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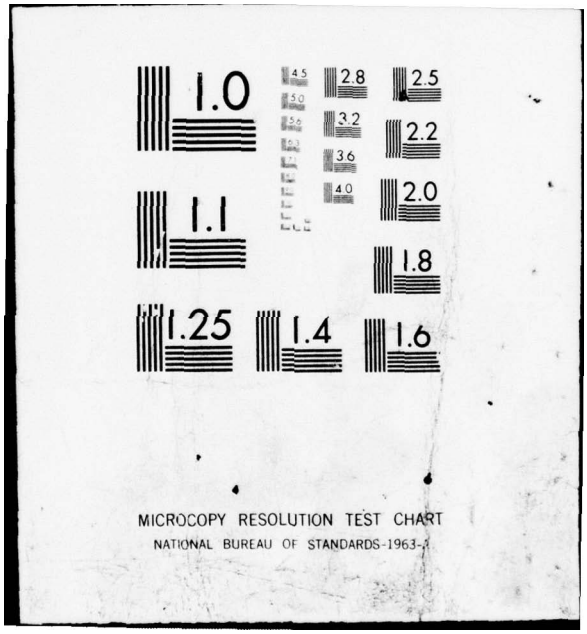
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RADIATIVE TRANSITIONS INDUCED IN GaAs BY MODEST HEAT TREATMENT

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REPORT SF 20

RADIATIVE TRANSITIONS INDUCED IN GALLIUM ARSENIDE BY MODEST HEAT TREATMENT

Technical Report: April 1979
ONR Contract N00014-76-C-0976
Contract Authority NR 243-015

by

Hülya Birey

and

James Sites

Report SF20

Department of Physics
Colorado State University
Fort Collins, Colorado 80523

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

(14) **REPORT DOCUMENTATION PAGE**

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER: SF20
2. GOVT ACCESSION NO.
3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle): (6) Radiative Transitions Induced in GaAs by Modest Heat Treatment
5. TYPE OF REPORT & PERIOD COVERED: (9) Technical Rept.
6. PERFORMING ORG. REPORT NUMBER

7. AUTHOR(s): (10) Hülya Birey and James Sites
8. CONTRACT OR GRANT NUMBER(s): (15) N00014-76-C-0976

9. PERFORMING ORGANIZATION NAME AND ADDRESS: Colorado State University, Fort Collins, Colorado 80523
10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: PE 61153N; RR 021-02-03; NR 243-015

11. CONTROLLING OFFICE NAME AND ADDRESS: Office of Naval Research, Electronic and Solid State Sciences Program, Arlington, VA 22217
12. REPORT DATE: (11) April 1979 (12)
13. NUMBER OF PAGES: 23 / 27p.

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office): (16) RR02102
15. SECURITY CLASS. (of this report): Unclassified
15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

(17) RR02102(3)

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

ONR Scientific Officer

Telephone: (202)696-4218

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Photoluminescence
Gallium Arsenide
Annealing
Doping

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Photoluminescence studies were made on three species of GaAs as a function of annealing time. The evolution of the spectra were carefully monitored and interpreted in terms of the creation of arsenic vacancies at the surface.

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

RADIATIVE TRANSITIONS INDUCED IN GALLIUM ARSENIDE BY MODEST HEAT TREATMENT

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ABSTRACT

Photoluminescence spectra from three species of n-GaAs, lightly Si-doped, heavily Si-doped, and lightly Te-doped, show the onset of additional radiative transitions upon modest annealing in the 550-700°C range. Etch back procedures reveal that the new structure is all surface related. It is attributed to the creation of arsenic vacancies at the surface which (1) allow electrical activation of silicon donors, (2) enhance the probability of silicon site exchange, and (3) lead to complex formation involving both donor and acceptor levels.

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I. INTRODUCTION

Photoluminescence (PL) has been extensively used to characterize many of the impurity and defect energy levels in gallium arsenide.[1-8] Some of these levels have been unambiguously identified, while others have been the subject of speculation and controversy. In particular, there are many situations in which the transition appears to involve a complex of more than one impurity or vacancy. These transitions are often rather broad, sample dependent, and have a peak wavelength that depends on excitation intensity.

The purpose of this paper is to study the effects of modest heating of bulk gallium arsenide, and to attempt to identify the fundamental changes in the material insofar as they can be deduced from the PL spectra. In this study, we have chosen three different types of n-doped GaAs, and have carefully measured the effects of controlled annealing on their PL emissions. We have then integrated our observations with those of other authors to form what we believe is a consistant model of the thermal effects.

II. EXPERIMENTAL

The GaAs samples studied were <100> orientation single crystal wafers. As far as we could establish, they were all grown in silica, as opposed to graphite crucibles, and thus unintentional impurities are likely to be silicon and unlikely to be carbon.

The PL measurements were made with the relatively straightforward apparatus shown in Figure 1. Samples were mounted on the cold finger of a cryogenic dewar that could be operated with either liquid helium or liquid nitrogen. Estimated sample temperatures for these two modes of operation were 12°K and 90°K respectively. The warm up rate after liquid helium boiled away from the chamber shown was about 15°K per hour, allowing

sufficient time to make temperature dependent measurements over the intermediate range. Further heating above 90°K was also possible. The actual dewar used had four light entry windows and was equipped to measure four samples each cool-down.

The PL excitation source was a 50 mW, 6328 Å He-Ne gas laser. The beam was attenuated by a variable density filter, modulated by a 40 Hz mechanical chopper and focused to approximately 1 mm^2 at the sample. The emitted radiation was focused onto the entrance slit of a 1/2 meter grating spectrometer, taking care that the reflected laser light missed the slit. A silicon p-i-n detector was mounted directly over the exit slit of the spectrometer. Its sensitivity covered the 8000 to 10,500 Å range of interest, although some correction must be made for magntidues of spectral lines above 10,000 Å. The current output from the detector was amplified and converted to a voltage which became the input signal of a lock-in amplifier referenced to the chopper driver. The lock-in output was connected to an x-y recorder for preserving the spectra.

Thermal annealing of the GaAs samples was done in a temperature controlled quartz furnace. They were exposed to a continuous flow of hydrogen gas. Annealing temperatures were concentrated in the 550-700°C range and times were varied from 10 to 150 minutes. Samples were kept in the flowing H_2 atmosphere until they cooled to ambient temperature. Etching procedures to establish the depth of the thermally induced transitions were done using a sulferic acid solution. A part of the sample was protected during the etch, and the depth of the resulting step was measured with a scanning electron microscope.

III. RESULTS AND DISCUSSION

A. As-Grown Material

The PL spectra from annealed samples of the three types of GaAs studied are shown in Figure 2 at a temperature of 90°K and again at 12°K in Figure 3. There are three classes of emission lines observable in PL spectra of corresponding samples. Both the lightly doped silicon ($n = 4 \times 10^{15}$ cm^{-3}) and the lightly doped tellurium (4.5×10^{16} cm^{-3}) samples show a narrow peak at 1.503 eV for 90°K and 1.514 eV at 12°K, in each case about 7 meV lower than the band gap for those temperatures.[10] This energy corresponds closely to that of a simple hydrogenic donor impurity[10] and is assumed to result from silicon on a gallium site (Si_{Ga}) in the first case and tellurium on an arsenic site (Te_{As}) in the second. We will refer to this donor-valence band (D - B) transition as Band I. This transition as well as the other assignments we make to the as-grown material are shown in Figure 4.

In the heavily doped silicon ($n = 3 \times 10^{18}$ cm^{-3}) material, there is a second peak labeled Band II at 1.474 eV for 90°K which moves progressively to 1.486 eV at 12°K (see Figure 5a). Band I is also present in these samples at the higher temperatures, but becomes unresolvable at about 35°K (Figure 5b). Both of these peaks are somewhat wider in the heavily doped material than Band I is in the lightly doped samples. We attribute Band II to a donor-acceptor ($Si_{Ga} - Si_{As}$) transition (see Figure 4) since silicon is known to be an amphoteric donor in GaAs, and is found on both gallium and arsenic sites in heavily doped material. The energy of Band II differs from Band I by 28 meV which is consistant with the energy of a hydrogenic acceptor impurity. The increasing dominance of the donor-acceptor transition is interpreted as the decrease in acceptor ionization with lower temperature. Further evidence that Band II involves a donor-acceptor pair comes from

shift of the line to higher energies with increased excitation intensity (1.2 meV/decade in our case) and narrowing of the line (1 meV/decade). Following the argument of Leite and DiGiovanni,[12] there is a coulombic term in a donor-acceptor transition with a spatial energy dependence, and higher intensity excitation light increases the probability of transitions from donors to nearby acceptors.

The third major feature in the unannealed samples is the broad peak near 1.2 eV appearing in both the tellurium and the heavily doped silicon samples. This structure, labeled Band III, is generally attributed to a complex consisting of a donor impurity and a gallium vacancy acting as singly charged acceptor.[13] The observed transitions (see Figure 4) would, therefore, be $(Si_{Ga} - V_{Ga})$ and $(Te_{As} - V_{Ga})$. In the tellurium case, this transition is observed, in agreement with Williams,[13] to occur at a slightly higher energy, presumably because the Group VI donor Te can be adjacent to the vacancy, while the Group IV donor silicon, must be at least a second neighbor site away. The lightly doped Si samples do not show Band III, due, one assumes, to a lack of gallium vacancies. In the other as-grown samples, the intensity of Band III is essentially independent of the measuring temperatures.

B. Heat Treated Material

The PL technique was next used to examine changes in the radiative transitions in GaAs due to heat treatment. All of the peaks observed in the PL spectra for the various samples both before and after annealing are tabulated in Table I. The evolution of the 90°K PL spectrum from lightly doped GaAs:Si for successively longer annealing times is shown in Figure 6. The first feature of interest is the growth in intensity of Band I, the donor to band transition. Similar behavior is observed in a slightly more

heavily doped sample from a different manufacturer. This increase in intensity involves only material in the first few microns from the surface, as determined from etch back procedures. Since the lightly doped material is known to be partially compensated, it is possible that the additional donors are being transfered from acceptor sites, a situation presumably enhanced in the surface region by a higher incidence of vacancies. An alternative explanation is that the additional silicon could diffuse into the GaAs from external sources, such as the quartz annealing tube.

The second major feature of the lightly doped GaAs:Si is the almost immediate appearance and then gradual decrease, of a peak at 1.36 eV (Band IV) and a smaller companion at 1.33 eV (Band IV'). This structure has been observed by many authors, and has been explained in different ways.[3,7,9,14] The major peak is interpreted by us as an arsenic vacancy-silicon complex ($V_{As} - Si_{As}$), and the smaller as the first phonon replica corresponding to the 36 meV LO phonon in GaAs. Since this peak, in our viewpoint, involves Si on As sites, it is logical that it would diminish as the donor peak grows. A very similar looking peak, incidentally, occurs in GaAs with carbon impurities at 1.41 eV and is accompanied by phonon replicas at 1.38 eV and 1.34 eV.[8,15] We assume that the 1.41 eV transition is ($V_{As} - C_{As}$). One report, in fact, finds the 1.36 eV structure when GaAs is annealed in contact with a silicon compound, and the 1.41 eV when it is adjacent to graphite.[16] It would seem reasonable to assume that other acceptors in GaAs will form similar complexes and may, in fact, be indistinguishable from ($V_{As} - Si_{As}$).

The final feature to appear upon modest annealing (Figure 6) for longer times is Band III, not previously seen in the lightly doped GaAS:Si samples. The explanation here is that eventually enough arsenic vacancies form near the surface that it becomes statistically favorable for the reaction $V_{As} + Si_{Ga} \leftrightarrow V_{Ga} + Si_{As}$ to proceed to the right, leading to a

finite concentration of gallium vacancies. At the same time, we see a very small feature at 1.47 eV, the position of the peak previously attributed to a ($Si_{Ga} - Si_{As}$) donor-acceptor transition. Both these peaks are eliminated by etching a few microns from the surface.

The time evolution of the 90°K PL spectrum from a heavily doped GaAs:Si specimen is shown in Figure 7. No significant differences in the evolution were observed at 12°K. In Figure 8a and b, the evolution of the major peaks in the lightly and heavily doped samples are compared, and one observes that the changes in the spectrum from the heavily doped material are much less dramatic. In Figure 8b, we do see a roughly parallel increase in Bands III and IV (1.2 and 1.36 eV) and corresponding decrease in Bands I and II for the single impurity transitions. Again we attribute the basic physical change in the GaAs to the in-diffusion of arsenic vacancies leading to complex formation and the subsequent creation of gallium vacancies through silicon site exchange. As before, the additional structure can be eliminated by etch back techniques.

Of additional interest in the heavily doped silicon material in the development of a peak at 1.44 eV (Band V). This peak is clearly distinguishable from the 1.47 eV peak and in fact is just resolvable in the as-grown material (Figure 7). It is presumably the peak first discussed by Queisser[17] in even more heavily doped GaAs:Si. Following the suggestion of Kressel, et. al.,[3] we think it reasonable to attribute Band V to a donor-acceptor complex of silicon on two adjacent sites, distinguishable from the standard $Si_{Ga} - Si_{As}$ transition at 1.47 eV. We observe that the next larger $Si_{Ga} - Si_{As}$ distance is nearly twice ($\sqrt{11/3}$) the near neighbor distance. After higher temperature annealing (Figure 7), there is a general decrease in the intensity of Band III and a further shift from Band II (1.47 eV) to Band V

(1.44 eV), explained as a tendency for silicon impurities to cluster on neighboring sites.

The GaAs:Te PL evolution is given in Figure 9. There is a small initial increase in Band I and the immediate appearance of Band IV (1.36 eV). We attribute these features to silicon impurities with identical effects as in the lightly Si-doped samples, silicon being likely present because of the silica container for the original crystal growth. The most prominent feature in Figure 9, however, is the appearance of a new peak at 1.31 eV, labeled Band VI. This peak is definitely not a phonon replica of the 1.36 peak. It is too large and occurs at the wrong energy; it would, however, mask any phonon replicas from Band IV. We believe that this new band probably results from a transition from the tellurium donor to a nearby arsenic vacancy acting as an acceptor ($Te_{As} - V_{As}$). It has a width and shape (no phonon replica) quite reminiscent of acceptor bands due to gallium vacancies, but its energy is much larger. This peak which is also seen in more heavily doped GaAs:Te from two different manufacturers, is presumably related to the primary dopant, and it seems very unlikely that tellurium would act as an acceptor. An arsenic vacancy, on the other hand, holds the possibility of pairing the extra electron almost as well by being an acceptor as by being a donor. An alternative explanation, however, is the existence of the other unknown impurity, creating a new deeper acceptor level in the crystal.

IV. CONCLUSIONS

Based on fairly careful photoluminescence measurements of moderate temperature annealed gallium arsenide, we have constructed (Figure 10) a tentative energy level diagram for the common impurities, silicon and

telurium, and their complexes with vacancies. With the exception of the 1.31 eV line in GaAs:Te, all the spectral features described above are represented.

The basic trigger, in our opinion, for the transitions which we see develop during modest anneal cycles is the formation of arsenic vacancies at the GaAs surface. These include (1) a growth of the 1.503 eV donor-band transition in lightly doped GaAs:Si, (2) the appearance of the 1.36 eV arsenic vacancy-acceptor complex transition in all samples, (3) the appearance of the 1.44 eV complex line in heavily doped GaAs:Si, and (4) the appearance of a broad 1.31 eV peak in GaAs:Te. We appreciate that this picture is subject to some discussion, but we feel that it is supported by the bulk of the evidence currently available.

ACKNOWLEDGMENTS

We are grateful to Joe Bowden for the construction of the original photoluminescence apparatus and to Harry Wieder and Larry Lum for many provocative and useful discussions. We particularly acknowledge the support of the U.S. Office of Naval Research through Contract N00014-76-C-0976, and the Scientific and Technical Research Council of Turkey and Istanbul University.

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TABLE I

Sample Properties	Annealing Temp (°C)	Annealing Time (min)	Temp during experiment (°K)	Emission Peaks (eV) band I (D-B)	band II (D-A)	band III complex	band IV complex	band IV' p. replica	band V (D-A)	band VI complex
GaAs:Si	none		90	1.503						
$n=4\times10^{15}cm^{-3}$	600	15	90	1.501			1.36	1.329		
<100>	600	30	90	1.499			1.36	1.329		
	600	60	90	1.501	1.475	1.22	1.36	1.329		
GaAs:Si	none		90	1.503	1.476	1.22				
$n=3\times10^{18}cm^{-3}$	none		12		(1.486)	(1.21)				
<100>	600	15	90	1.495	1.467	1.26			1.44	
	600	60	90	1.495	1.467	1.27	1.36		1.44	
	600	60	12		(1.482)	(1.250)	(1.37)		(1.44)	
	700	15	90			1.26	1.36		1.44	
GaAs:Te	none		90	1.503		1.29				
$n=4.5\times10^{16}cm^{-3}$	none		12	(1.512)		(1.23)				
<100>	600	15	90	1.503		unresolved	1.352			1.31
	600	30	90	1.503	1.47	unresolved	1.352			1.31
	600	30	12	(1.512)	(1.486)	unresolved	(1.365)			(1.32)
	600	60	90	1.503	1.474	unresolved	1.352			1.31
	700	15	90	1.50	1.474	unresolved	1.352			1.31

FIGURE CAPTIONS

Figure 1. Schematic of photoluminescence apparatus.

Figure 2. PL spectra of three types of GaAs examined at 90°K.

Figure 3. PL spectra at 12°K.

Figure 4. Suggested energy diagram for as grown GaAs:Si, Te.

Figure 5. Temperature dependence of (a) PL peak energy, (b) PL intensity for heavily doped GaAs:Si.

Figure 6. Evolution of PL spectrum with annealing time for $n = 4 \times 10^{15}$ cm^{-3} GaAs:Si.

Figure 7. Evolution of PL spectrum for $n = 3 \times 10^{18}$ GaAs:Si.

Figure 8. PL intensities vs. annealing time for (a) n^- - GaAs:Si (b) n^+ - GaAs:Si (c) n - GaAs:Te.

Figure 9. Evolution of PL spectrum for $n = 4.5 \times 10^{16}$ GaAs:Te.

Figure 10. Suggested energy diagram for common transitions in heat treated GaAs. 1.31 eV transition for Te-doping not shown (see text).

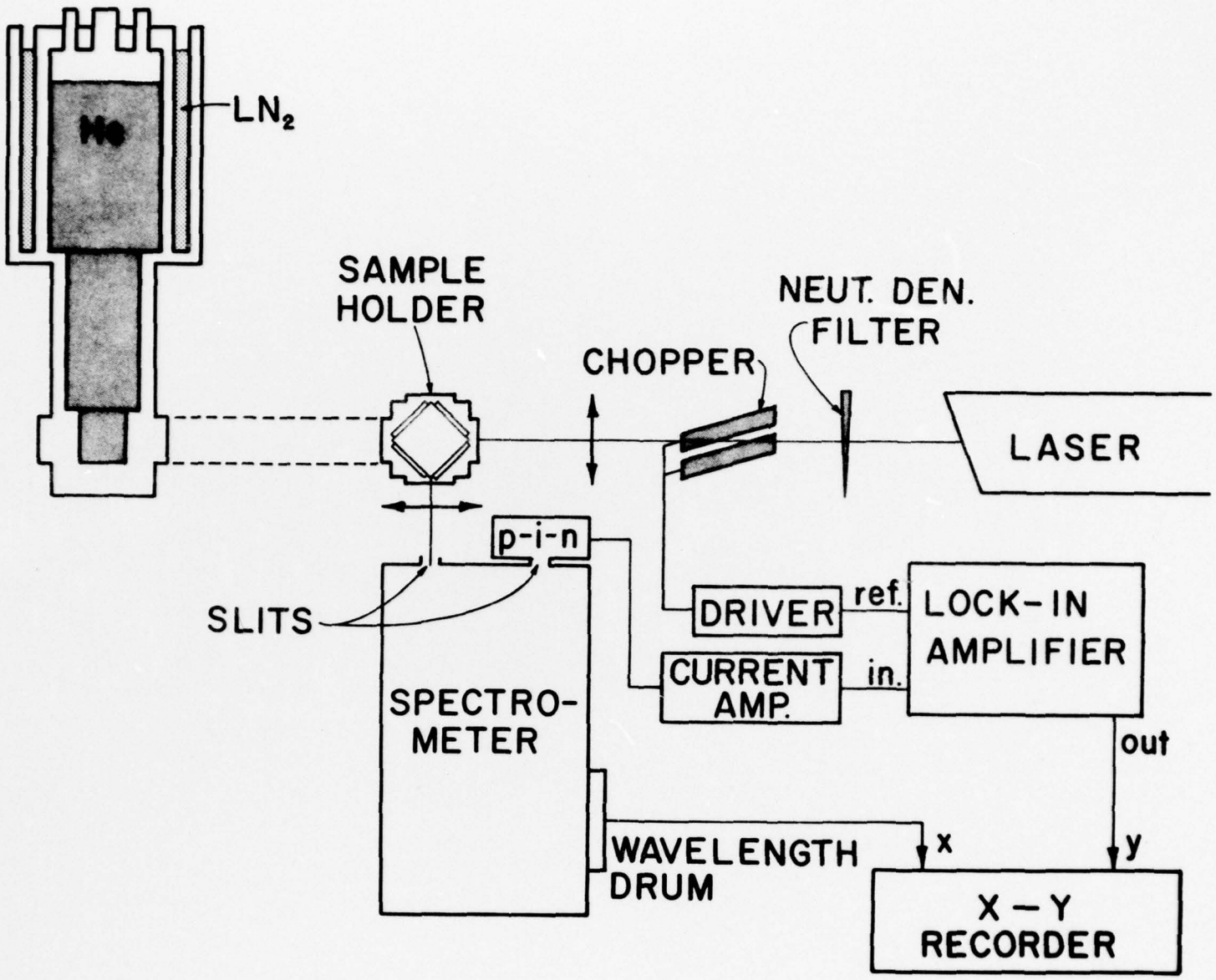
LN2
He
SAMPLE HOLDER
CHOPPER
NEUT. DEN. FILTER
LASER
p-i-n
SLITS
DRIVER
ref.
LOCK-IN AMPLIFIER
CURRENT AMP.
in.
SPECTRO-METER
out
WAVELENGTH DRUM
x
y
X – Y RECORDER

Fig. 1

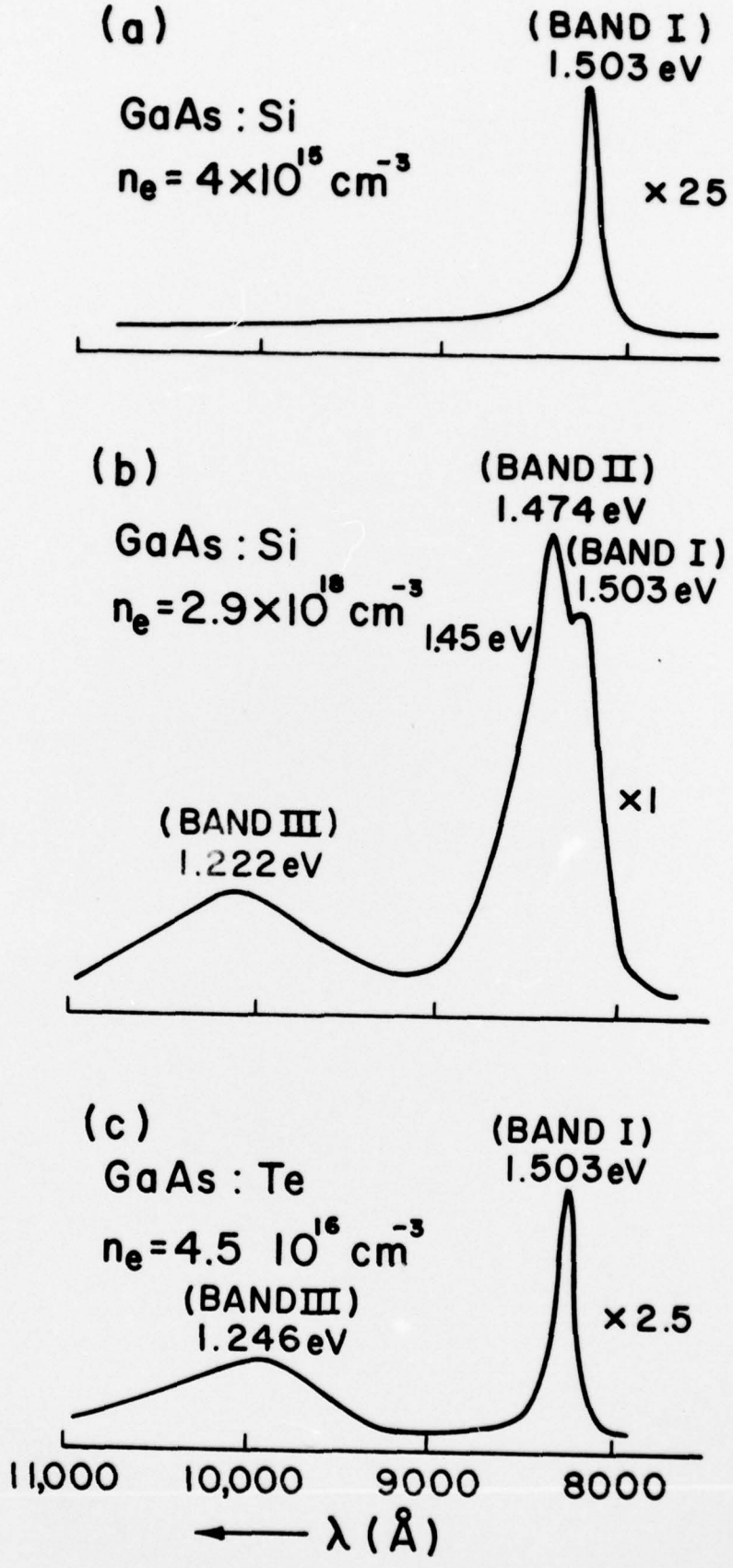
(a)
(BAND I)
1.503 eV
GaAs : Si
$n_e = 4 \times 10^{15}\ cm^{-3}$
× 25
(b)
(BAND II)
1.474 eV
GaAs : Si
(BAND I)
1.503 eV
$n_e = 2.9 \times 10^{18}\ cm^{-3}$
1.45 eV
× 1
(BAND III)
1.222 eV
(c)
(BAND I)
1.503 eV
GaAs : Te
$n_e = 4.5\ 10^{16}\ cm^{-3}$
(BAND III)
1.246 eV
× 2.5
11,000
10,000
9000
8000
← λ (Å)

Fig. 2

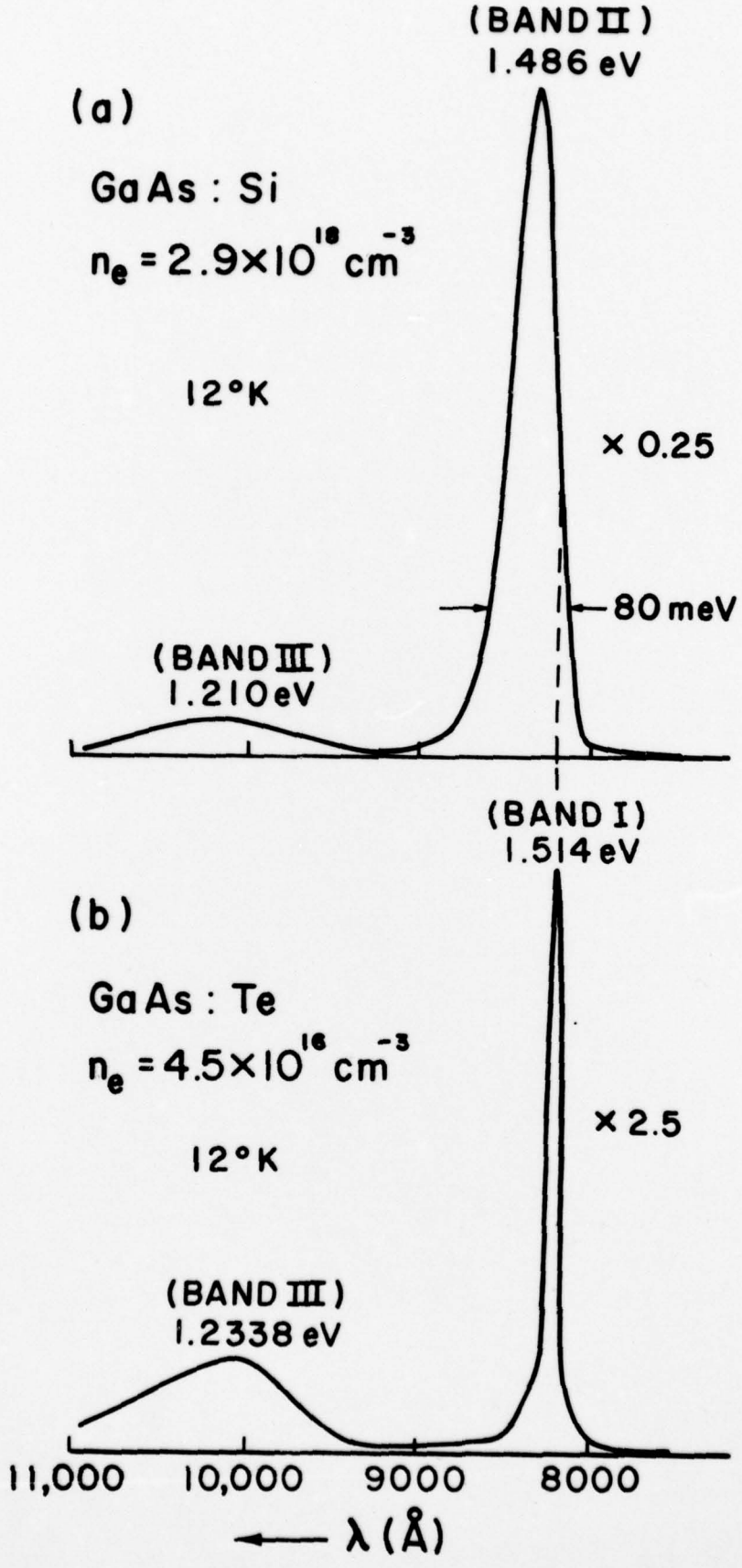
(BAND II)
1.486 eV
(a)
GaAs : Si
$n_e = 2.9 \times 10^{18} cm^{-3}$
12°K
× 0.25
80 meV
(BAND III)
1.210 eV
(BAND I)
1.514 eV
(b)
GaAs : Te
$n_e = 4.5 \times 10^{16} cm^{-3}$
12°K
× 2.5
(BAND III)
1.2338 eV
11,000
10,000
9000
8000
← λ (Å)

Fig. 3

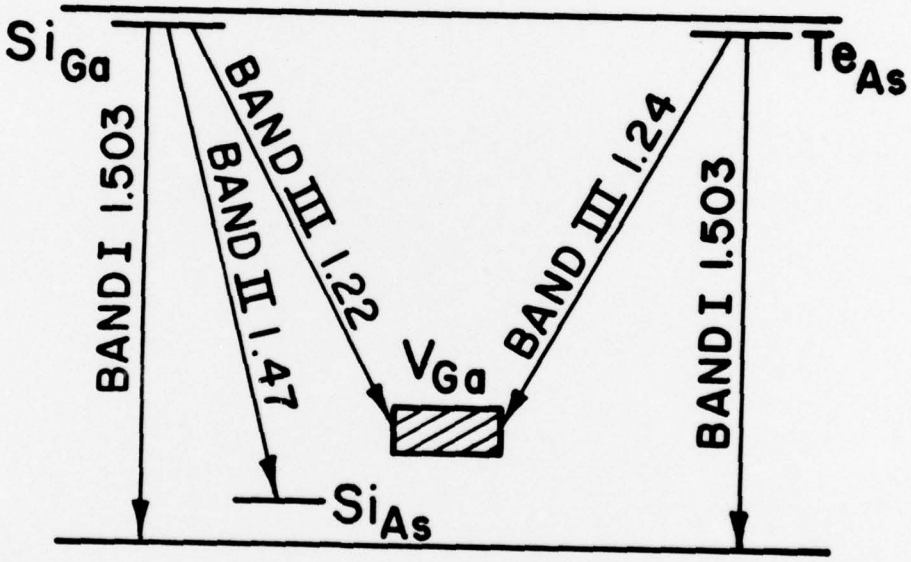
Si_{Ga}
Te_{As}
BAND I 1.503
BAND II 1.47
BAND III 1.22
V_{Ga}
BAND III 1.24
BAND I 1.503
Si_{As}

Fig. 4

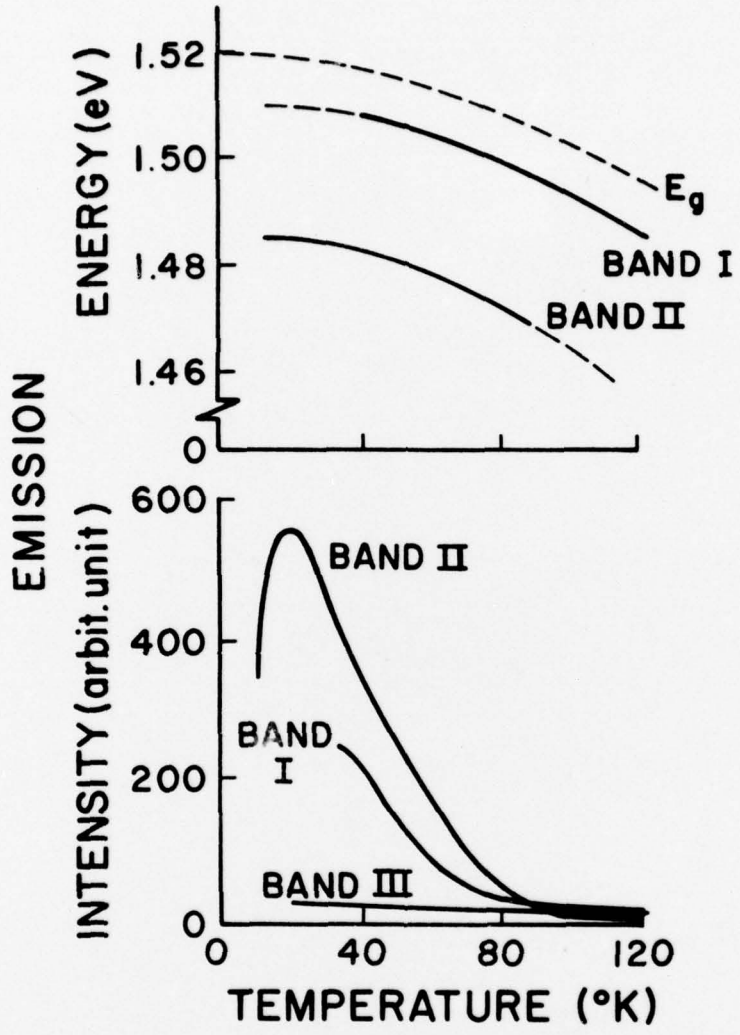
EMISSION
ENERGY (eV)
1.52
1.50
1.48
1.46
0
E_g
BAND I
BAND II
INTENSITY (arbit. unit)
600
400
200
0
BAND II
BAND I
BAND III
0
40
80
120
TEMPERATURE (°K)

Fig. 5

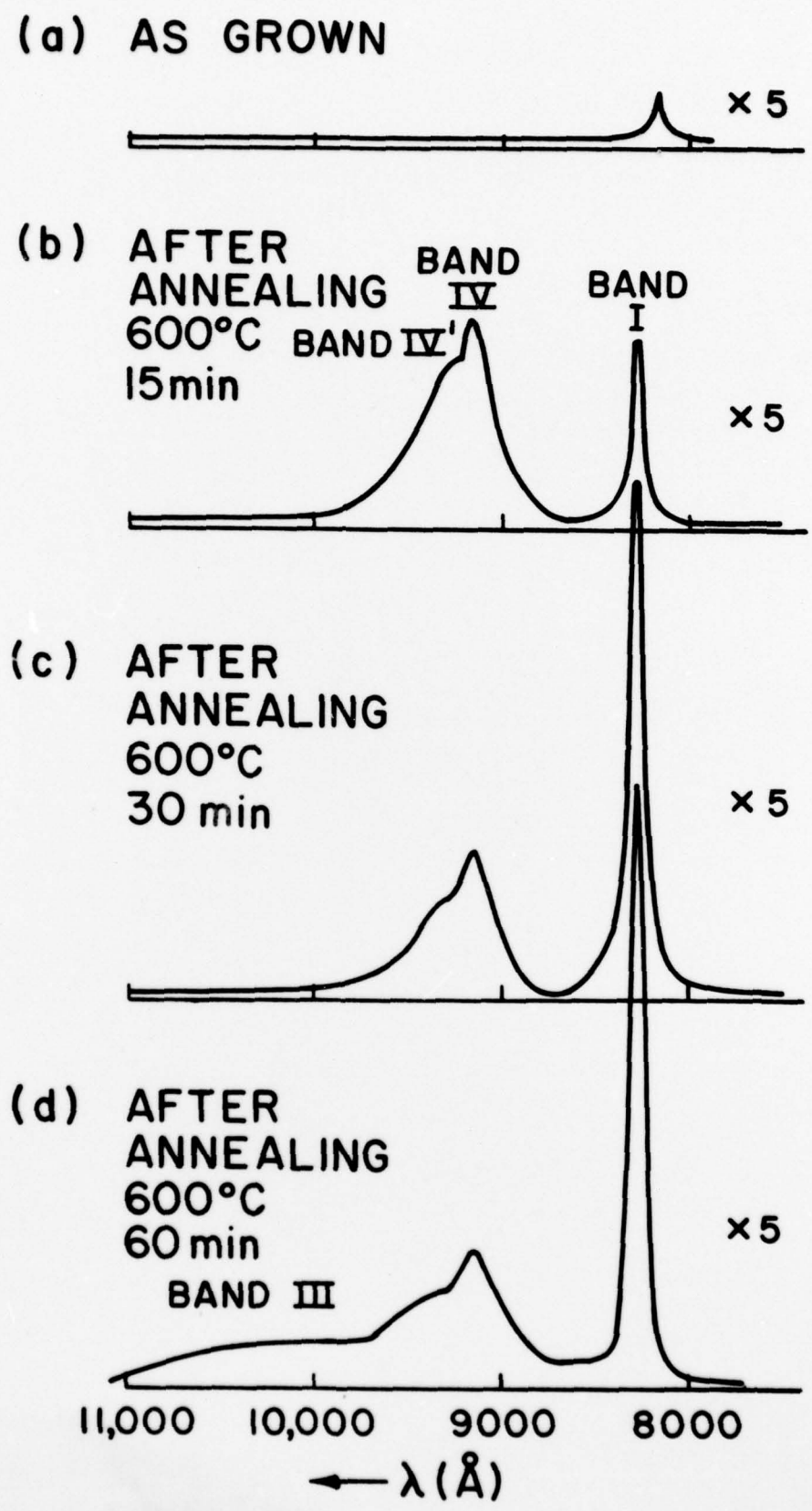
(a) AS GROWN
×5
(b) AFTER ANNEALING 600°C 15min
BAND IV
BAND IV'
BAND I
×5
(c) AFTER ANNEALING 600°C 30 min
×5
(d) AFTER ANNEALING 600°C 60 min
BAND III
×5
11,000
10,000
9000
8000
← λ (Å)

Fig. 6

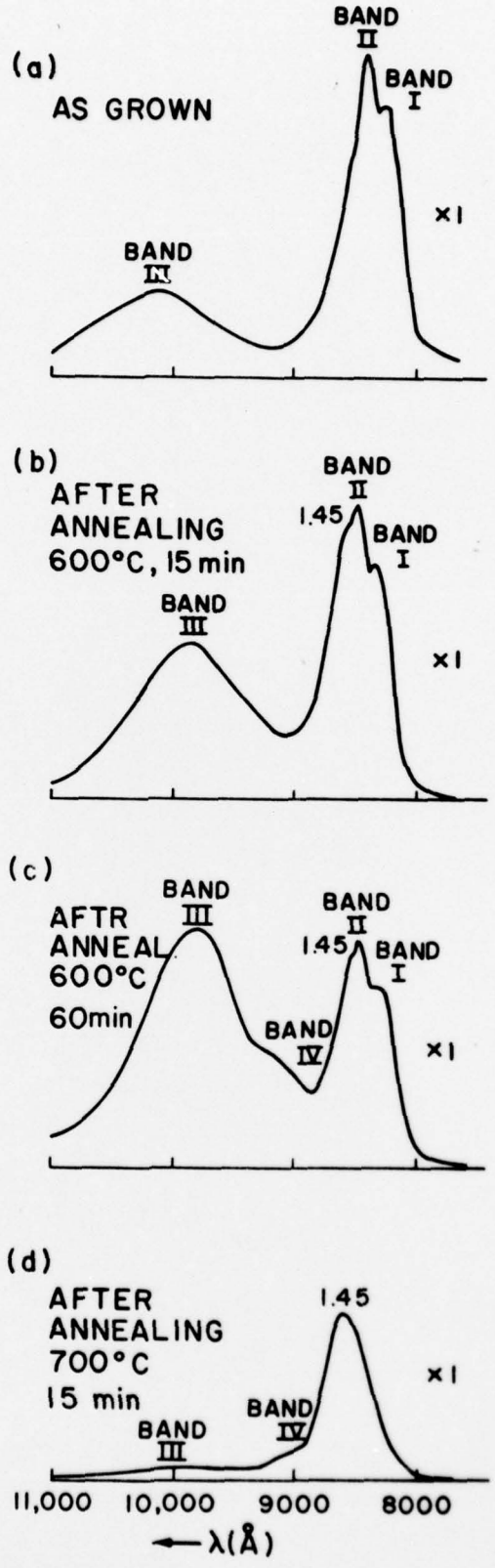
(a)
AS GROWN
BAND II
BAND I
BAND III
×1
(b)
AFTER ANNEALING 600°C, 15 min
BAND II
1.45
BAND I
BAND III
×1
(c)
AFTR ANNEAL 600°C 60min
BAND III
BAND II
1.45
BAND I
BAND IV
×1
(d)
AFTER ANNEALING 700°C 15 min
1.45
×1
BAND IV
BAND III
11,000
10,000
9000
8000
← λ(Å)

Fig. 7

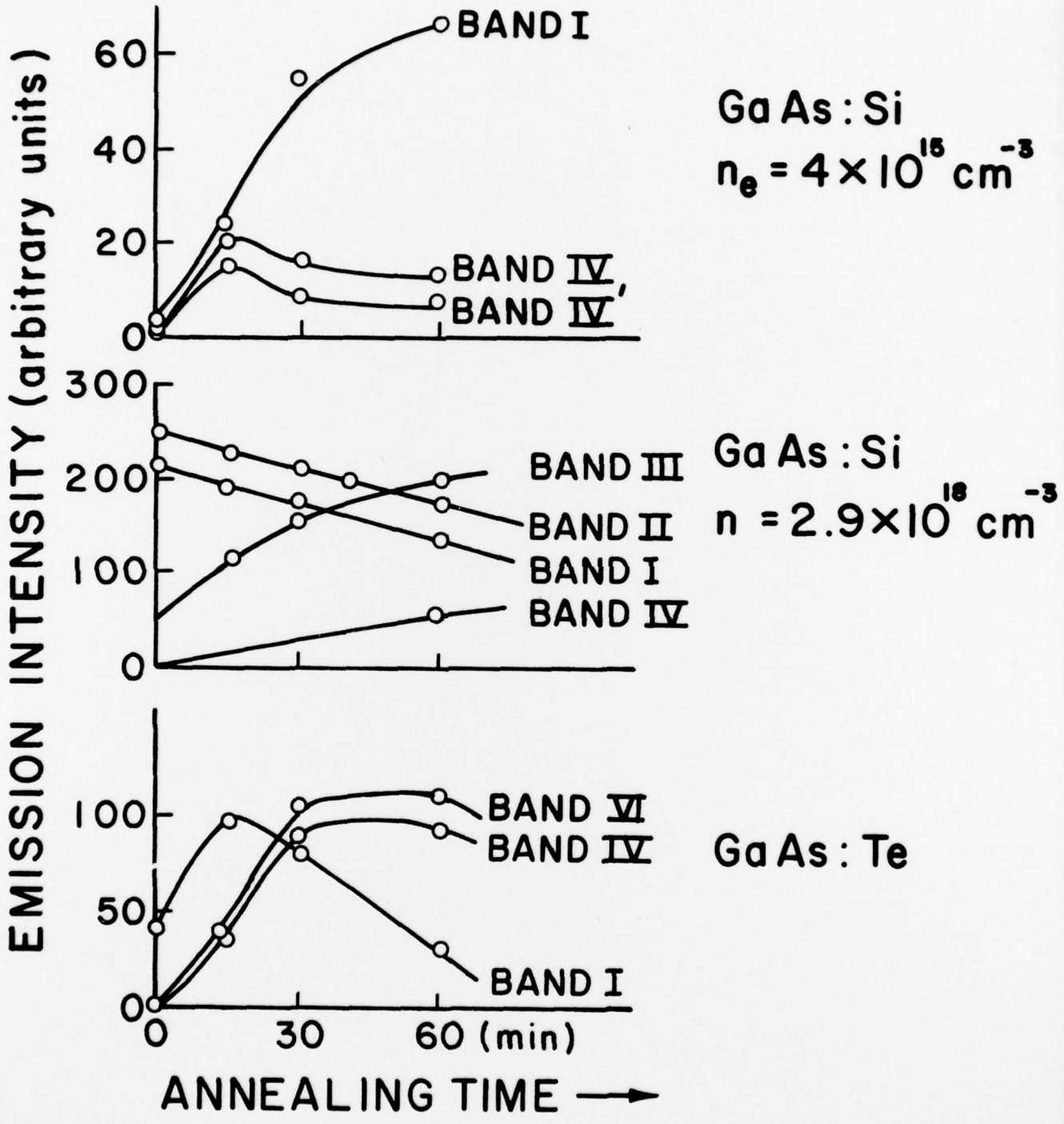
BAND I
60
40
20
0
BAND IV,
BAND IV
Ga As : Si
$n_e = 4 \times 10^{15}$ cm^{-3}
300
200
100
0
BAND III
BAND II
BAND I
BAND IV
Ga As : Si
$n = 2.9 \times 10^{18}$ cm^{-3}
100
50
0
BAND VI
BAND IV
BAND I
Ga As : Te
0
30
60 (min)
ANNEALING TIME →
EMISSION INTENSITY (arbitrary units)

Fig. 8.

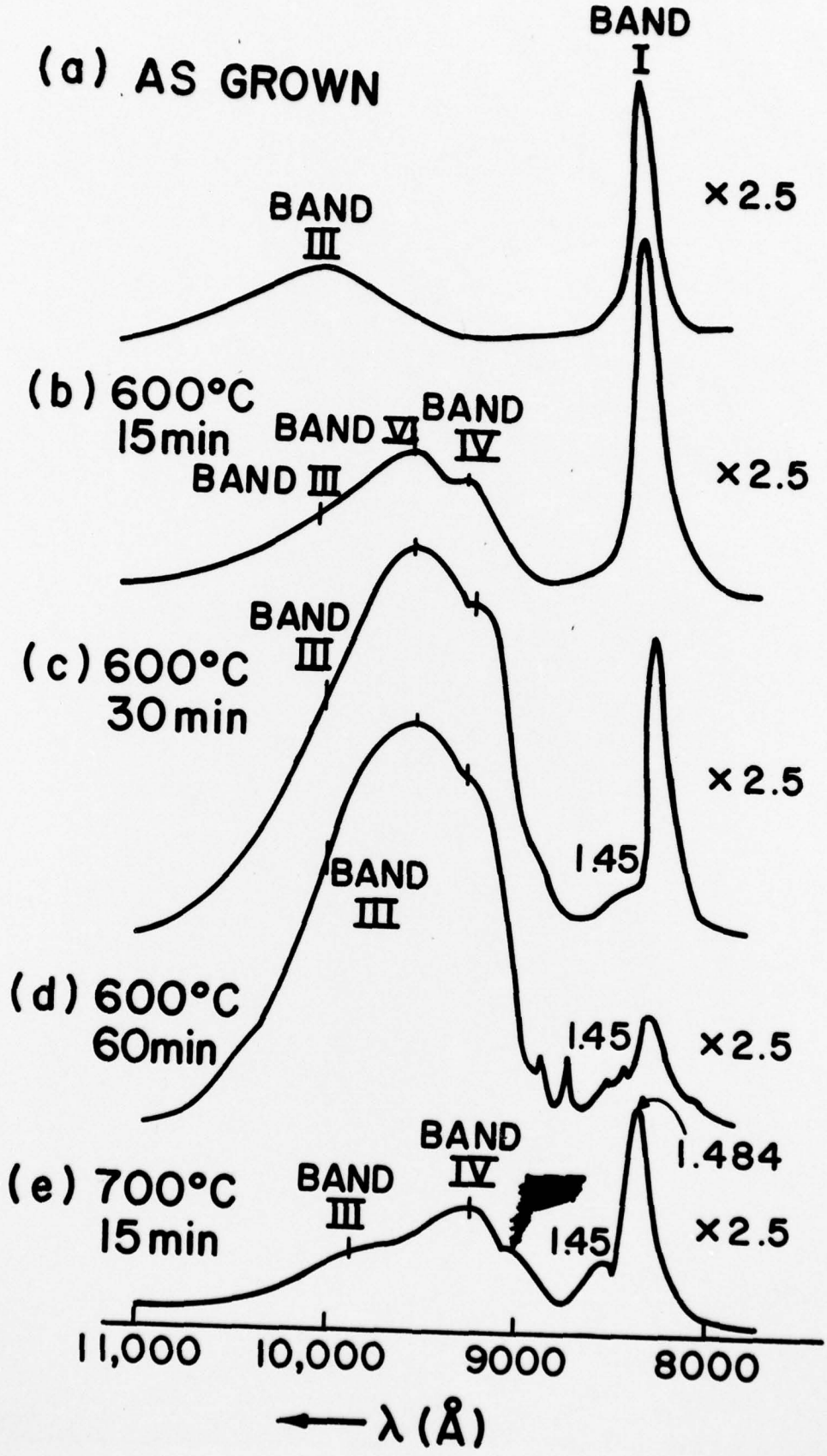
(a) AS GROWN
BAND I
BAND III
×2.5
(b) 600°C 15min
BAND VI
BAND IV
BAND III
×2.5
(c) 600°C 30min
BAND III
×2.5
1.45
BAND III
(d) 600°C 60min
1.45
×2.5
BAND IV
1.484
(e) 700°C 15min
BAND III
1.45
×2.5
11,000
10,000
9000
8000
← λ (Å)

Fig. 9

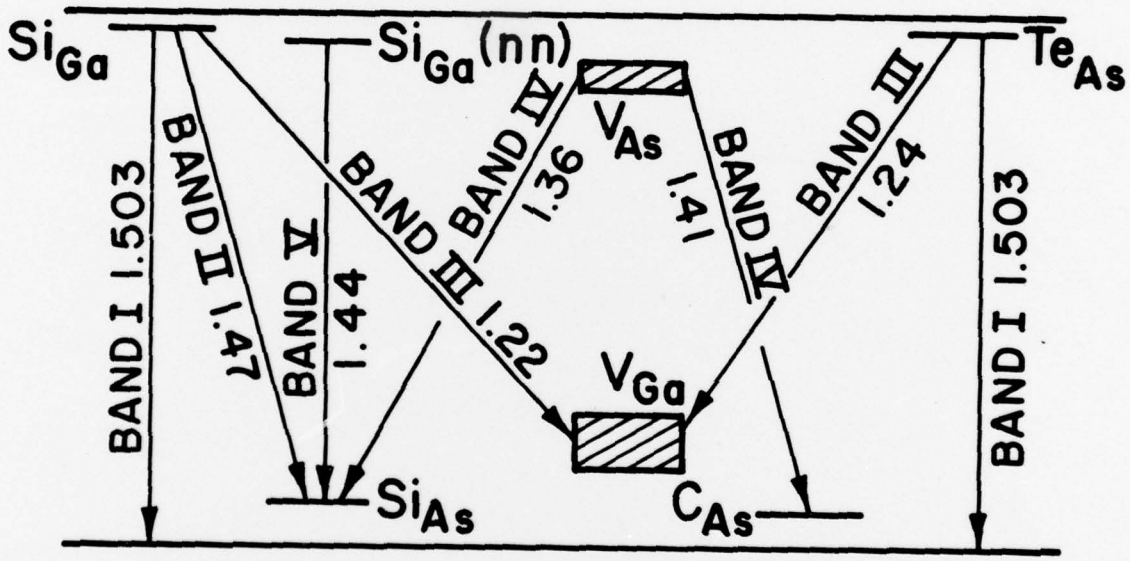
Si_{Ga}
$Si_{Ga}(nn)$
Te_{As}
BAND I 1.503
BAND II 1.47
BAND V
1.44
BAND III 1.22
BAND IV
1.36
V_{As}
1.41
BAND III
BAND IV
1.24
BAND I 1.503
V_{Ga}
Si_{As}
C_{As}

Fig. 10

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