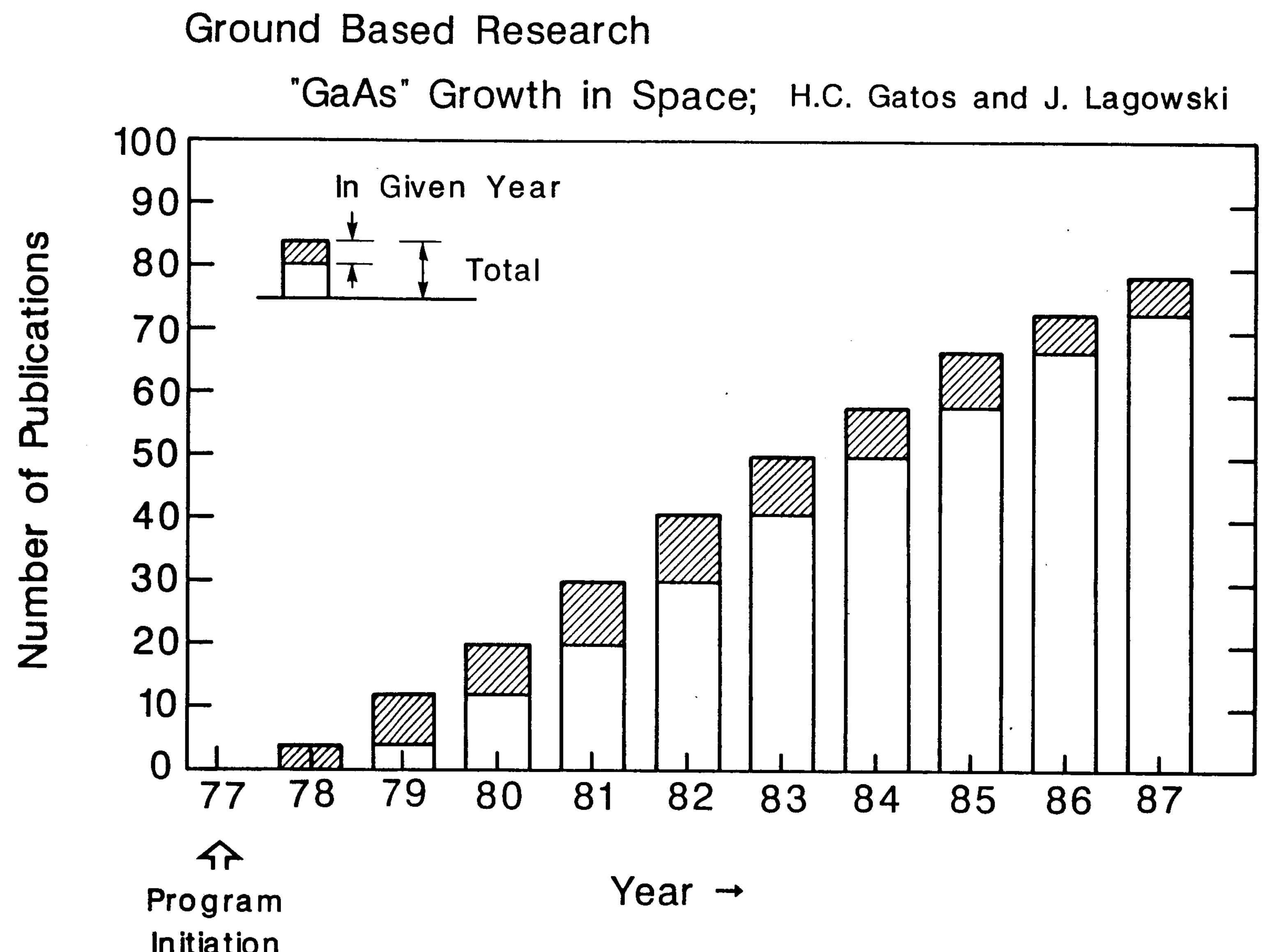
Our program on "Crystal Growth of Device Quality GaAs in Space" was initiated in 1977. The initial stage covering 1977-1984 was devoted strictly to ground-based research. By 1985 the program had evolved into its next logical stage aimed at space growth experiments; however, in accord with the new philosophy implemented by NASA after the Challenger disaster, the program has been maintained as a ground-based program awaiting activation of experimentation in space.

The overall program has produced about eighty original scientific publications (see Figure 1 and the list of publications) on GaAs crystal growth, growth-property relationships, defect engineering, crystal characterization, and new approaches to space processing. The publications completed during the last three years are listed as Part II. Their key results are outlined below and discussed in detail in about twelve selected publications enclosed with this report. The work has also resulted in five Ph.D. theses completed during the last three years.

II. DEFECT ENGINEERING IN GaAs

Modern trends in GaAs emphasize the engineering of native defects as a means for tailoring materials properties for device processing. Our comprehensive study has demonstrated the positive role of native defects in achieving new electronic properties and a new response to processing steps.

In the experimental approach we took advantage of nonstoichiometric crystal growth, rapid cooling rates (followed



Ground Based Research
"GaAs" Growth in Space; H.C. Gatos and J. Lagowski
100
90
80
70
60
50
40
30
20
10
0
Number of Publications
In Given Year
Total
77
78
79
80
81
82
83
84
85
86
87
Program
Initiation
Year →

Figure 1.

by programmed annealing) and changes in the Fermi energy. A proper combination of these factors makes possible the differentiation between commonly recognized growth factors such as growth velocity (pulling rate) and heat transfer in the melt, and newly recognized factors, i.e., melt stoichiometry and the rate of cooling.

Effects of Stoichiometry. The finite existence region is the dominant factor accounting for the increased defect concentration in crystals grown from nonstoichiometric melts. The results of our MIT group on Horizontal Bridgment (HB) growth of GaAs with the melt stoichiometry varied by changing the temperature of the arsenic source, T_{As}, have shown that the concentratiopn of the dominant midgap donor, EL2, increases with increasing partial pressure over the melt (i.e., with increasing arsenic atom fraction in the melt). Similar EL2 behavior was observed in LEC-grown GaAs. Only recently we have extended the nonstoichiometry range to the gallium-rich region, with an arsenic atom fraction below 0.47. As shown in Fig. 2, this range corresponds to the disappearance of EL2 and also to the appearance of a double acceptor defect with concentration increasing with increasing gallium fraction (see Fig. 3). The double acceptor apparently corresponds to the gallium antisite defect Ga_{As}.

Thermal History and ITC Treatment. In the course of the investigation of post-solidification processes we found that fast cooling of GaAs crystals leads to a significant decrease in the EL2 concentration (see Fig. 4). This finding led to the

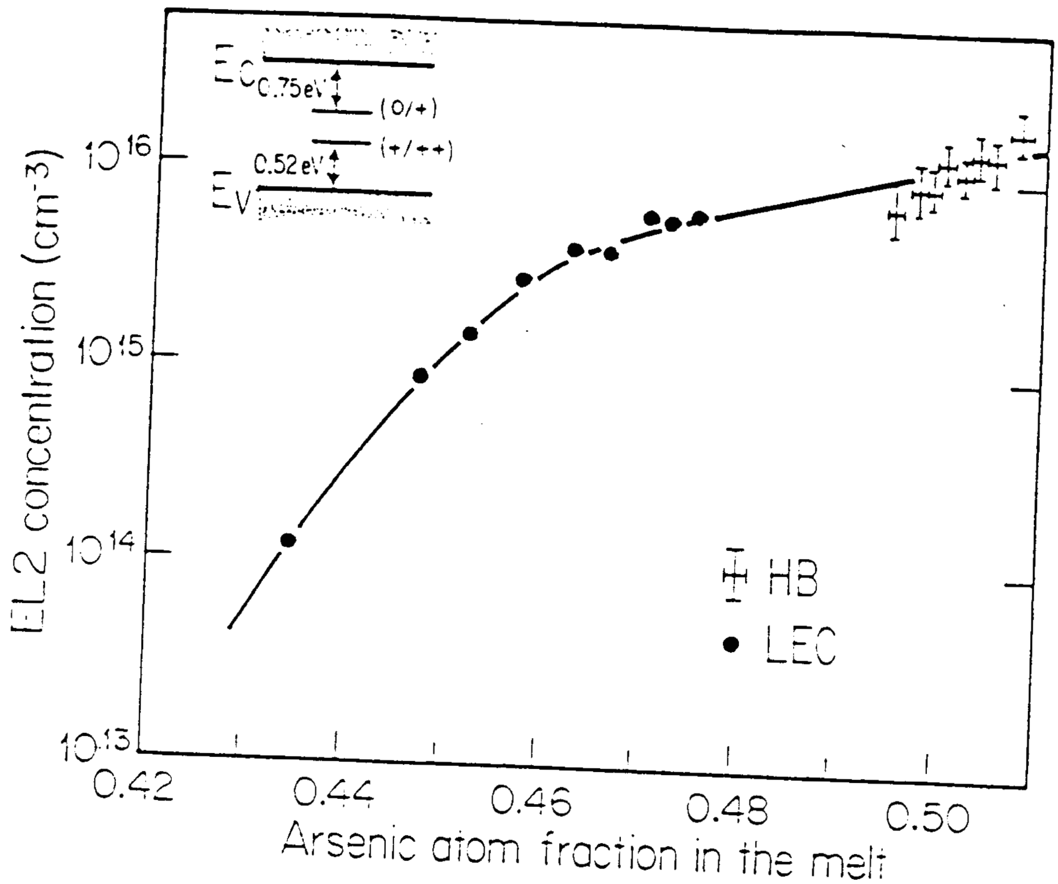

Ec
0.75 eV
(0/+)
(+/++)
0.52 eV
Ev
10^{16}
10^{15}
10^{14}
10^{13}
EL2 concentration (cm^{-3})
HB
LEC
0.42
0.44
0.46
0.48
0.50
Arsenic atom fraction in the melt

Fig. 2.

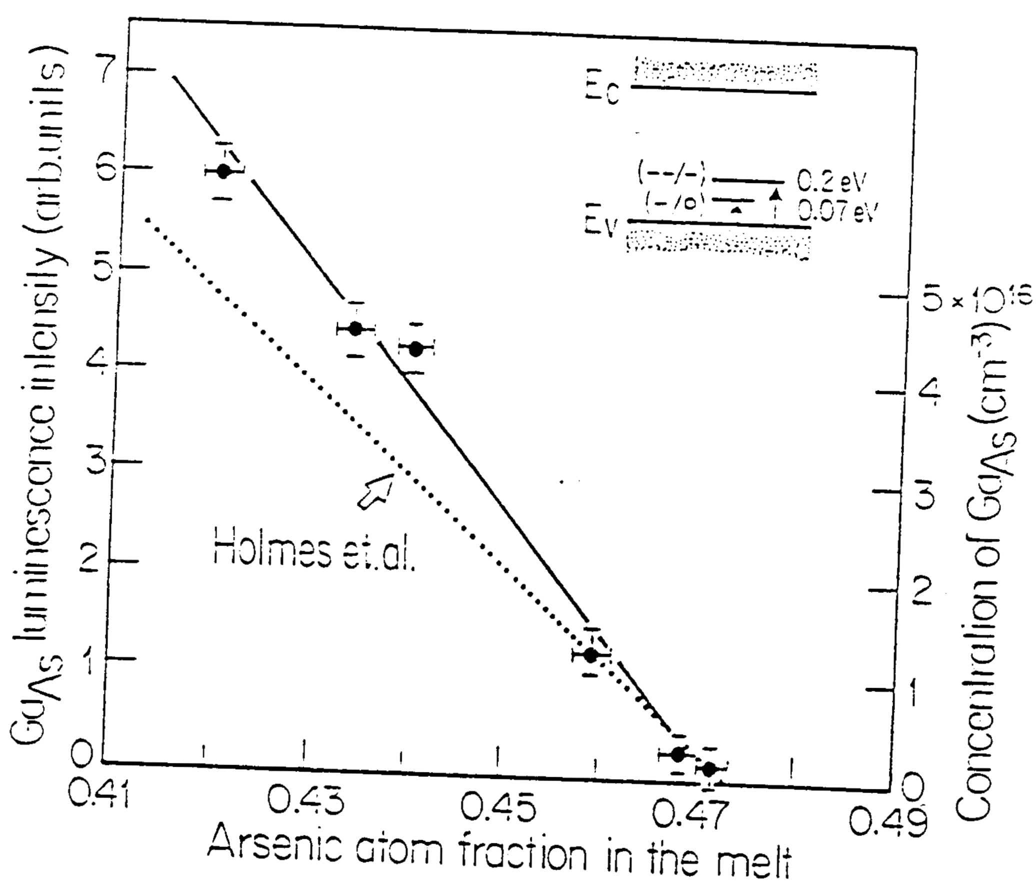

Ec
(--/-)
0.2 eV
(-/o)
0.07 eV
Ev
Ga_{As} luminescence intensity (arb.units)
7
6
5
4
3
2
1
0
5×10^{16}
4
3
2
1
0
Concentration of Ga_{As} (cm^{-3})
Holmes et.al.
0.41
0.43
0.45
0.47
0.49
Arsenic atom fraction in the melt

Fig. 3.

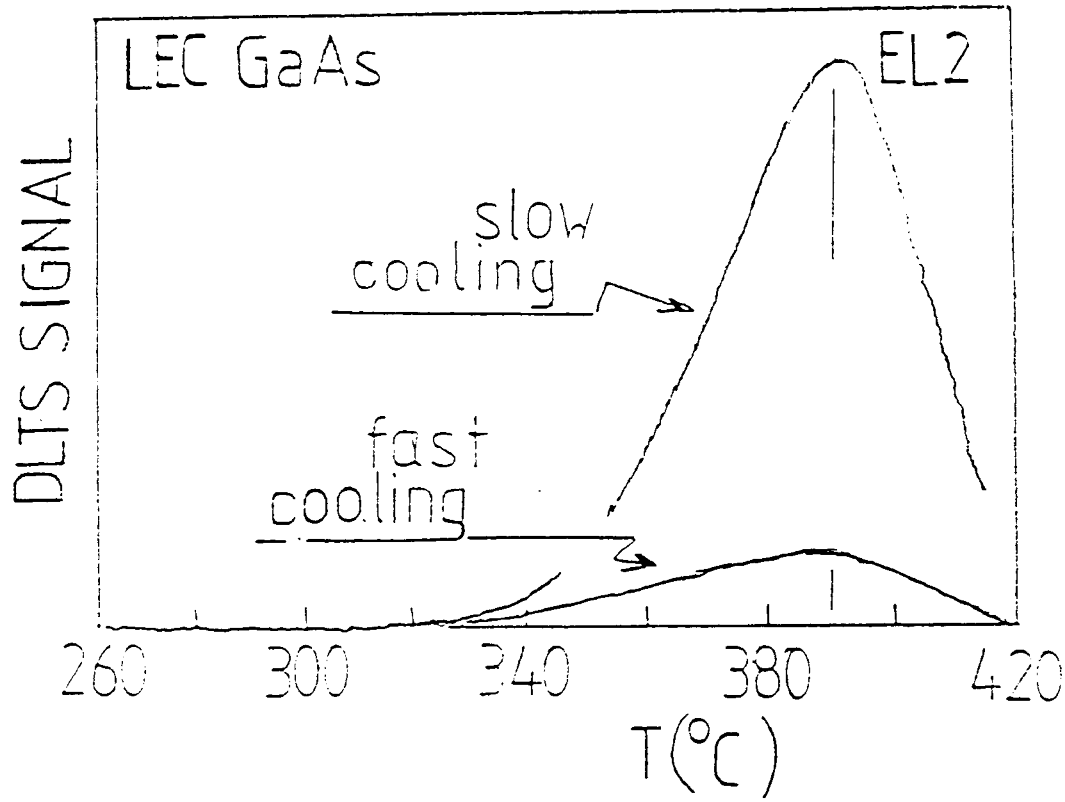

LEC GaAs
EL2
DLTS SIGNAL
slow cooling
fast cooling
260
300
340
380
420
T(°C)

Figure 4.

development of the ITC treatment which essentially wipes off the thermal history of the crystal and yields virtually EL2-free material (see Figs. 5 and 6). The ITC treatment involves annealing at a very high temperature of about 1200°C in equilibrium arsenic ambient (preventing arsenic loss) and followed by rapid cooling to room temperature. The transformation of ITC can also be achieved by incorporating rapid cooling into an actual crystal growth process. The high cooling rates and, thus, rapid transition through the 900-700°C range prevents the formation of the native midgap donor EL2. Subsequent annealing in the 750-900°C range leads to the controllable formation of EL2 (see Fig. 6); its concentration depends on the annealing temperature and time. It is a very important useful feature of the undoped ITC GaAs that its resistivity passes through a maximum during annealing (see Fig. 7). The maximum value is close to the theoretical resistivity limit for single crystalline GaAs at 300 K $\rho_{max} \approx 10^9$ Ωcm. The resistivity behavior of ITC GaAs during annealing implies that deep acceptors are converted to EL2 donors. Our rapid cooling experiments have identified three temperature regions corresponding to different EL2 behavior (see Fig. 8):

1. Very high temperature range (1230°C-1050°C) corresponds to EL2 annihilation;
2. Medium temperature range (7000°C-1050°C) involves EL2 formation;
3. Low temperature range (below 600°C-700°C) where EL2 is not yet formed in melt-grown crystals but some of the primary

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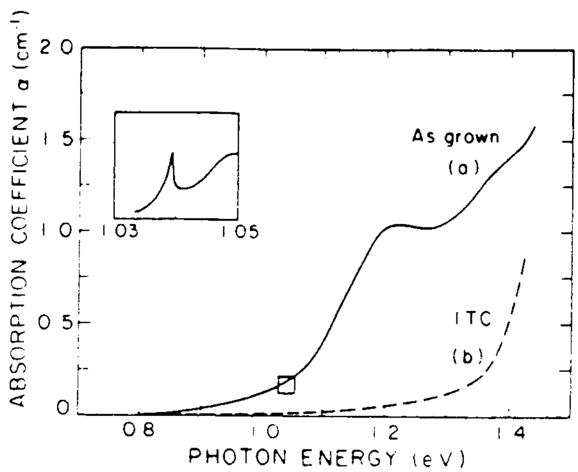

FIG 5 Low-temperature (4.2 K) optical absorption spectra of n-type melt-grown GaAs in the spectral range of photoionization and intracenter transitions of the occupied EL2 donor. (a) As-grown crystal. (b) ITC crystal, i.e., after annealing at 1200 °C for 16 h and quenching. Note elimination of EL2 absorption

Figure 5.

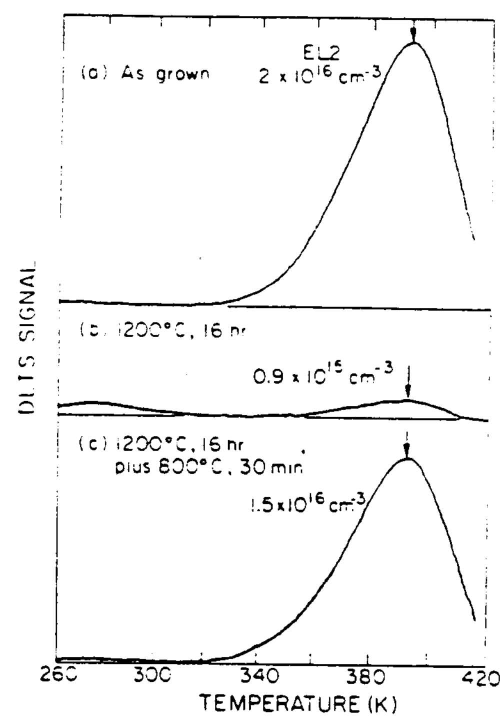

FIG. 6 DLTS spectra of n-type melt-grown GaAs. (a) As-grown crystal. (b) after annealing at 1200 °C for 16 h and quenching to room temperature, and (c) after additional annealing at 800 °C for 20 min. Gates t_1/t_2 = 5 ms/10 ms. The DLTS signal was normalized to reflect the deep level concentration.

Figure 6.

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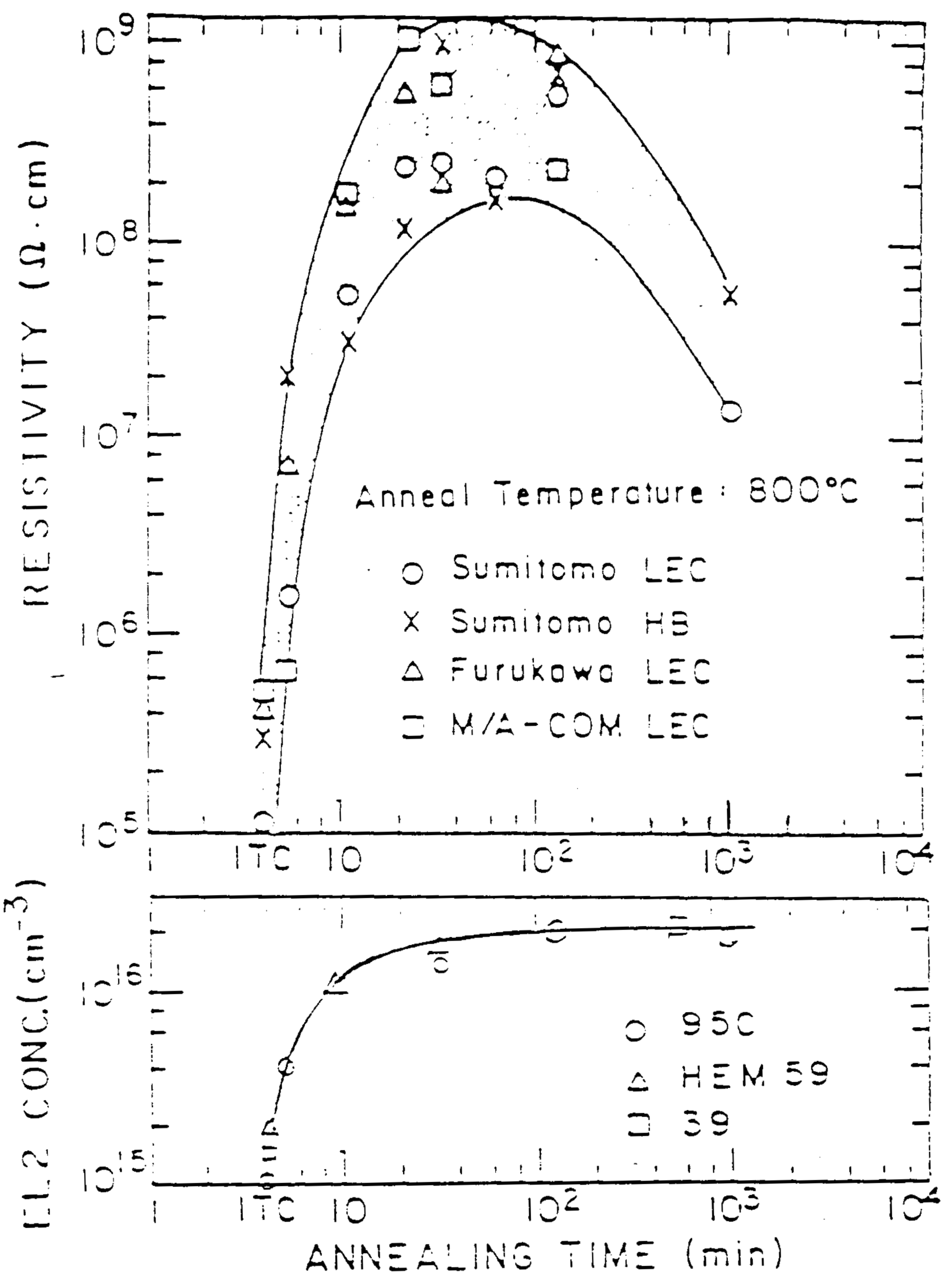

Figure 7.

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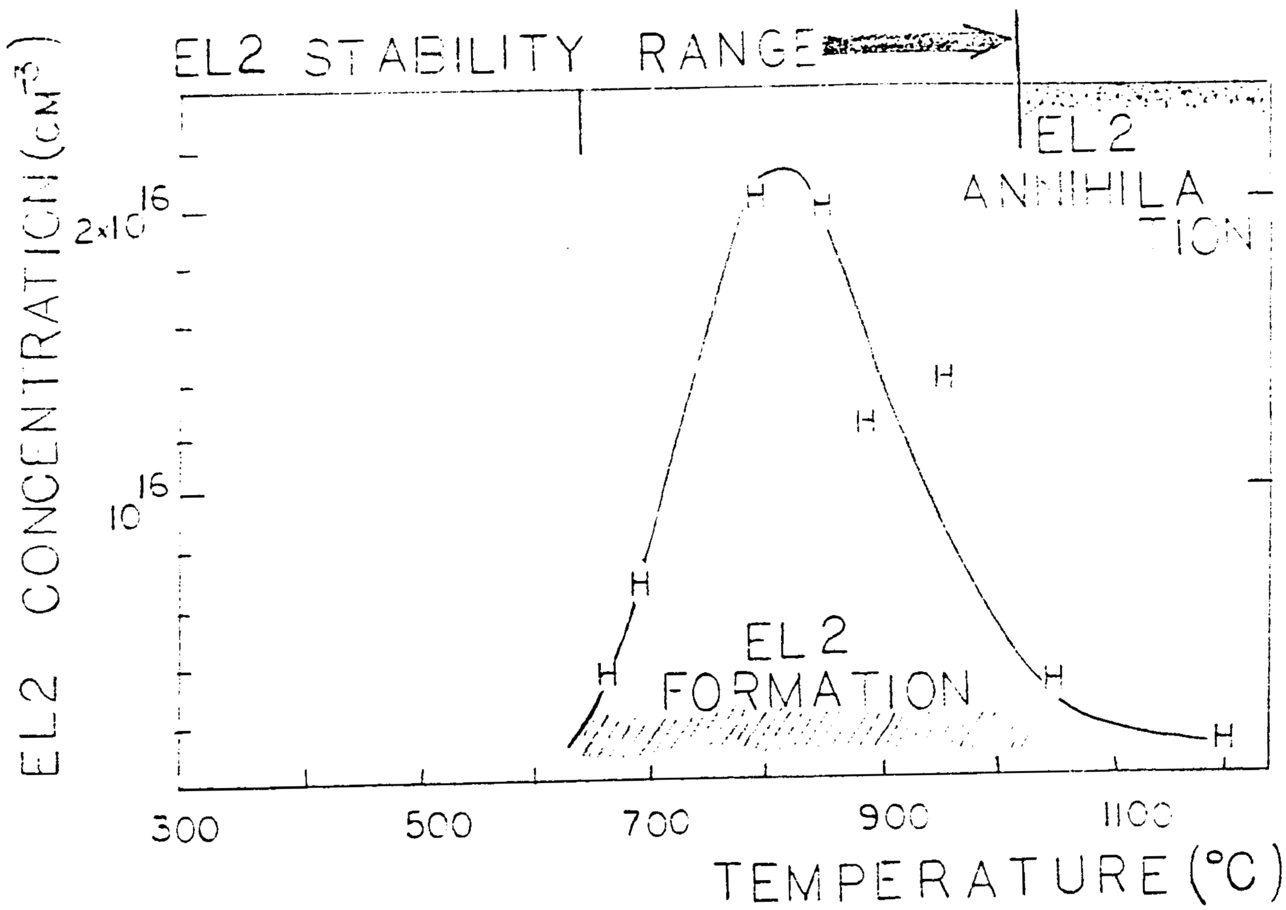


Figure 8.

defects observed in ITC GaAs do anneal out.

Fermi Energy. Defect interaction paths in GaAs are to a large extent controlled by the Fermi energy in the crystal cooled after growth. Our study has shown that the Fermi energy can inhibit (or enhance) formation of EL2 as well as the formation of dislocations. These results were discussed in detail in our previous reports.

Nature of Key Defects in GaAs; EL2 Problem. In a series of carefully designed experiments, based on annealing-rapid cooling procedures for the controlled creation and annihilation of EL2, we identified basic rules which must be obeyed in any atomic model of the EL2 defect in order for such a model to be compatible with experiment. The relevant findings are as follows: (1) the binding energy of the EL2, determined for the first time, is (3.6±0.5) eV; (2) the EL2 defect is neutral when its midgap level is occupied, and no EL2 acceptor or donor levels exist in the upper half of the energy gap; (3) four EL2 manifestations are all associated with one and the same defect, namely, the double donor levels (0/1+ at E_V+0.52 eV and 1+/2+ at E_V+0.75), the 1.039 eV zero phonon absorption line, the optically detected double nuclear resonance signal, and the optically induced transfer to the metastable state.

The major rules corresponding to the above results are: (1) very high EL2 stability, (2) charge states of EL2, and (3) intimate interdependence of four EL2 manifestations. Rules (1) and (2) are not obeyed by an intensively pursued model based on the weakly interacting pair As_{Ga}-As_I. Thus, the theoretical model yields a binding energy less than 1 eV, and an EL2 charge state 1+/2+, and also involves the presence of a shallow EL2 donor. Rule (3) unifies the EL2 properties, but it also raises a series of questions with regard to all EL2 models proposed thus far.

Reexamination of the Gallium Antisite Levels. Two energy levels at 77 meV and 200 meV above the valence band are commonly found in GaAs grown from gallium-rich melts. These levels attracted a lot of attention as native acceptors potentially involved in a compensation scheme in semi-insulating (SI) GaAs. Based on circumstantial evidence, Yu et al proposed the isolated gallium antisite, Ga_{As}, double acceptor defect to account for the presence of both levels. This appears to be the most widely accepted interpretation at the present time.

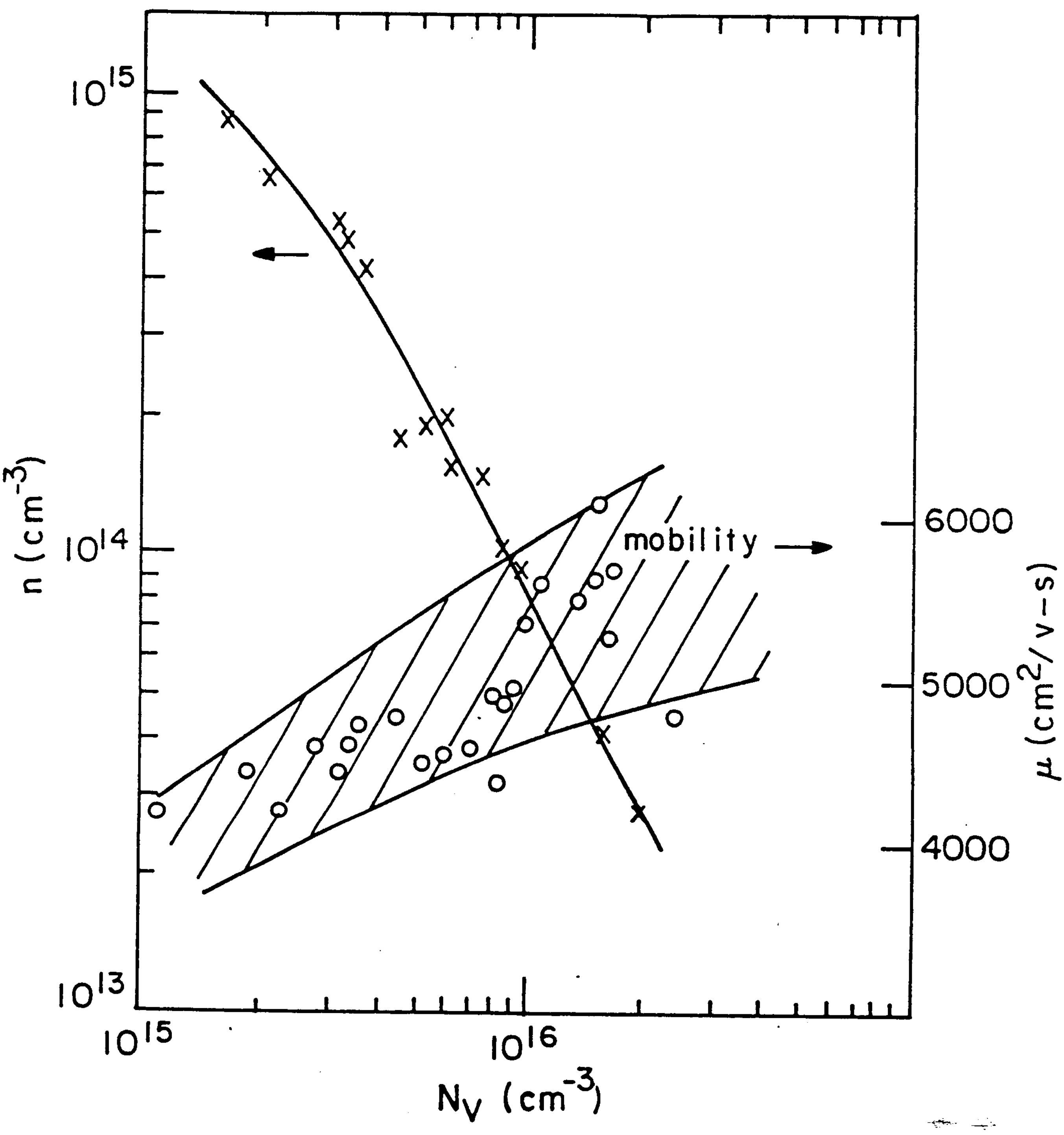
In the course of the accepted study of a large number of n- and p-type ingots grown from the melts of different stoichiometry we have carefully reexamined all properties of the levels in question. The results of combined measurements of photoluminescence, photocapacitance spectroscopy, and standard DLTS clearly demonstrated the inadequacy of previous studies fragmentary in scope. They also exposed a serious interpretational conflict between photoluminescence data alone and the quantitative results of transient capacitance spectroscopy. Thus, the photoluminescence characteristics (unique temperature and excitation dependences in n- and p-type samples) alone are in good agreement with a model attributing both levels to two charge states of one and the same native defect. Dependence of stoichiometry is also consistent with the Ga_{As} defect. The first systematic DLTS measurements on p-type crystals confirmed the presence of two hole traps with activation energies 77 meV and 200 meV, respectively. The concentration of both traps indeed increased with increasing Ga/As ratio in he growth melt. However, the concentration at the 200 meV level was always about one order of magnitude smaller than that of the 77 meV level. This result apparently rules out the widely accepted interpretation.

III. ROLE OF CHEMICAL INTERACTIONS IN GaAs GROWTH

Gettering of Donor Impurities by Vanadium. Doping of GaAs with vanadium has recently attracted a great deal of attention as an "apparent" means for obtaining semi-insulating GaAs with improved thermal stability. In a series of HB and LEC growth experiments we have demonstrated the vanadium does not introduce any midgap level and thus it cannot directly contribute to a compensation mechanism in SI-GaAs. This finding generated a very important question: How can vanadium facilitate the growth of semi-insulating GaAs without any effect on compensating midgap levels? The answer to this question was provided by our discovery of the gettering of donor impurities by vanadium. Thus, we found that vanadium added to the growth melt gettered silicon and sulfur through chemical interaction. As a result, vanadium doping caused the reduction of shallow donor concentration below a critical value required for achieving semi-insulating material.

According to our findings, the addition of vanadium to the growth melt plays a role similar to that of oxygen, i.e., it purifies the crystal with respect to donor impurities. As shown in Fig. 9, the electron concentration decreases and the electron mobility increases with increasing the concentration of vanadium added to the GaAs melt, N_V. Thermodynamic analysis of the gettering reactions showed that the purification is most likely caused by formation of stable vanadium compounds (V_3Si and VS) in the melt and their rejection at the solidification front. Employing this gettering mechanism we achieved consistent growth of semi-insulating GaAs by the HB and LEC methods. The compensation mechanism was controlled by the midgap EL2 level, while vanadium doping reduced the concentration of shallow donors below the concentration of residual acceptors.

Enhancement of Si Contaminaton by Boron. Boron is a common constituent of GaAs growth systems, and it originates from a pyrolytic boron nitride (pBN) boat or crucible and/or B_2O_3 encapsulant. As a group III element B is isoelectronic with Ga and is not expected to be electrically active in GaAs. We found, however, that in HB growth, boron significantly increases the concentration of free electrons, making it impossible to grow high resistivity crystals. In LEC crystals the presence of boron had no apparent effects on electrical properties. Searching for clarification of this unusual behavior, we found that boron enhances silicon contamination in GaAs crystals grown by the Bridgman method. Systematic analysis of crystals intentionally doped with B combined with thermodynamic considerations of the Ga-As-B-Si-O system has led to a successful explanation of the



10^15
10^14
10^13
n (cm^-3)
mobility
6000
5000
4000
μ (cm^2/v-s)
10^15
10^16
N_V (cm^-3)

Fig. 9.

contamination mechanism. A key role is played by the reaction between SiO in the growth ambient and B in the GaAs melt. This reaction controls Si concentration in the melt and leads to a characteristic two-thirds power dependence between Si and B concentration in the grown crystals. A dissociation of pyrolytic boron (pBN) boat during growth is identified as the unintentional source of B with profound consequences for growing semi-insulating GaAs by the pBN-Bridgman method.

IV. NEW SEMI-INSULATING MATERIALS

In our pursuit of intentionally doped semi-insulating III-V compounds, we have examined the question of Ti-related deep levels in GaAs and InP. Thus, we have carried out a systematic study of the effects of Ti doping on the electrical and optical properties of GaAs and InP. Utilizing deep level transient spectroscopy, Hall effect, photoconductivity, and optical absorption measurements, we found that Ti introduces two deep levels in GaAs at E_C-0.28 eV and E_C-1.00 eV, which were identified as the Ti^{3+}/Ti^{2+} acceptor and Ti^{4+}/Ti^{3+} donor, respectively. (See diagram in Fig. 10.) In InP the Ti^{4+}/Ti^{3+} donor was found near the midgap aa E_C-0.63eV, while the Ti^{3+}/Ti^{2+} acceptor was found to lie within the conduction band. The midgap position of the donor offered a means for producing semi-insulating InP based on doping with Ti to compensate shallow acceptors. This was the first case where a semi-insulating III-V compound had the compensation mechanism based on a deep donor impurity. In view of the very low diffusivity of Ti, this new semi-insulating InP should exhibit greater thermal stability than

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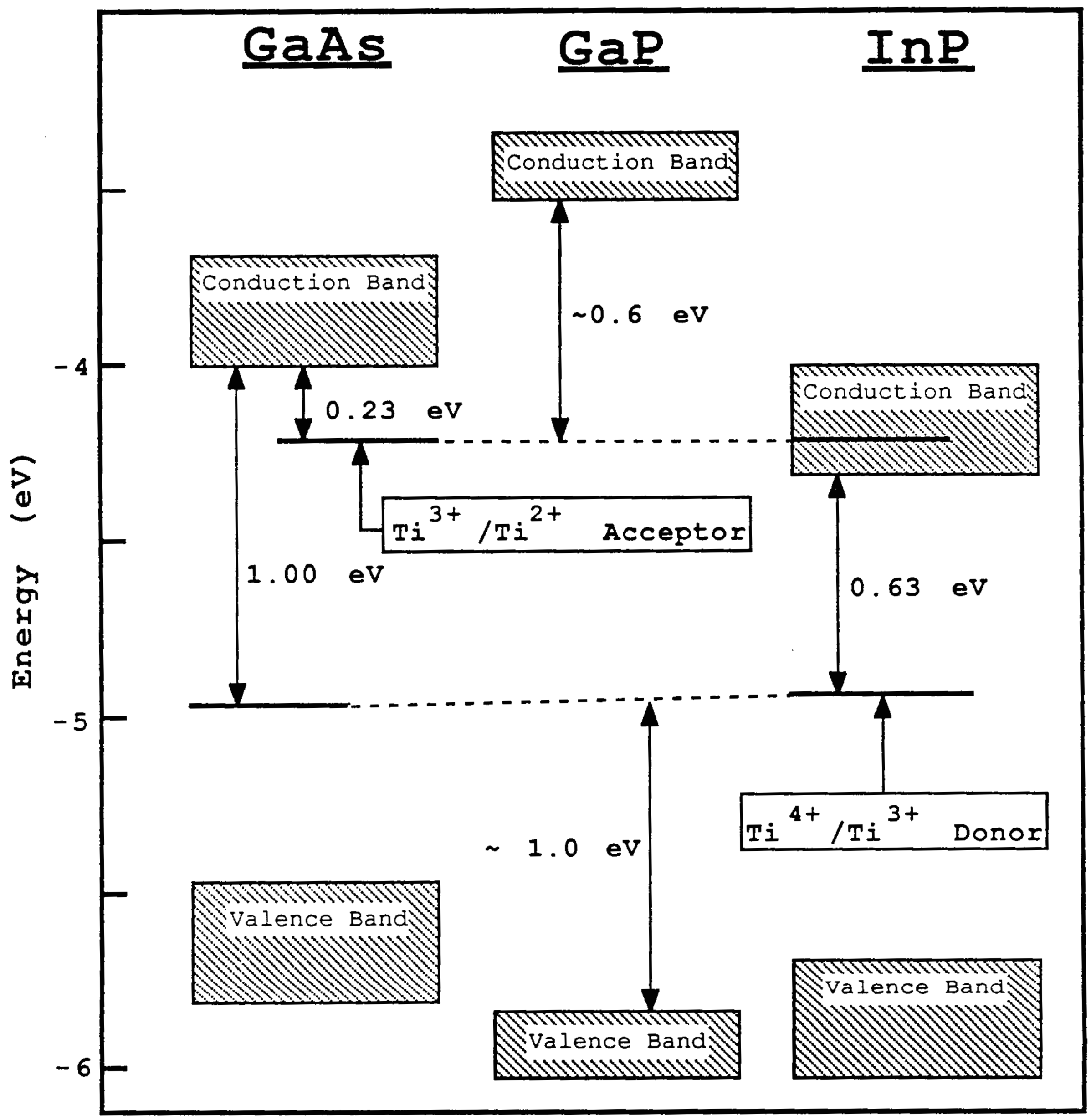


Fig. 10

presently available Fe-doped SI InP.

V. GROWTH OF GaAs IN PARTIAL CONFINEMENT

Under zero gravity, melts acquire a shape corresponding to the minimum surface energy. Unconfined melts, generally preferred to avoid contamination, acquire a spherical shape, which is not suitable for directional single crystal growth. In previous space growth experiments, cylindrical containers were employed. This confinement, however, leads to major problems in controlling the melt stoichiometry and interactions between the melt and the crucible. In order to overcome these problems, we have proposed a novel "partially confined configuration." Fundamental aspects of this configuration were verified experimentally employing the simplest case of a triangular prism growth ampul.

This completely novel growth configuration should permit growth in space with

- controlled melt stoichiometry during the growth process;
- cylindrical shape of growing crystal;
- provisions for accommodation of volume expansion during solidification;
- minimized contact between the melt and the container.

A series of ground-based growth experiments was designed to answer the following questions:

1. Is growth under partial confinement feasible in 1G conditions?
2. What are the key technical problems which have to

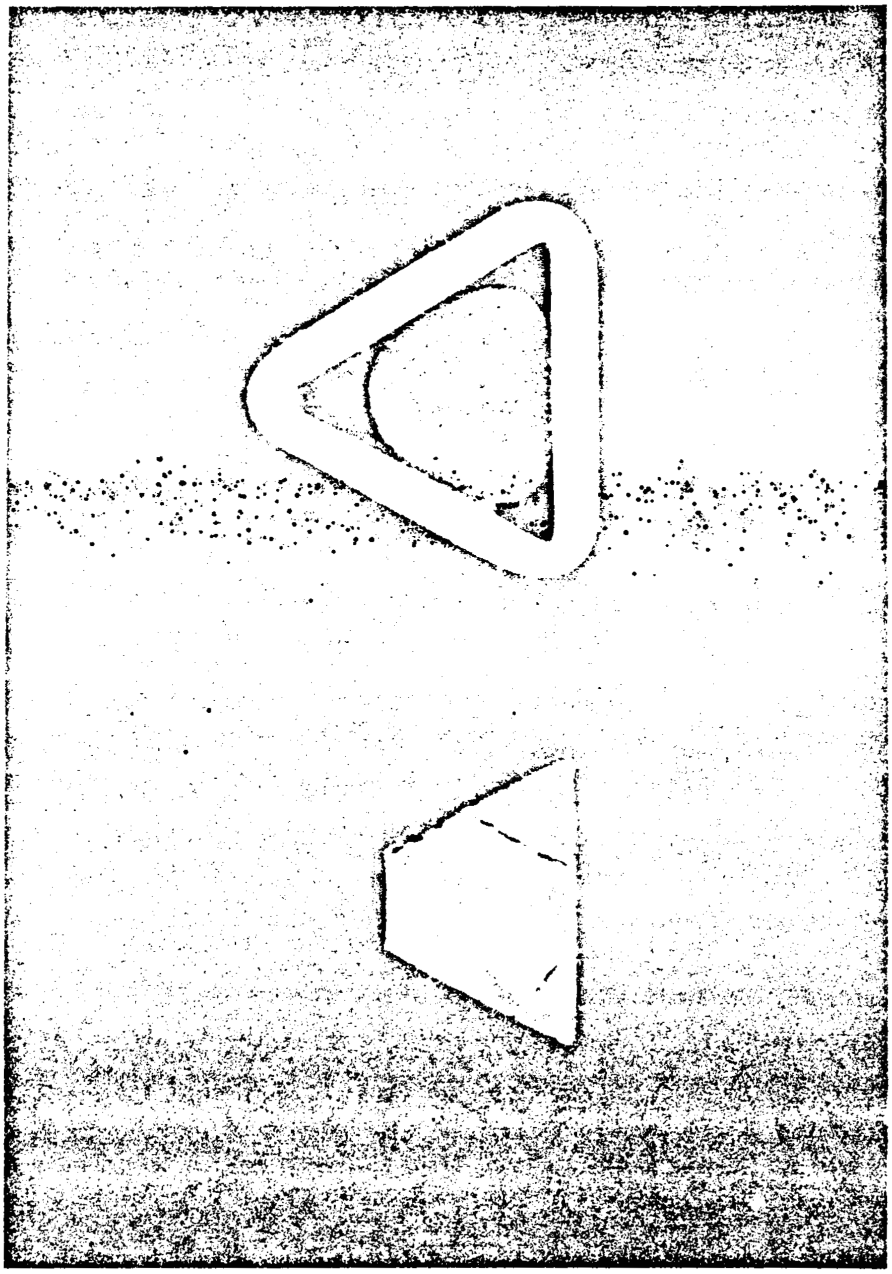
be overcome?

3. Does the partial confinement lead to new phenomena not observed in a standard configuration?

4. What are the electronic properties and defect structure of GaAs crystals grown under partial confinement?

Our investigation is in progress, and experiments still await completion. Nevertheless, the experiments already performed make it possible to answer questions 1-4 with reasonable confidence. Crystals about 3 mm in cross section and about 8 cm long were grown under stoichiometric conditions (arsenic source temperature $T_{As} \approx 617°C$) in a triangular quartz prism ampul placed in a 3-zone horizontal furnace.

Question 1. Employing a triangular prism ampul we achieved growth of GaAs crystals with a shape very close to our theoretical predictions. A cross section of such a crystal and the ampul is shown in Fig. 11b. Successful partial confinement is manifested by rounded corners of the crystals and empty channels along the triangular prism corners. In spite of gravity the melt and solidified crystal assumed a rounded shape due to the surface tension effects. A case corresponding to Fig. 11b was observed when GaAs did not wet the inner surface of the quartz ampul especially treated with sandblasting and cleaning. When wetting took place, the melt forced by gravity assumed a shape the same as that of an ampul. The ampul and the crystal both cracked during solidification as a result of volume expansion. A cross section of such a GaAs crystal is shown in Fig. 11a. Experiments were repeated about a dozen or so times,



(a) (b)

Fig 11

and the crystal shape controlled by the surface tension was always achieved when wetting did not take place. The answer to Question 1 is thus highly positive. GaAs growth in partial confinement is indeed possible even on the ground. Projection of the results to the microgravity environment leads us to the conclusion that the growth of cylindrical GaAs crystals with circular cross sections should be possible in space using partial confinement.

Question 2. As pointed out above, the wetting between the GaAs melt and the ampul was identified as a critical issue. For the typical pBN and quartz container, this problem can be overcome by designing a proper treatment of the ampul surfaces.

Question 3. Photographs of two representative GaAs crystals grown in triangular prisms shown in Fig. 12 reveal characteristic oscillations of the shape. For clarity these oscillations are also shown schematically in Fig. 13. The regions of larger diameter contain microscopic inhomogeneities forming a striation-like pattern of equally spaced lines separataed by about 16 μm. Such striation pattern is revealed on the crystal cross section by differential etching. The origin of this behavior is currently being investigated. Two possibilities, i.e., capillarity-like instabilities and surface tension-related instabilities, are being considered as possible sources of this unusual behavior.

Question 4. Crystals were characterized using Hall effect measurements, deep level transient spectroscopy, and photoluminescence microprofiling. Carrier concentration,

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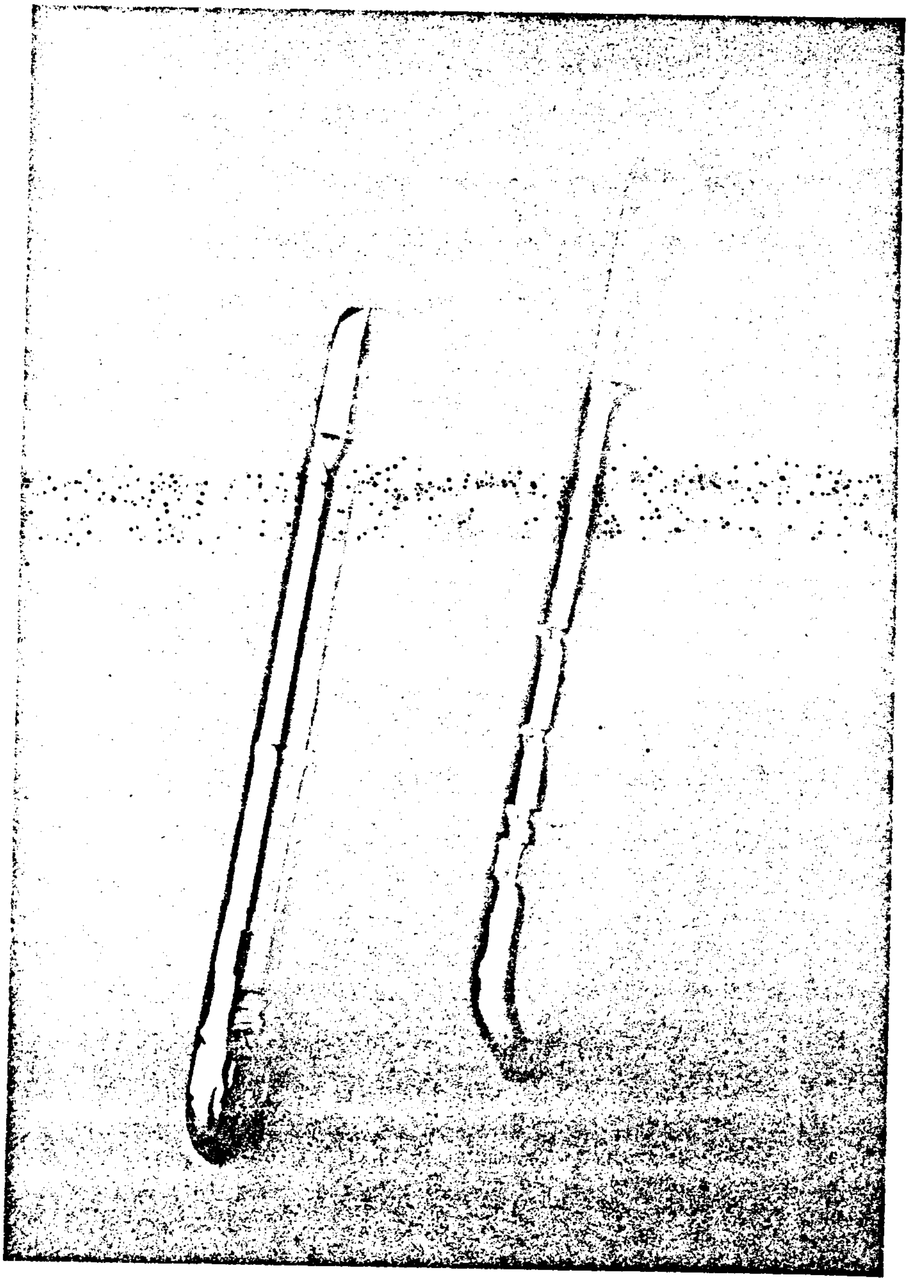

Fig 12

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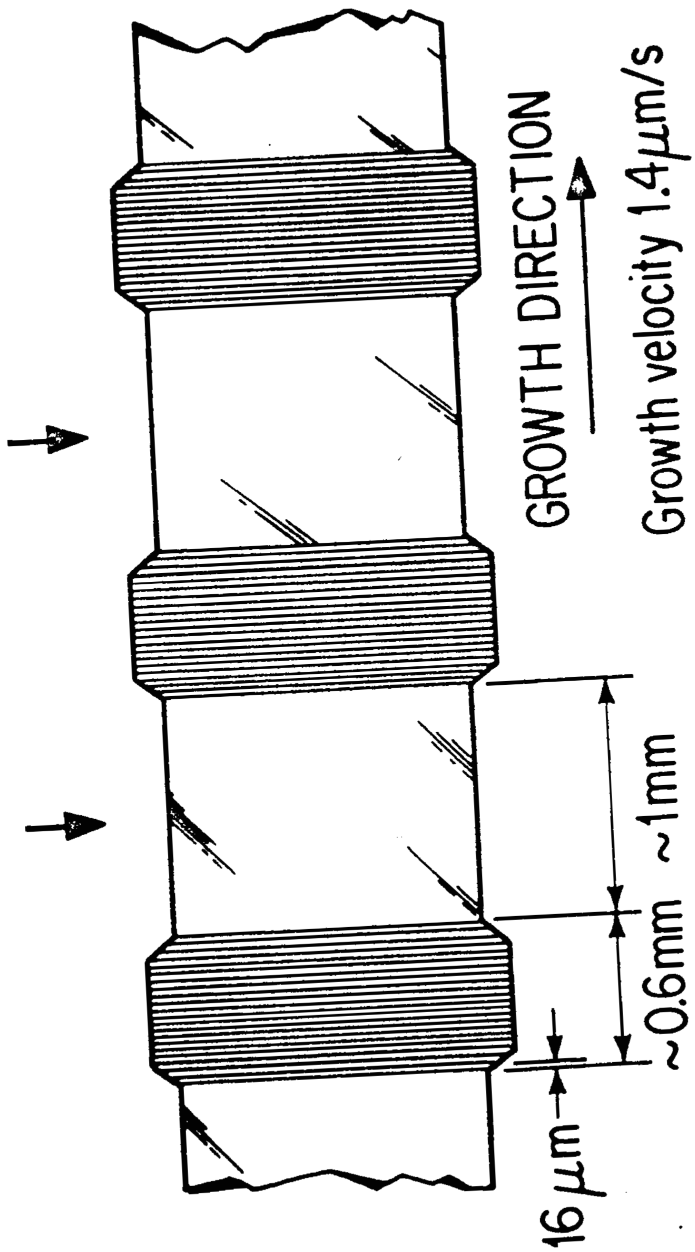

Fig. 13

mobility, and deep level characteristics were found very similar to those in GaAs grown by the conventional HB method. However, the low temperature (4.2 K) photoluminescence microprofile (Fig. 14) along the growth direction revealed a striation pattern which corresponds to the crystal shape inhomogeneities shown in Fig. 13. As pointed out above, the origin of these unusual inhomogeneities is currently being investigated.

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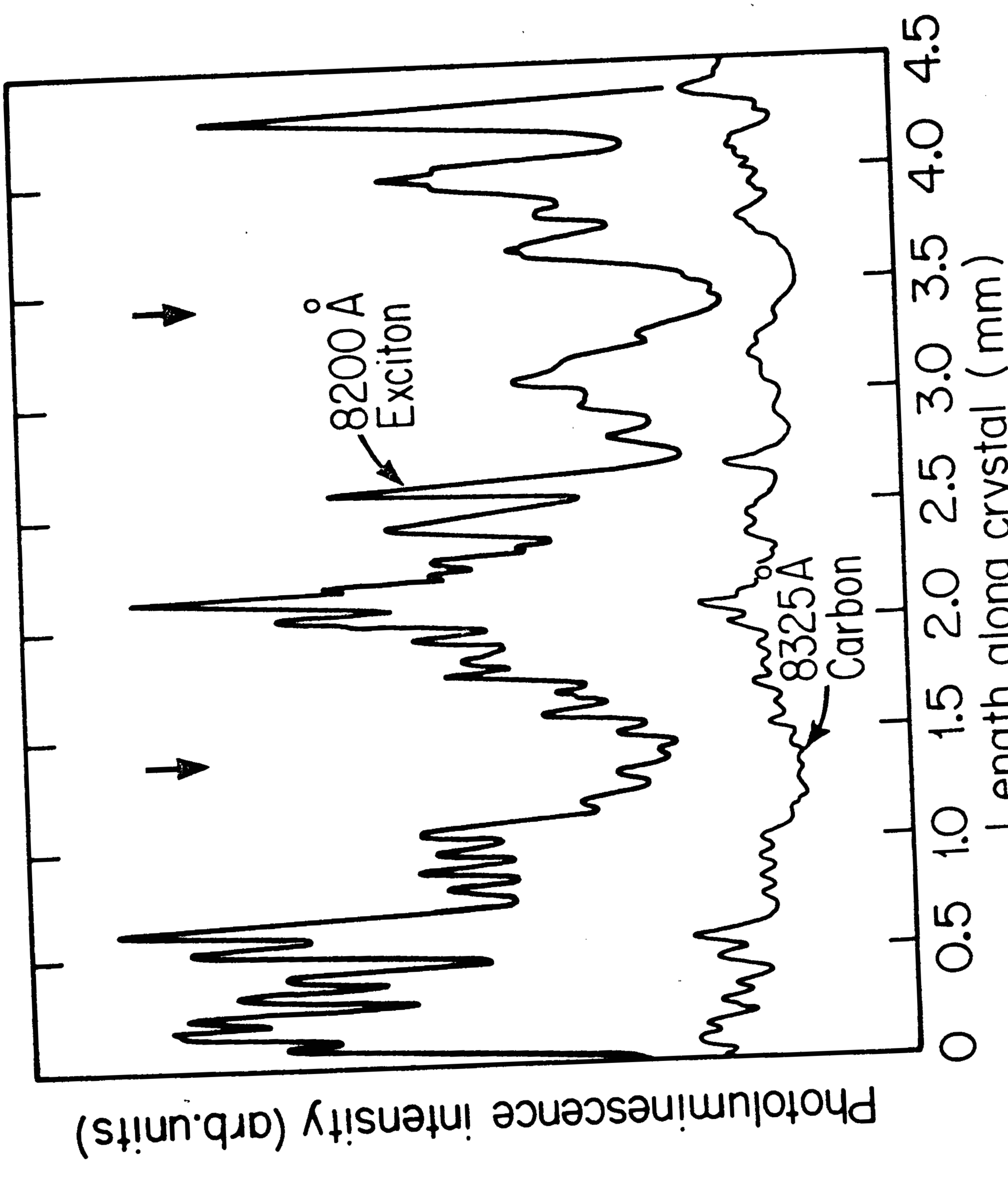

Fig. 14

Part I - Period 1878-1985

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