REPORT DOCUMENTATION PAGE

OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	4/30/95	12/1/93 - 4/30/95 FINAL

4. TITLE AND SUBTITLE

Electrically Pumped Erbium Pumped Silicon Lasers

5. FUNDING NUMBERS

G

F49620-92-J-0162

Project Task 2305/ES

61102F

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8. PERFORMING ORGANIZATION REPORT NUMBER

1

AFOSR-TR- 95-0443

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

AFOSR/~~PKD~~ NE
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Building 410
Bolling AFB, DC 20332-6448

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

F49620-92-J-0162

11. SUPPLEMENTARY NOTES

-

12a. DISTRIBUTION/AVAILABILITY STATEMENT

- Approved for public release; distribution unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

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Cathodoluminescence (CL) measurements have yielded strong Er^{3+} luminescence in Er and Oxygen implanted GaN films on sapphire and Er and O in sapphire. Multiple spectral peaks from the IR, through the visible, and into the near UV have been identified as atomic 4f shell transitions from the Er in sapphire. Photoluminescence (PL) using an Argon laser (514 nm) on one GaN:Er:O sample showed weaker luminescence at 1540 nm. The CL temperature dependence of the 1540 nm peak was found to drop by only a few percent from 6 K to 300 K in the GaN:Er:O. The intensity dropped to 25% of the 6 K value at 380 K. Refractive index measurements of some GaN:Er samples are also presented.

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14. SUBJECT TERMS	15. NUMBER OF PAGES
silicon optoelectronics, rare earth doped semiconductors, erbium doped silicon, Gallium nitride	8
	16. PRICE CODE: -

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	-

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
298-102

Electrically Pumped Erbium Doped Silicon Lasers
Final Report: Dec. 1, 1993 - April 30, 1995
AFOSR Award F49620-92-J-0162

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Executive Summary

Rare earth doped semiconductors offer the possibility of electrically pumped, efficient, and inexpensive integrated optical amplifiers and CW sources for use in optical communications systems. This final report discusses research into Er doped GaN and Si materials.

Cathodoluminescence (CL) measurements have yielded strong Er^{3+} luminescence in Er and Oxygen implanted GaN films on sapphire and Er and O in sapphire. Multiple spectral peaks from the IR, through the visible, and into the near UV have been identified as atomic inner 4f shell transitions from the trivalent Er in sapphire. Photoluminescence (PL) using an Argon laser (514 nm) on one GaN:Er:O sample showed much weaker, but detectable luminescence at 1540 nm. The CL temperature dependence of the 1540 nm peak was found to drop by only a few percent from 6 K to 300 K in the GaN:Er:O. The intensity dropped to 25% of the 6 K value at 380 K. Refractive index measurements of some GaN:Er samples are also presented.

Various Er-doped Si samples were studied. One set were implanted with Er ions while the other set were grown epitaxially with in situ Er doping using chemical vapor deposition. The sample preparation conditions are described. PL of these samples was studied using a 40 mW, 1490 nm laser diode, an 80 mW 980 nm laser diode and an argon laser at 514 nm. PL signals have been detected from some Er and F doped Si samples. Future plans are discussed.

ACRONYMS

CL:	Cathodoluminescence, light emission from sample bombarded with high energy electron beam.
Cr:	chromium, a common masking material.
CVD:	chemical vapor deposition, an epitaxial growth technique for semiconductors.
CW:	continuous wave.
EL:	Electroluminescence, light emission due to electric current flowing through the sample.
Er:	erbium, a rare earth element, number 68 on the periodic table.
F:	fluorine, element number 9 on the periodic table.
FWHM:	full width at half maximum.
GaN:	gallium nitride, a wide bandgap III-V semiconductor material.
Ge:	germanium, useful as detector from 0.8 - 1.7 μm.
IR:	infrared region of the spectrum, $\lambda > 700$ nm.
NIST:	National Institute of Standards and Technology, a local government laboratory.
PL:	Photoluminescence, light emission from sample illuminated with light of higher energy than the measured signal.
RBS:	Rutherford backscattering, materials analysis technique.
RIE:	reactive ion etching.
RTA:	rapid thermal anneal.
Si:	silicon, element number 14 on the periodic table.
SIMS:	secondary ion mass spectrometry, materials analysis technique.
TEC:	thermoelectrically cooled.
UV:	ultraviolet region of the spectrum, $\lambda < 400$nm.

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I. INTRODUCTION

Rare earth doped semiconductors offer the possibility of electrically pumped, efficient, and inexpensive integrated optical amplifiers and CW sources for use in optical communications systems. This report discusses our research into Er doped GaN and Si materials.

This project consists of EL, CL, and PL studies of Er in crystalline Si and GaN. The goal is to develop an electrically pumped CW laser and/or amplifier operating at 1540 nm for compact and lightweight optical fiber communication systems, which may be integrated with standard Si electronic circuits. The goal is to directly impact-excite the Er in the semiconductor by hot electron bombardment to generate the Er luminescence. By pumping the Er directly using CL, as well as indirectly with PL (i.e.with intermediate electron-hole pairs in the Si), we hope to gain a greater understanding of the excitation and relaxation mechanisms of this system.

Favennec et al. [Favennec] showed that the characteristic thermal quenching of the Er^{3+} related luminescence decreases as the bandgap increases in the host crystal. This along with the many desirable material properties of GaN [Pankove], such as high thermal and electrical conductivities, chemical and structural stability and hardness, makes it a good candidate for room temperature, and even high temperature, rare earth doped optical devices. The Er-doped GaN films have exhibited both CL and PL. CL studies of Sapphire:Er yielded a detailed spectrum with many strong Er^{3+} related peaks. Also GaN:Er exhibited, to the best of our knowledge, the first known electron beam excited luminescence at 1540 nm. This transition is between the Er atomic levels $^4I_{15/2}$ and $^4I_{13/2}$. This wavelength is of great importance in optical fiber communication since it corresponds to low loss and low dispersion in silica based fibers. The temperature dependencies of the Er related luminescence at 1540 nm in GaN and at 410 nm in Sapphire:Er have been investigated. The GaN:Er 1540 nm PL signal only dropped by a few percent between 6 K and 300 K. This is very promising in terms of future device realization.

We first describe the measurement systems. Then we show CL and PL measurements of Er implanted GaN. We also discuss the issues involved in fabricating waveguides in this material. In conclusion we sum up the work done to date and discuss future work .

II. MEASUREMENT SYSTEMS

II.1 PHOTOLUMINESCENCE SYSTEM

A liquid nitrogen cooled PL system (Figure 1) is used to measure luminescence. The system is designed so a sample may be excited by a laser source or by electrical means. The system includes a Stanford Research lock-in amplifier, an IBM-compatible computer, and computer controlled Instruments SA grating monochromator, model HR 320. Detectors are discussed below.

The collected luminescence passes through a chopper before it reaches the monochromator. There is a also bandpass filter (1520-1600 nm) before the detector. The computer scans wavelengths by controlling the monochromator and reads the output voltage from the lock-in amplifier.

This system may be used for PL of bulk samples as well as waveguiding samples. Also, since the cryogenic chamber is on a stage, it is possible to study several samples that are mounted inside the chamber with minimal realignment.

For waveguiding samples, the collimated pump beam is focused on the edge of the waveguide with a microscope objective and the output light is gathered by another objective. The output light is then focused on the slit of the monochromator by a cylindrical lens.

We presently have a 1485 nm, 40 mW fiber coupled laser diode for pumping. An interference filter centered at 1490 nm blocks the spontaneous emission noise of the pump sources which extend into the Er luminescence band. We also have recently received a 980 nm, 100 mW laser diode from IBM. Both these wavelengths are absorbed by Er.

II.1.2 DETECTORS

The PL system was originally used with an EG&G TEC Ge detector. The TEC Ge detector is reverse biased with 4 volts in series with a load resistor (typically 100 kΩ to 500 kΩ). We

concluded that the TEC Ge detector did not have adequate sensitivity for this work.

We received a damaged liquid nitrogen-cooled North Coast Ge detector donated by the National Renewable Energy Laboratory in Golden, Colorado. We were able to repair some of the damage to the detector making it useable. There are a few problems with the detector. The response is very slow; chopping the incoming signal faster than 15 Hz results in a significant drop off in signal strength. We are unable to put the proper high voltage bias across a cooled low-noise JFET amplifier in the detector reducing the amplification. Therefore, the detector yields less than the rated $5x10^9$ Volts per Watt of optical input power. However, it still provides significantly better sensitivity than the TEC Ge detector.

To improve the performance of the EG&G J16 TE2 TEC Ge detector, a low-noise amplifier was added. This amplifier is a Burr-Brown OPA111B low noise precision operational amplifier. The OPA111B was chosen for it's low bias current requirements as well as its low noise characteristics.

OPA111B Characteristics

Parameter	Typical Value	Max	Units
Noise (f_0 = 100 Hz)	11	30	nV / √Hz
Bias Current	0.5	1	pA
Open-Loop Voltage Gain	120	125	dB

The circuit was designed as a transimpedance amplifier with approximately no bias across the detector (Figure 2). Room was left in the amplifier circuitry for additional feedback resistors, allowing different gain values. The completed detector/amplifier circuitry was tested and the responsivity was measured to be 3.2×10^7 V/W.

II.2 CATHODOLUMINESCENCE SYSTEM

There is also a computer controlled, cryogenic CL system that can reach temperatures of 6 K (Figure 3). The electron gun used is a Thermionics Inc., type VE052, that can provide currents up to 25 μA and an accelerating potential of 20 kV. The luminescence is measured with a Perkin Elmer automated prism spectrometer and either of the Ge detectors mentioned above, or a dry ice cooled PbS detector for infra-red measurements. The output is connected to a Princeton Applied Research lock-in amplifier.

III. ERBIUM DOPED SILICON

There were two groups of Er-doped Si samples available for measurements during the past year. One group is epitaxially grown Er-doped Si on a <100> Si substrate. These samples were grown by Prof. W. Varhue at the University of Vermont. The second group is Er-implanted SIMOX supplied by Dr. F. Namavar of Spire Corporation. In addition, we have a number of Er implanted Si on sapphire samples.

III.1 MATERIAL PREPARATION

III.1.1 EPITAXIAL ERBIUM DOPED SILICON

Dr.Varhue is using low temperature CVD growth to epitaxially grow a layer of Er-doped Si on top of a Si substrate. The epitaxial Er-doped wafers are doped with approximately 10^{19} cm^{-3} Er. Some of the samples are co-doped with oxygen at concentrations approximately ten times that of the Er. The substrate is oriented in the <100>plane. These samples were grown at low temperatures (350 C). The samples were not annealed. Light confinement in the Er:Si layer is an area to be studied, and is essential for waveguiding devices.

III.1.2 SIMOX

The SIMOX samples provided by Dr. F. Namavar were implanted with Er^{2+} ions at 400 keV. These samples are also codoped with oxygen at various densities. A SIMOX wafer is a Si

wafer with a crystalline Si surface layer above a narrow layer of SiO_x. The SIMOX samples are advantageous for the fabrication of waveguides. An oxide layer beneath the Er-doped Si layer provides optical confinement to any light in the Er-doped Si top layer, which has a much larger index. Otherwise, much of the light would travel in the optically inactive portions of the wafer.

III.1.3 ERBIUM DOPED SILICON ON SAPPHIRE

Planar waveguides in Er implanted Si grown on sapphire (Al_2O_3) substrates have been investigated. Although there is a lattice mismatch between Si and sapphire, the Si films are crystalline and of good optical quality. The refractive indices of Si and sapphire are 3.4 and 1.8 respectively. The large index difference in the two materials makes the material system a good candidate for fabricating single mode waveguides with strong confinement of the mode. In the active medium, a thin layer of Er-doped Si, the Er ions will then have a greater overlap with the pump field. Unfortunately, the mode matching to optical fibers is poor.

III.1.4 IMPLANTATION

Six 2-inch wafers with Si films (varying thickness) grown on sapphire were donated by Dr. Richard Soref at the USAF Rome Laboratory. One whole wafer, and one half of another wafer were implanted with Er by Dr. Stephen Withrow at Oak Ridge National Laboratory . The implantation conditions are listed in Table I below:

Table I - Implantation Conditions:

	Sample:	Implants:
1.	ErSi:	0.69×10^{14} Er^+/cm^2 at 1 MeV and
	(1 μm Si on sapphire)	0.31×10^{14} Er^+/cm^2 at 600 keV
2.	ErSi:	0.69×10^{14} Er^+/cm^2 at 1 MeV and
	(1.5 μm Si on sapphire)	0.31×10^{14} Er^+/cm^2 at 600 keV

The implantation depths were calculated to be 329 nm for 1 MeV with a straggle of 23 nm, and for 600 keV a depth of 190 nm with a straggle of 16 nm.

III.1.5 ANNEALING

The samples were rapid thermal annealed (RTA) by Ms. Carol Lee at The Army Research Lab., Ft. Monmouth, NJ, through a collaboration with C. Lee, K.K. Choi, and Wayne Chang. The samples were annealed in a nitrogen atmosphere. The Si flaked off sample 1 during the first anneal, probably due to the mismatch in coefficients of thermal expansion in Si and sapphire. Visual inspection showed that the following anneals of the remaining samples went well. However, the temperature may not have been sufficient to repair all the damage to the lattice that occurred during the implantation. The relevant parameters used in the rapid thermal anneal are listed in Table II below. The sample numbers correspond to the implant conditions listed above in Table I.

Table II - Annealing (RTA) parameters for Si on sapphire:

Sample:		Anneal	Temp.	Duration	Ramp time
1.	ErSi	RTA	900C	15 sec	?
2a.	ErSi	RTA	600C	15 sec	170 sec
2b.	ErSi	RTA	600C	15 sec	113 sec
2c.	ErSi	RTA	600C	15 sec	74 sec

III.2 OPTICAL MEASUREMENTS

III.2.1 ABSORPTION IN EPITAXIALLY GROWN ERBIUM-DOPED SILICON

Absorption spectra of three, 3 inch wafers of epitaxially grown Er:Si layers grown on Si substrates were measured. The epitaxial layers were grown by Prof. Varhue at The University of

Vermont. The absorption measurement was performed using a Cary spectrophotometer. A weak absorption signal was found at 1540 nm in wafer UVM-ER9 (Figure 4). There was no detectable Er related absorption on the other two samples. PL and CL were also performed on these samples with no luminescence signal detected.

A new sample, PA49, provided by Prof Varhue of UVM, did show Er PL using a 980 nm pump and 2 mm slits at 77 K (Figure 5). There is also a plot of the PL at room temperature. Ths sample was grown at 400 C and was not annealed following growth. The Er source is an amido compound synthesized at UVM [Varhue].

III.2.2 PHOTOLUMINESCENCE

The Er-doped Si samples were mounted inside the cryogenic chamber. The first measurements involved shining the laser straight through the sample. The beam travels through the Er-doped surface of the sample as well as the substrate. The Si bandgap of 1.1 eV provides a substrate which is optically transparent for the 1485 nm pump wavelength. The difficulty with this sort of measurement is the very thin Er-doped region which only absorbs approximately $5 * 10^{-6}$ of the incident pump light, leading to very small signal powers. A theoretical luminescence calculation for the sample shows that luminescence emission will be very weak. The theoretical result [Pankove2] for our system is only 16 μV of signal for 1 W of 1480 nm pump power.

III.2.3 LUMINESCENCE MEASUREMENT AT NIST

Dr. K. Malone at NIST provided access to a high powered Ti-sapphire and argon lasers for our use to measure some GaN and Si samples. The Ti-sapphire laser emitted up to 1 Watt of 980 nm light and the argon laser could produce nearly 10 Watts of light at 514 nm. Time limitations allowed us to measure only one silicon sample (pumping down and cooling the cryogenic chamber takes a significant amount of time). This sample was excited with the Ti-sapphire laser, but no Er luminescence was detected.

III.2.4 CATHODOLUMINESCENCE MEASUREMENT

Several SIMOX and epitaxially grown samples were studied in the cryogenic CL system. The CL system was cooled to approximately 6 K. Electrons were directed at the sample with a current of 20 μA and 15 kV potential. A luminescent Er-doped GaN sample was used as a standard for alignment. No luminescence was found in any of the Er:Si samples.

III.2.5 ANNEALING

We theorized that the reason for the optical inactivity of the Er was a need for interaction between the Er and oxygen atoms in order to place the Er in the optically active Er^{3+} state. The epitaxially grown samples were never heated above 350 C, so the atoms were not free to move in the Si lattice and will not interact. The implanted SIMOX samples were only annealed with RTA which does not allow adequate time for the atoms to move and interact. In order to allow the oxygen time to move in the lattice, and possibly bond with Er, we annealed some of each sample type for 5 hours at 625 C followed by 20 minutes at 900 C. The anneal was performed in a furnace with a mostly nitrogen atmosphere. Following the anneal, CL measurements were performed. At this time, these measurements have yet to yield any Er luminescence signals.

III.2.6 WAVEGUIDING SAMPLES

Waveguiding samples get around the problem of the short interaction length for pump light incident on a sample with only a thin Er layer. The light is guided through 1 cm or more of material doped with Er increasing the absorption and the signal. The first waveguide samples measured were slab waveguides of Er-doped Si on Si grown by Prof.Varhue. The samples were mounted on cold fingers. Microscope objective lenses were used to focus the pump beam into the guide and to gather the light leaving the guide. A Micronviewer 7290 Infrared Camera was acquired in order to view the infrared laser beam and assist in aligning the pump beam into the guiding structure. No luminescence was detected.

III.3 PHOTOLUMINESCENCE WITH VARYING DOPANT DENSITIES

It is important to study the luminescence of Er-doped silicon with respect to dopant and co-dopant densities. Comparing varying densities will provide the knowledge to produce optimized luminescent devices and also may provide some insight into the environment necessary for efficient luminescence.

Dr. Namavar produced an Er-doped silicon wafer in a 4 by 4 grid. In one direction the Er dose was varied and in the other direction the F co-dopant dose was varied. The Er doses are 10^{14}, $5x10^{14}$, 10^{15}, and $2x10^{15}$ cm^{-2}. The Er was implanted at 190 keV double charged (i.e. Er^{++}. equivalent to 380 keV for a single charged ion). The F co-dopant doses are 10^{15}, $5x10^{15}$, 10^{16}, and $5x10^{16}$ cm^{-2}. The F^{+} ions were implanted at 100 keV. The sample dosages are displayed in Table III.

Table III. Row and Column Er and F doses

Sample #	Col 1	Col 2	Col 3	Col 4	Erbium Dose =
Row 1	1	2	3	4	$1x10^{14}$ cm^{-2}.
Row 2	5	6	7	8	$5x10^{14}$ cm^{-2}.
Row 3	9	10	11	12	$1x10^{15}$ cm^{-2}.
Row 4	13	14	15	16	$2x10^{15}$ cm^{-2}.
Fluorine = Dose	$5x10^{16}$ cm^{-2}	$1x10^{16}$ cm^{-2}	$5x10^{15}$ cm^{-2}	$1x10^{15}$ cm^{-2}	

III.3.1 UNANNEALED SAMPLE MEASUREMENT

Photoluminescence measurements were conducted with these 16 samples. The samples were cooled to liquid nitrogen temperatures and pumped with 80 mW from a 980 nm diode laser. The germanium liquid nitrogen-cooled North Coast detector was used in the measurements. The measurement system was set up as described in Section II.1. A strongly luminescent piece of Er-doped GaN was also in the cryo-chamber for alignment and comparison. All samples were measured with 2 mm input and output slit widths on the grating monochromator. Some of the samples were also measured with narrower slits in order to find additional structure in the luminescence spectra, but no additional structure was observed.

Weak luminescence (compared to the Er:GaN) was found in all of the samples. The luminescence was relatively broad in wavelength; beginning at approximately 1520 nm and tapering off slowly at 1595 nm (where our band-pass filter cuts off). Within the broad luminescence, three peaks were observed at 1534 nm, 1545 nm, and 1565 nm (the 1565 nm peak is less reliable). The 1545 nm peak is the strongest peak. However, in many of the most luminescent samples, the 1534 nm peak is nearly as strong.

III.3.2 UNANNEALED SAMPLE ANALYSIS

The grid rows 2 & 3 with Er dose $5x10^{14}$ and 10^{15} cm^{-2} show a trend of increasing luminescence as the F co-dopant density is increased (Figures 6-7). Row 1 with the lowest Er dose (10^{14} cm^{-2}) has the greatest luminescence when there is 100 times more F in the sample (Figure 8). While Row 4 with a $2x10^{15}$ cm^{-2} Er dose has the lowest measured luminescence peaks when there is $5x10^{15}$ cm^{-2} F (Figure 9). There was no discernible pattern for constant F densities and varying Er incorporation.

Samples 4, 7, and 10 all have an F:Er dose ratio of 10:1. As the sample number increases, so do the doses. As seen in Figure 10 for the unannealed samples, as the dose increases, the luminescence increases. This suggests that for a 10:1 ratio of F to Er, higher doses would produce stronger luminescence. However, while there is no 10:1 ratio at the $2x10^{15}$ cm^{-2} Er dose level, the data suggests that the luminescence will be reduced. Also, the annealed data show a different trend.

Samples 8, 11, 12, 15, and 16 have near equal doses of Er and F (Figure 11). These same samples also have the most nearly equal intensities of the 1545 nm and 1534 nm peaks (Figure

12). It is difficult to devise an explanation for this trend. These 5 samples include the most and least luminescent samples of the 16 studied.

Sample 2 is unusual. It is the only sample which has a large difference in its PL signal at the two different wavelengths, 1545 nm and 1534 nm.

III.3.3 ANNEAL AND MEASUREMENT

Other researchers have shown that most of the Er implanted in Si is optically inactive. In order to activate more of the Er ions, the material must be annealed. It is believed that the anneal procedure allows the co-dopant atoms to move around in the lattice and interact with the Er atoms. The anneal also repairs damage done to the Si lattice by the ion implantation. The lattice damage can result in non-radiative recombination or reduced pumping efficiency. Thus annealing is required for reasonable quality material.

With facilities provided by Prof. B. Van Zeghbroeck at the University of Colorado, the 16 samples from the wafer were annealed for 30 minutes at 875 ^{o}C in a nitrogen atmosphere. These annealed samples were then measured for PL in the same fashion (Figure 12). While none of the samples had as strong a peak signal as the GaN sample, several of them appear to have comparable integrated intensity. None exhibited the normally seen large Er peak at 1537 nm as do the GaN:Er samples.

III.3.4 ANNEALED SAMPLE ANALYSIS

The effects of the anneal on the spectral shape were surprising. The samples lost almost all structure to the luminescence. The peaks of the luminescence were generally at 1545 nm, the same as the strongest peak in the unannealed samples (Figures 6-9). The FWHM for these luminescence peaks is approximately 75 nm. On several measurements the slits of the monochromator were narrowed to attempt to find more structure in the spectra, but with no success. It is possible that the temperature was not 77K during these measurements, since our thermocouple was inoperative. A higher temperature would tend to broaden the spectrum. However, given the time interval between the reservoir being filled with liquid nitrogen and the measurements, we doubt this explanation.

After annealing, samples 8, 12, and 16 all had a slight photoluminescence peak at 1530 nm (Figure 13). While this peak is close to typical peak wavelengths for Er-doped Si, the spectra of these samples still lacks as much structure as is usually seen. All of these samples had the lowest dose of F, 10^{15} cm^{-2} and $5x10^{14}$ cm^{-2}, $1x10^{15}$ cm^{-2}, and $2x10^{15}$ cm^{-2} Er doses respectively. Surprisingly, the strongest PL came from the most heavily Er-doped sample, #16.

In the samples with equal Er doses, the most luminescent sample was the sample with a $5x10^{15}$ cm^{-2} F dose (7, 11, and 15), except for sample 2 with Er dose of $1x10^{16}$ cm^{-2}. In all columns of equal F density the greatest PL is exhibited by the sample with the lowest Er dosage (Row 1, Figure 8). This suggests that the optimal Er dose is smaller than that used for these samples. Additionally, Figure 10 shows that for samples 4, 7, and 10 with 10:1 F to Er ratios, a smaller Er dose resulted in a stronger luminescence intensity. This is opposite the trend of the unannealed case.

III.3.5 DISCUSSION

It is difficult to find a pattern in the luminescence results from the unannealed samples. Ignoring the anomalous sample 2, the optimal luminescence appears to occur when the Er dose is $1x10^{14}$ cm^{-2} and the F concentration is 10^{15} cm^{-2} to 10^{16} cm^{-2}. Higher Er concentrations may exceed a concentration quenching threshold. Higher luminescence from a lower co-dopant dose is not surprising in unannealed samples since the co-dopant probably does very little to aid luminescence without an anneal to position the atoms; and the implantation of the co-dopant atoms induces significant damage to the Si lattice.

The annealed samples generally photoluminesce much more strongly. The anneal unquestionably results in more optically active sites. Also, the luminescence is clearly being quenched by the high Er concentrations. An apparent reduction in the concentration quenching could be due to larger Er defect sites when one includes the surrounding F atoms. This would

effectively reduce the distance between defect sites and increase concentration effects. Annealed samples 8, 12, and 16 do not follow the concentration quenching trend. This can be explained by their relative lack of F. Less F means few of the Er defect sites will be expanded by surrounding F co-dopant and, at the same time, few Er atoms will be made optically active by the co-dopant.

The unusual shape of the Er luminescence in the annealed samples may be the result of an incomplete anneal. A longer anneal, possibly at a higher temperature, may narrow the luminescence peak. An additional anneal for 10 minutes at 1100 C is planned. Also, Dr. F. Namavar is producing another wafer grid of varying Er and F implantations. This next grid will have lower Er implantation doses than the samples just studied. This should produce more luminescent samples and allow more careful study of concentration quenching.

IV. ERBIUM DOPED GALLIUM NITRIDE

Three separate sets of Er implanted GaN films have been investigated. The GaN films were grown on R-plane sapphire at RCA, NJ (1970's) and on the basal plane by Astralux Inc., Boulder CO. One of the sets were implanted with Er^{2+} only, while the other two sets were co-implanted with Oxygen. The CL spectra were measured, and a variety of inner 4f shell Er^{3+} related peaks were identified. Er related luminescence was found in the IR region at 1540 nm from the GaN films. In addition, there were signals seen from the IR, throughout the visible, and into the UV part of the spectrum from Er doped sapphire. Only the 1540 nm line was seen in the GaN. The temperature dependence of the 1540 nm GaN:Er peak was studied. The intensity of the 1540 nm peak decreases only a few percent from 6 K to room temperature. Also the 410 nm peak of Sapphire:Er was studied, and thermal quenching was found to be minimal (20% at room temperature [Qiu].

IV.1 MATERIAL PREPARATION

IV.1.1 IMPLANTATION:

The implantation was done at Spire Corporation, by Dr. F. Namavar. Two different implantations were performed; one set with Er^{2+} only, and two sets with both Er^{2+} and O^+ as shown in Table IV below. The implantation depth was calculated to peak at 0.15-0.2 μm from the surface, with a FWHM of about 9 nm. Note that the O^+ implantation energy is smaller that of Er^{2+}. The implantation energies are calculated to make the Er and O peak concentrations overlap.

Table IV. GaN Implantation conditions:

RCA samples:	
#67539	2×10^{14} Er^{2+} /cm^2 at 400 keV
#67547	2×10^{14} Er^{2+} /cm^2 at 400 keV
#67555	2×10^{14} Er^{2+} /cm^2 at 400 keV
#384	10^{15} Er^{2+} /cm^2 at 400 keV, and 10^{16} O^+/cm^2 at 80 keV
Astralux samples:	
GN 88	10^{15} Er^{2+} /cm^2 at 400 keV, and 10^{16} O^+/cm^2 at 80 keV
GN110	10^{15} Er^{2+} /cm^2 at 400 keV, and 10^{16} O^+/cm^2 at 80 keV
GN160	10^{15} Er^{2+} /cm^2 at 400 keV, and 10^{16} O^+/cm^2 at 80 keV
GN166	10^{15} Er^{2+} /cm^2 at 400 keV, and 10^{16} O^+/cm^2 at 80 keV
GN171	10^{15} Er^{2+} /cm^2 at 400 keV, and 10^{16} O^+/cm^2 at 80 keV
GN66031	10^{14} Er^{2+} /cm^2 at 400 keV, and 10^{16} O^+/cm^2 at 80 keV
#092393	10^{15} Er^{2+} /cm^2 at 400 keV, and 10^{16} O^+/cm^2 at 80 keV

IV.1.2 ANNEALING

The annealing was performed by Astralux Inc. in the metal organic CVD reactor in an ammonia atmosphere to prevent GaN decomposition. The samples were cleaned in trichlorethylene, acetone, isopropyl alcohol and HF acid before being loaded into the reactor. The

chamber was pumped down to 10-20 Torr. A load-lock was used to transfer the samples into the evacuated chamber. The chamber was then heated to the final annealing temperature as shown below in Table V below.

IV.2 OPTICAL MEASUREMENTS

IV.2.1 CATHODOLUMINESCENCE OF GN 166 and GN 66031

The CL was performed in the system described in Section II.2 above. The IR luminescence measurements were performed using a dry-ice cooled PbS detector, and the visible/UV spectra were obtained using a RCA 31025C GaAs photomultiplier.

Table V. GaN Annealing conditions:

RCA samples:		Astralux samples:	
#67539	900°C at 20 Torr for 0.5 hour	GN 88	900°C at 10 Torr for 1.0 hour
#67547	900°C at 20 Torr for 0.5 hour	GN110	950°C at 20 Torr for 1.0 hour
#67555	900°C at 20 Torr for 0.5 hour	GN160	950°C at 20 Torr for 1.0 hour
#384	900°C at 10 Torr for 1.0 hour	GN166	950°C at 20 Torr for 1.0 hour
		GN171	950°C at 20 Torr for 1.0 hour
		GN66031	950°C at 20 Torr for 1.0 hour
		#092393	950°C at 20 Torr for 1.0 hour

The GaN samples did not show any Er luminescence before they were annealed. After annealing many of the GaN samples exhibited strong Er related CL lines at 1540 nm. Some of the sapphire substrates were only partially covered by GaN, and consequently these sapphire areas were also implanted with Er and O. CL from these areas (Sapphire:Er:O), yielded detailed spectra from the IR and up through the UV, with numerous transitions attributed to Er (Figure 14). The different spectral responses of the detectors has not been taken into account. All the samples with luminescence had similar spectra. The most extensively studied sapphire:Er samples were GN 166 and GN 171, since they yielded the strongest CL signal. Figure 15 shows a typical spectra of GaN:Er found in sample GN 66031. These results are published in the paper by Qiu et al. [Qiu].

IV.2.2 INTENSITY COMPARISON FOR SAPPHIRE:Er:O

The luminescence of all the implanted samples was measured and compared. Experimental parameters such as accelerating voltage, beam current and slit width are the same in this comparison. The spectral peak measured is at 3.02 eV (0.41 μm), which was the strongest visible peak. The results are shown below in Table VI.

Table VI. Relative PL intensity:

	Intensity relative to GN 166:	Oxygen co-implant
RCA samples:		
#67539	0.08	No
#67547	0	No
#67555	0	No
#384	0	Yes
Astralux samples:		
GN 88	0	Yes
GN110	0.44	Yes
GN160	0	Yes
GN166	1	Yes
GN171	0.64	Yes
#092393	0.42	Yes

IV.2.3 HIGH RESOLUTION SPECTROSCOPY OF SAPPHIRE:Er:O

The high resolution CL spectra yielded more detail (Figure 16). By reducing the slit width we were able to resolve several more peaks, but this also reduced the signal power. These studies were performed with slit widths ranging from 80 μm to 150 μm. The previous spectra in figures 14 and 15 were taken with a slit width of 500 μm. The spectral differences may be due to the Kramer degeneracy [Ennen] being split and/or the Er ion sitting in both interstitial and substitutional lattice sites. We measured the FWHM of the strongest line to be 6 meV. We are limited in this measurement by our monochromator and detector.

Several more samples have been sent to Spire Corporation for Er and O implantation. Both GaN films and bare sapphire substrates have been included to investigate the observed spatial dependence.

IV.2.4 PHOTOLUMINESCENCE OF GN 166

PL of GaN:Er was measured at 1530 -1540 nm when excited by an Argon laser at NIST in Boulder with the cooperation of Dr. Kevin Malone. The 514 nm line was used, and the pump power was 4.3 Watts. The signal found was very weak, but clearly out of the noise level as shown in Figure 17. The CL signal was strong, while the PL signal was weak because of the 10^4 higher capture cross section of Er^{3+} for impact excitation compared to optical excitation [Payne and Yu].

IV.2.5 THERMAL QUENCHING OF Er LUMINESCENCE

The temperature dependence of the 0.80 eV (from GaN) and 3.02 eV (from Sapphire) peaks were studied in sample GN 166 (See figure 18). The CL of the two peaks was measured as a function of temperature from 6 K to 400 K. The intensities were normalized to the strongest intensity measured. The IR peak was measured using the dry-ice cooled PbS detector, while a photomultiplier was used for the purple peak.

IV.3 OPTICAL WAVEGUIDE ISSUES

IV.3.1 WAVEGUIDE FABRICATION

There are several issues to be resolved in the fabrication of optical quality rib waveguides in GaN/sapphire. As a starting point we need GaN films with a small number of crystalline defects, fairly uniform thickness, and a reasonably precise thickness. These growth issues are being addressed by Astralux Inc. and by Prof. Morkoc's group at the University of Illinois, both of whom have grown GaN/sapphire.

With the films in hand, we have to: 1) polish the surface to an optical finish of much less than 100 nm surface roughness without making the film non uniform, 2) etch the rib waveguides in GaN with a good reproducibility, and 3) be able to etch or polish good optical quality endfaces on the guides for coupling light in and out.

IV.3.2 POLISHING

We have been able to polish the surface of the GaN films to an acceptable optical quality (Figure 19). The roughness after polishing is approximately 100 nm, which with an index of refraction of about 2.0 at 1540 nm, is approximately $\lambda/8$. Polishing the edges is more difficult since the GaN film tends to flake off. We use a Beuhler Inc. wafer polisher, and polish with diamond paste. The procedure is:

1) 3 mm diamond paste, 120 rpm, ~15 min, 8 lbs
2) 1 μm diamond paste, 120 rpm, ~15 min, 8 lbs
3) 0.25 μm diamond paste, 120 rpm, ~15 min, 8 lbs
4) 0.05 μm Al_2O_3 mixture, 120 rpm, ~15 min, 8 lbs (if needed)

IV.3.3 REACTIVE ION ETCHING

We have made several etches of GaN/sapphire with a PlasmaTherm Reactive Ion Etching

Chamber using a Freon 12 (CCl_2F_2) gas in Prof. Van Zeghbroecks lanboratory at CU (Figure 20). We have consistently achieved etch rates of about 45 nm/min, which is consistent with Adesida et al. [Adesida]. The etch parameters are usually as follows:

Power = 500 W	RF Frequency = 13.56 MHz
Pressure = 100 mT	Flow = 10 sccm
DC bias = -450 V	Base Pressure < $5x10^{-5}$ Torr.

The pattern transfer is done with conventional optical photolithography, with or without an evaporated Cr layer evaporated on the GaN depending on the desired etch depth. If a deep etch (> 1 μm) is needed, a Cr etch mask is employed. If the etch to be done is less than 1 μm, hard baked photoresist is used as the etch mask (etch rate ~80 nm/min).

Although the GaN can be etched, we have had a problem with "grass" formation during the etching. To prevent sputtering of the aluminum electrodes and redeposition on the samples, teflon and quartz plates were made to cover the electrodes in the RIE chamber. This reduced the grass formation somewhat. Using the Cr masks also seemed to promote the grass formation, so a pure photoresist (PR) mask is preferred. Even with the PR mask grass is formed, and we are currently working on varying the etch parameters to reduce this phenomenon.

IV.3.4 ENDFACES

Making optical quality endfaces for coupling light into and out from the waveguides has proven to be a challenge. We have made a few attempts at the polishing of the endfaces (Figure 21), but to date we have not sufficient high quality films to study. One problem with the films is the strain between the GaN film and the sapphire substrate. Thus the GaN tends to flake off when polished. This is expected to be improved with GaN films grown with an AlN buffer layer, that reduces the strain.

IV.3.5 WAVEGUIDE SIMULATIONS

We have performed waveguide simulations of GaN rib waveguides on Sapphire using the effective index method. The waveguide simulator is a C program re-written by Lee Nichols [Nichols] and based on Chanbeta, a software routine developed by Prof. Mickelson's Guided Wave Optics Laboratory here at the University of Colorado. We have restricted the study to include rib waveguides with the dimensions compatible to optical fibers. That means fixing the width of the rib guide to approximately 8-9 μm. The free parameters are the etch depth and the thickness of the GaN film (Figure 22).

Since the index of refraction differences of the materials used are large (n_{GaN}~ 2.3, $n_{sapphire}$ = 1.8, and n_{air} = 1 all at 1540 nm) the optical power is strongly confined to the GaN layer in the vertical dimension. However, the large index change at the material interfaces can also lead to a highly multi-mode structure at desirable waveguide dimensions (Figure 23). To achieve single mode waveguides with widths of 8-9 μm, would require extremely thin films, which leads to inefficient coupling in of the pump laser. Thus optically pumped devices in the GaN/sapphire material system will probably be multi-mode. This problem can be avoided if the material system is changed to, for example GaN/AlGaN/sapphire, where the refractive index changes are less dramatic.

For an electrically pumped laser, the pump coupling problem does not exist, so the GaN/sapphire system might still be used.

To accurately simulate and make these GaN on sapphire waveguides there are several materials issues that have to be resolved. We have to know the refractive index of the GaN films at the design wavelength (1540 nm), and we have to be able to physically fabricate the rib waveguides.

IV.3.6 REFRACTIVE INDEX MEASUREMENTS

We need to know the index of refraction of the GaN films quite accurately to be able to predict the waveguide performance. The most accurate method of measuring the index of

refraction of a thin film is ellipsometry at 1540 nm. We therefore inquired with Prof. Woollam at Woollam Inc. in Lincoln, Nebraska about a collaboration, which he generously accepted. He agreed to measure the index of refraction of 3-4 films of GaN/sapphire before and after Er and O implantation. We sent him 3 GaN/sapphire films grown by Astralux Inc., and 1 GaN/sapphire film grown by Prof. Morkoc's group at University of Illinois. The films grown by Astralux had been polished prior to being sent to Woollam Inc., since they typically have a few 100 nm's surface roughness (Figure 19). The sample from Prof. Morkoc had not been polished. The ellipsometry measurements were not successful due to surface roughness and thickness nonuniformities in the films. The other samples from Astralux Inc. did not yield any results due to thickness nonuniformities from growth. Ellipsometry is a polarization-sensitive measurement and is therefore very sensitive to thickness nonuniformities, and the accompanying software is only capable of handling slight surface roughness. We therefore proceeded to polish Prof. Morkoc's sample, and returned the sample for further ellipsometric studies. We expect to have the results shortly.

We have also measured the index of refraction by studying the interference fringes from a reflection measurement. We used a Cary Inc. Spectrophotometer, and measured the reflection spectrum of a couple GaN/sapphire films in the range 300 nm to 2500 nm. We studied Prof. Morkoc's sample (n-type), and a GaN/sapphire (p-type) sample grown by Prof. I. Akasaki. In this measurement which is performed at close to normal incidence, where the polarization effects are negligible, we observed many interference fringes. We used the standard equations for the intensity maximum,

$$2\, d\, n(\lambda) = \lambda\, (m+1/2), \tag{1}$$

where m is an integer, d is the film thickness, and $n(\lambda)$ is the index of refraction, and for the intensity minimum,

$$2\, d\, n(\lambda) = m\, \lambda. \tag{2}$$

Using the measured λ_{max} and λ_{min} values, and fitting the data to previously published GaN index data (in the visible on an n-type sample) by I. Akasaki [Akasaki], we were able to determine the index of refraction in the near IR by matching the index at 2.5 eV (Figure 24). Note that both the samples grown by Prof. Morkoc and Prof. Akasaki show a decreasing index of refraction in the IR. We think this might be due to free carrier absorption, but we avait confirmation of this trend from the ellipsometry measurements.

This method of determining the index of refraction is not as accurate as ellipsometry, but provides a good fit to Akasaki's data where the data overlap. We hope to verify the validity of our data with the ellipsometry measurements. We are currently working on measuring additional films.

IV.4 DISCUSSION

CL measurements have been performed on several Er implanted GaN samples yielding a detailed spectra with multiple peaks attributed to to Er^{3+} related luminescence. The location of the spectral peaks are similar in all samples, but they vary in intensity. Thermal quenching has been investigated, and we found that the intensity of the IR 1540 nm (0.81 eV) peak is only reduced by a few percent at room temperature with respect to the intensity at 6 K. Currently we are in the process of fabricating GaN:Er waveguides using reactive ion etching. Following Adesida et. al. [Adesida], we are using a chlorine-based gas which will react with the gallium to form gallium chloride. We will characterize the etching using a CCl_2F_2 plasma, while Adesida et al. used silicon tetrachloride ($SiCl_4$). Waveguide loss measurements with and without Er will be performed. PL with several pump wavelengths (1485 nm, 980 nm, 514 nm) will be investigated.

V. OTHER WORK

Wang and Wessels [Wang] at Northwestern University have measured thermal quenching of Er in GaP. They theorize that the luminescence falls with increased temperature because of Er-related isoelectronic trap energies. The paper proposes that bound excitons are an intermediary in the excitation of the Er centers. As temperature increases, the bound excitons are more likely to break up. This offers an explanation for the poor luminescence of high photon energy pumped Er:Si at room temperature. Co-doping the samples to form Er compounds may increase the luminescence in Si by increasing the depth of the trapping energy level, resulting in stronger binding of the excitons. The work of Franzo et al. [Franzo] also showed much reduced thermal quenching in reverse biased Si:Er LED's, where the pumping is direct impact excitation, versus pair (exciton) recombination as mentioned above.

At the Electronic Materials Conference held June 22–24, 1994 at the University of Colorado at Boulder, J. Michel of MIT presented a paper entitled "Er-O and Er-F Reactions in Silicon" [Ren]. A model for optimal dopant concentrations was developed and combined with experimental results. The group concentrated on F co-doping, which was found to produce 100 times greater emission than oxygen co-doping. They also used significantly lower doping concentrations than we have. The crux of the paper was the use of annealing to optimize emission. They found that annealing a sample with $5x10^{17}$ cm^{-3} Er and $1x10^{18}$ cm^{-3} O or F at 900 C for 0.5 hours optimized emission. The optimal temperature decreased to 800 C for F concentrations of $4x10^{18}$ cm^{-3}. The process model suggested that the Er emission was optimized when the concentration of ErF_3 was greatest.

VI. SUMMARY

We have seen strong Er^{3+} related CL and weaker PL in several different GaN:Er and Sapphire:Er samples. The temperature dependence has been studied, and we found the intensity of the GaN:Er IR peak at 1540 nm to only drop by only a few percent at room temperature with respect to the signal at 6 K. The peak wavelength remains constant, but it broadens slightly as the temperature rises. These are very important results in light of future device applications. More GaN samples, grown by Astralux Inc., have been sent out for implantation, and will be studied in the near future. We are currently working on fabricating good quality single mode GaN:Er waveguides. A detailed study of reactive ion etching using a CCl_2F_2 plasma is underway. We are very optimistic about the feasibility of etching waveguides due to the recent publication by Adesida et al. [Adesida] where they etched good quality ribs in GaN using a silicon tetrachloride ($SiCl_4$) plasma. A study of cutting optical quality end faces on GaN on sapphire using a wafer saw is also in progress.

The results of measurements of Er doped Si have been poor. A new implanted wafer (provided by Dr. F. Namavar of Spire Corp.) with a checkerboard pattern of Er and F with differing concentrations of each, at each of 16 locations, will be evaluated to determine the optimum Er to F ratio. This may lead to major improvements in luminescence as Ren et al. [Ren] have noted in their work.

VII. ACKNOWLEDGMENTS

We would like to thank the following groups for their gracious collaborative efforts; Dr. F. Namavar at Spire corporation and Dr. S. Withrow at Oak Ridge National Laboratory for ion implantations, Dr. Kevin Malone at NIST for the use of their high powered Argon laser. The work with the wafer saw was possible due to the help from Ms. L. Rohlev and Prof. A. Mickelson. Further, we owe thanks to Prof. Morkoc for donating a GaN/Sapphire film, and Prof. J. P. Woollam for performing ellipsometry, and Prof. B. Van Zeghbroeck in the ECE department at University of Colorado at Boulder for the use of their reactive ion etch (RIE) system. Also we would like to thank Dr. M. Leksono and Dr. C. Qiu of Astralux Corporation for growth of GaN films and assistance in measurements. Also Ms. Carol Lee at The Army Research Lab., Ft. Monmouth, NJ, through a collaboration with C. Lee, K.K. Choi, and Wayne Chang for rapid thermal anneal.

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IX. LIST OF FIGURES

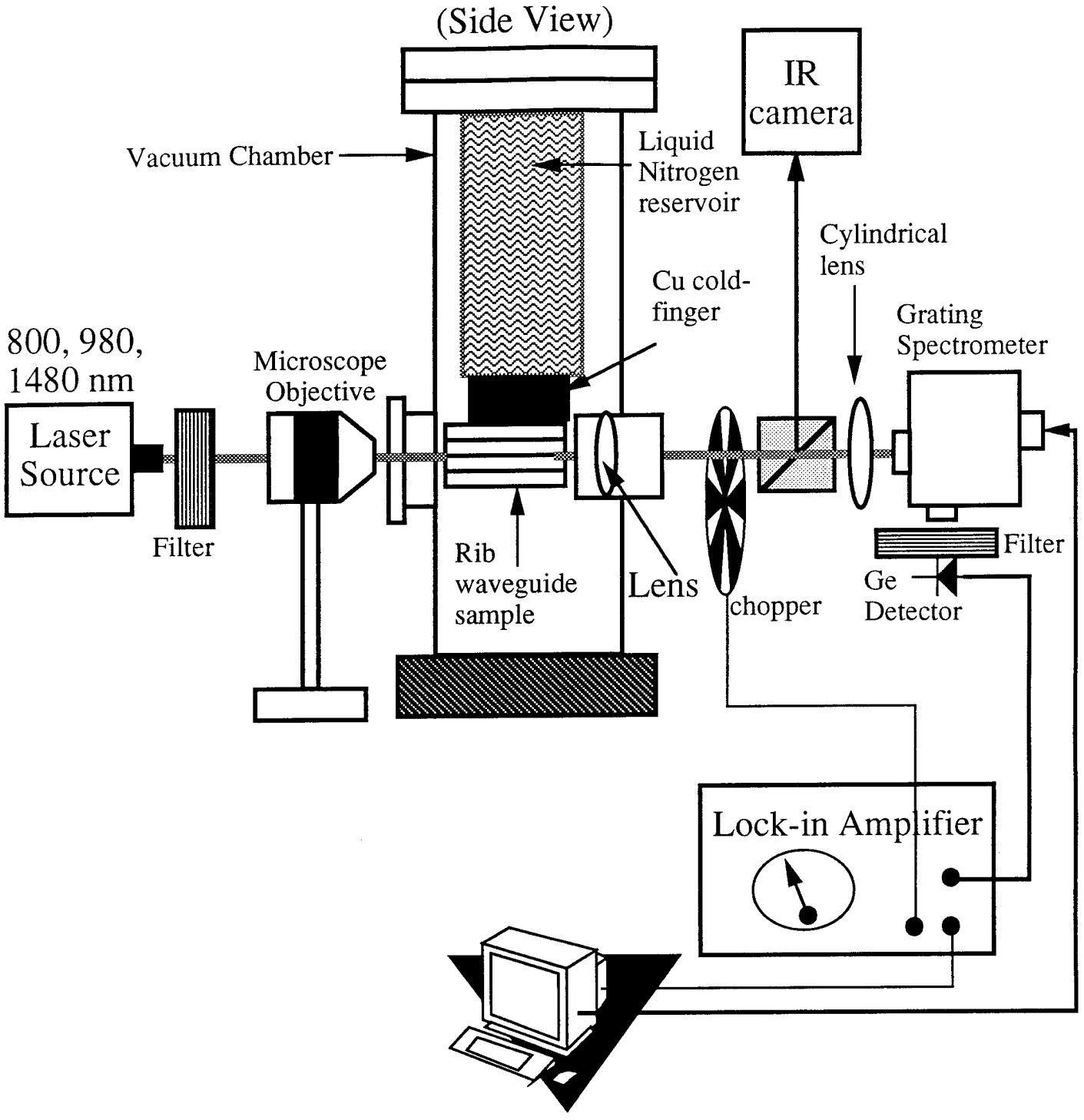


Figure 1. PL measurement system

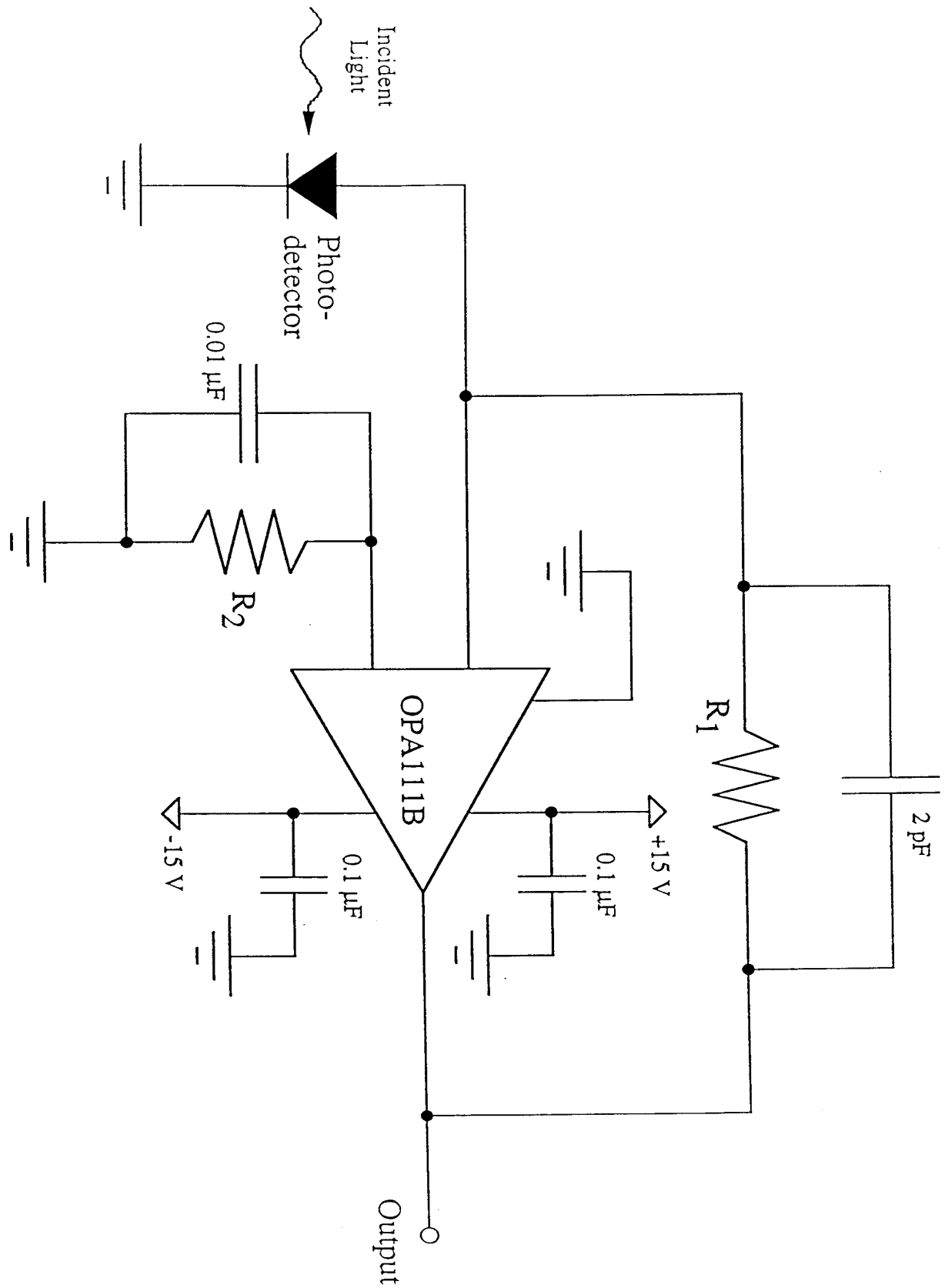


Figure 2: Photodetector Amplification Circuitry. ($R_1 = R_2 = 43$ MΩ)

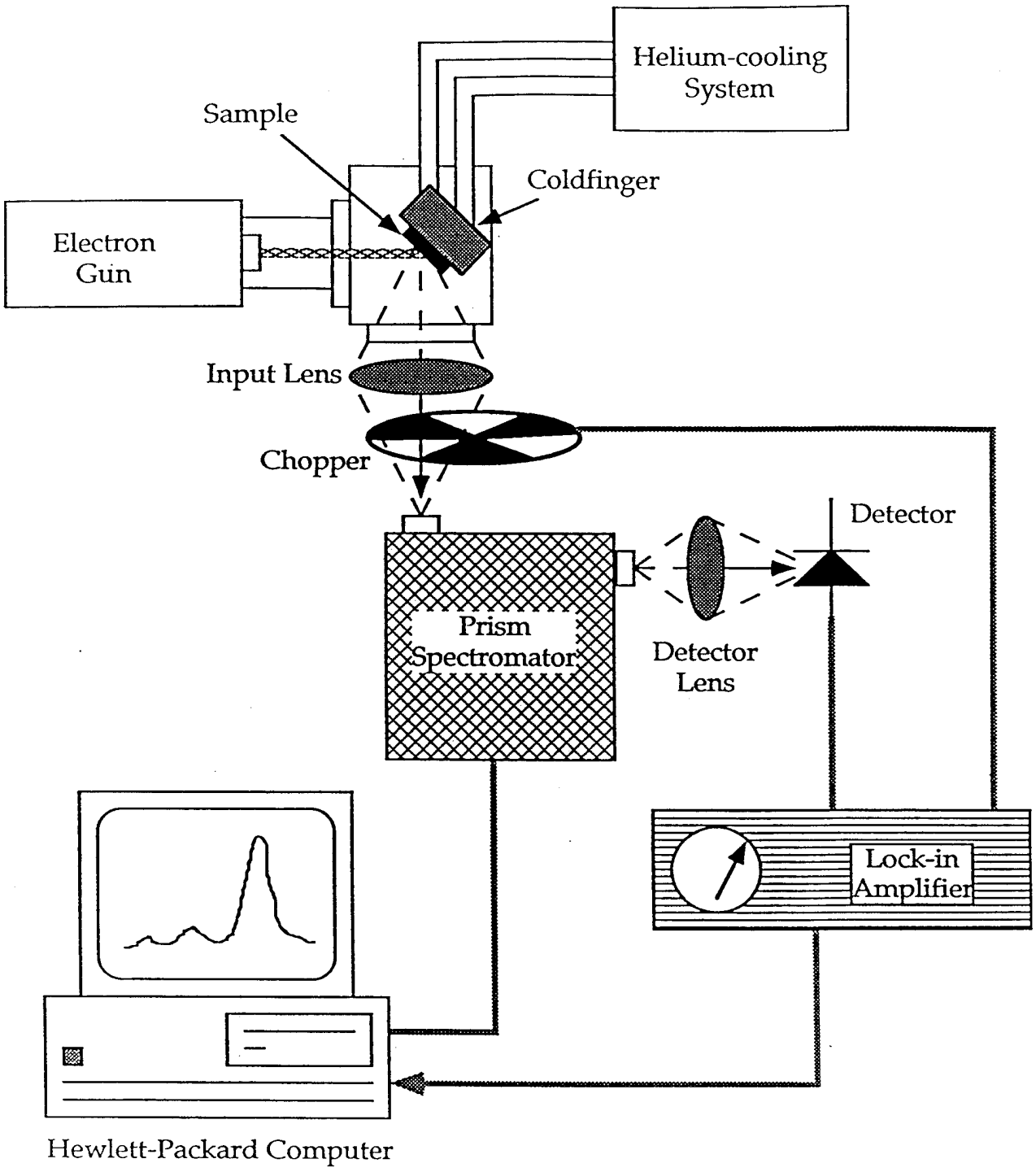


Figure 3: CL Measurement System.

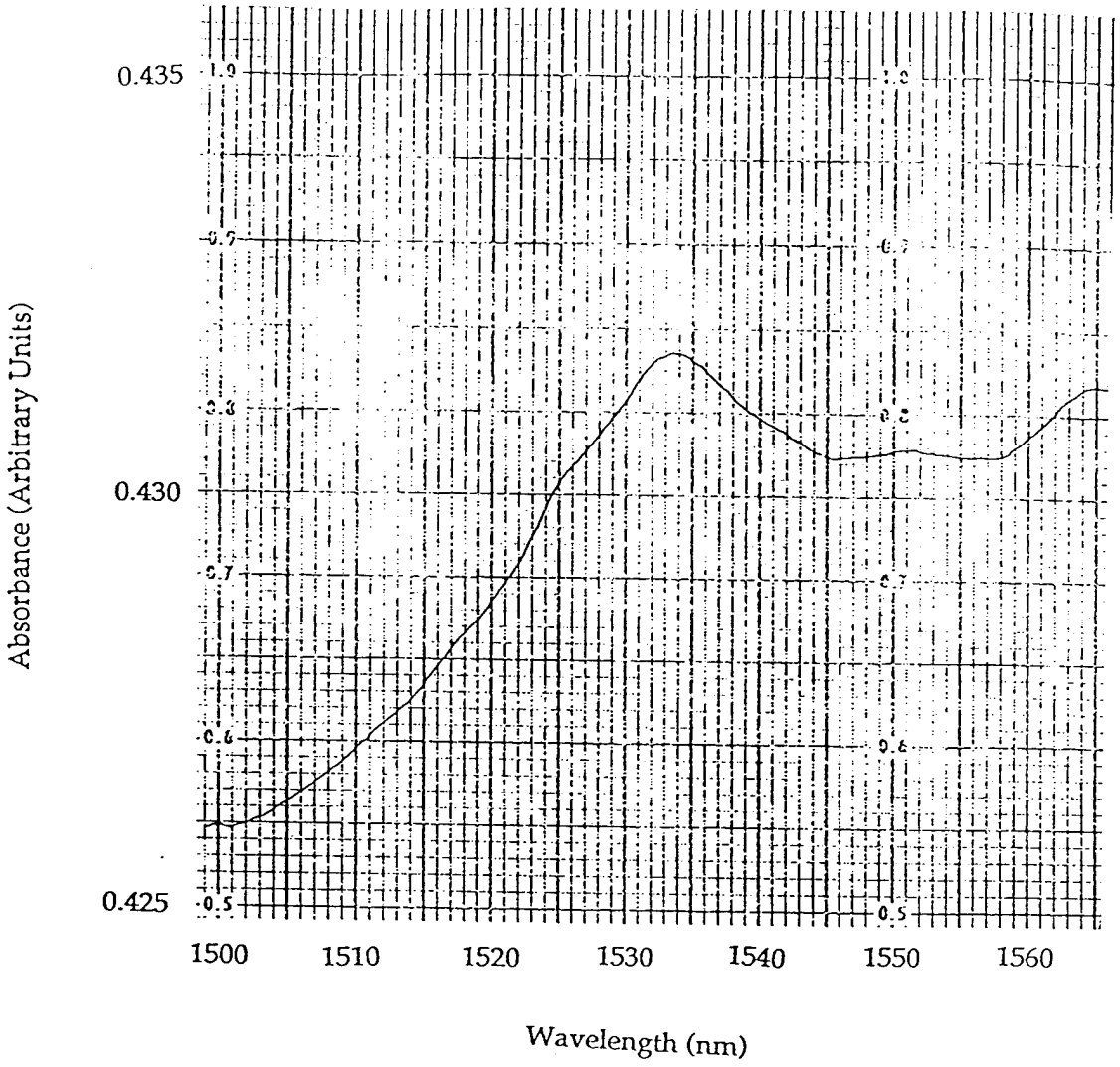


Figure 4: Absorption Measurement of Epitaxially Grown Er-doped Si

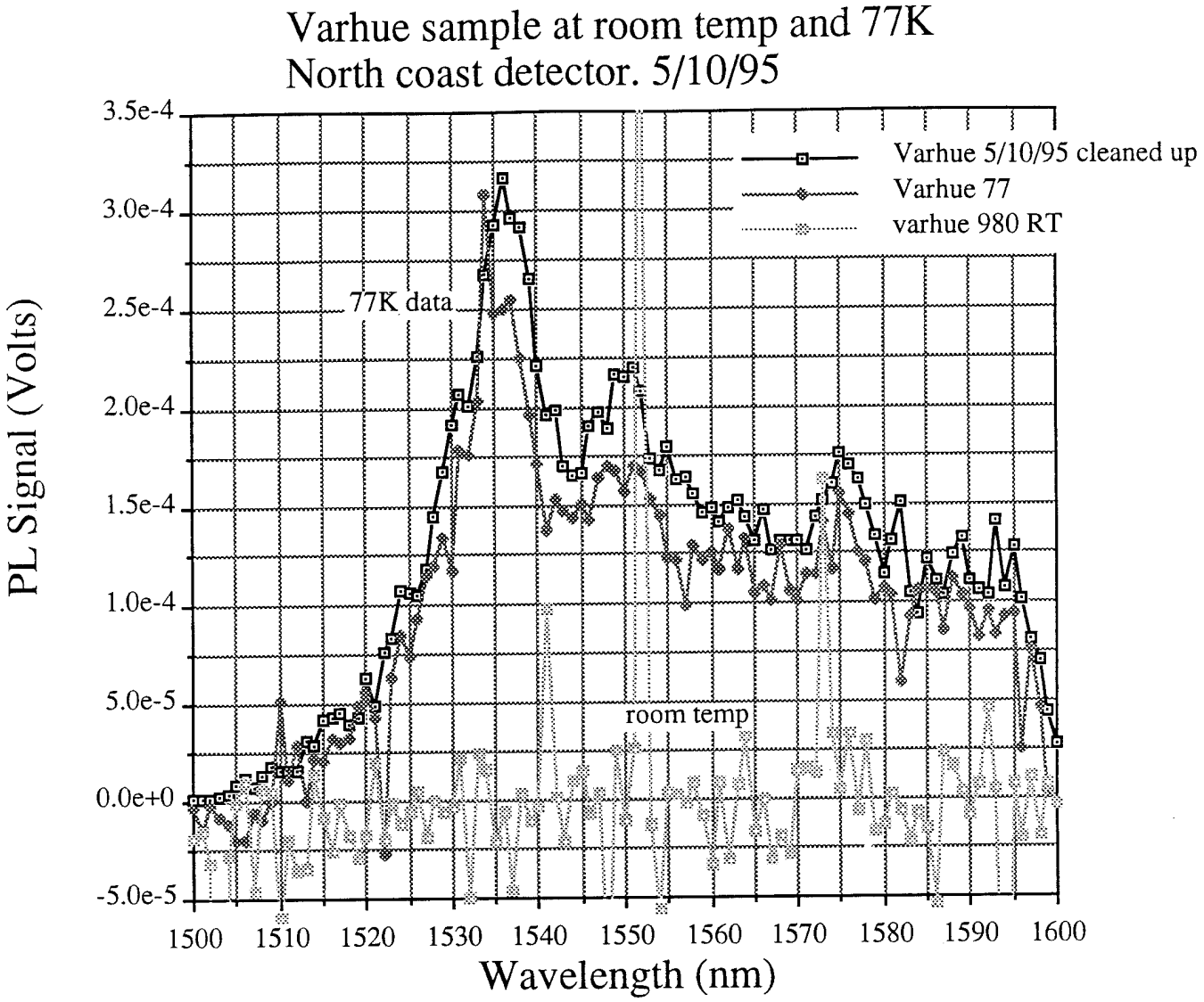


Figure 5. Varhue sample PA 49 PL spectrum with 980 nm pump laser, at 300 K and 77 K.

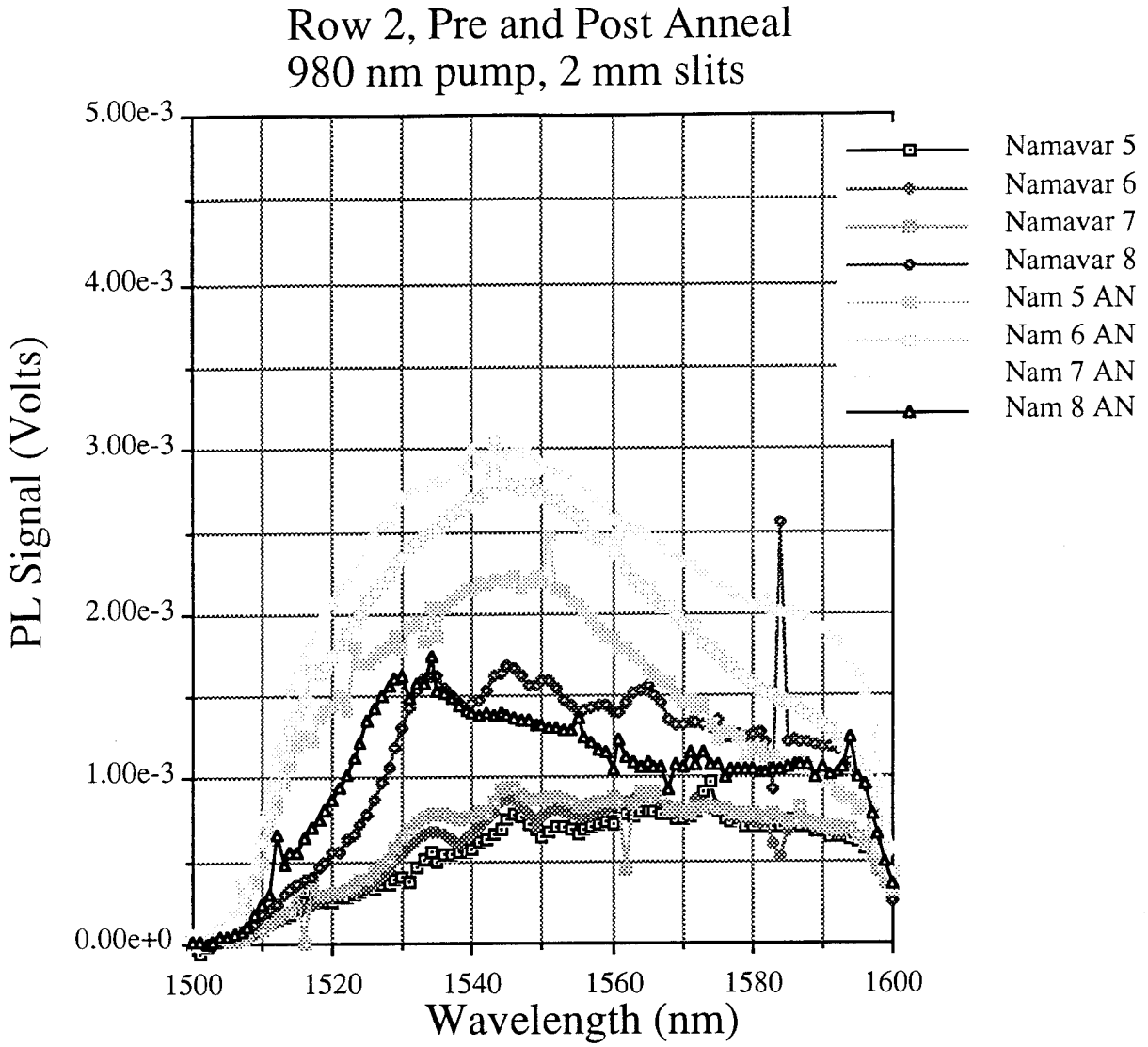


Figure 6. Row 2 PL with 980 nm pump laser, before and after annealing.

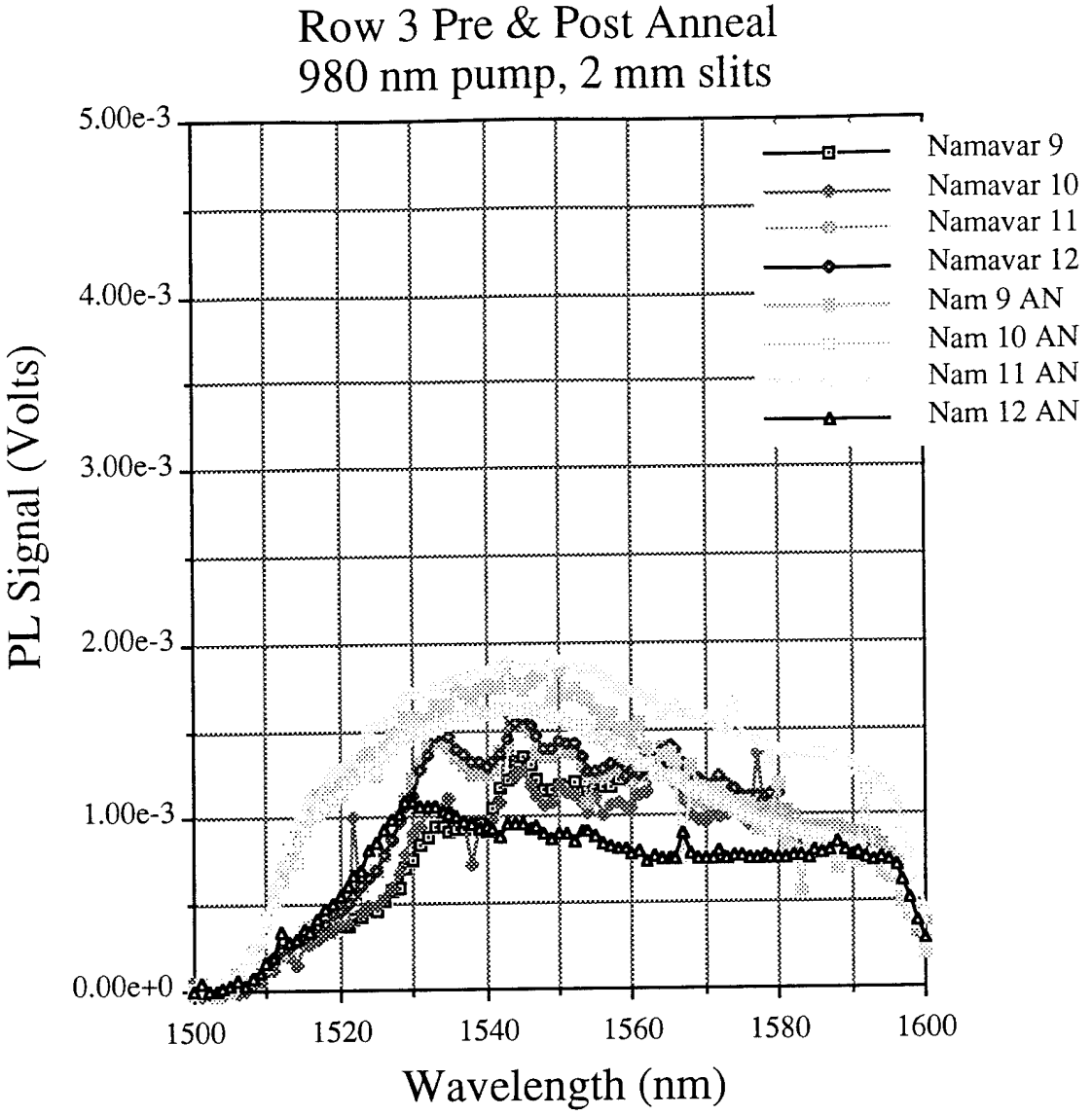


Figure 7. Row 3 PL with 980 nm pump laser, before and after annealing.

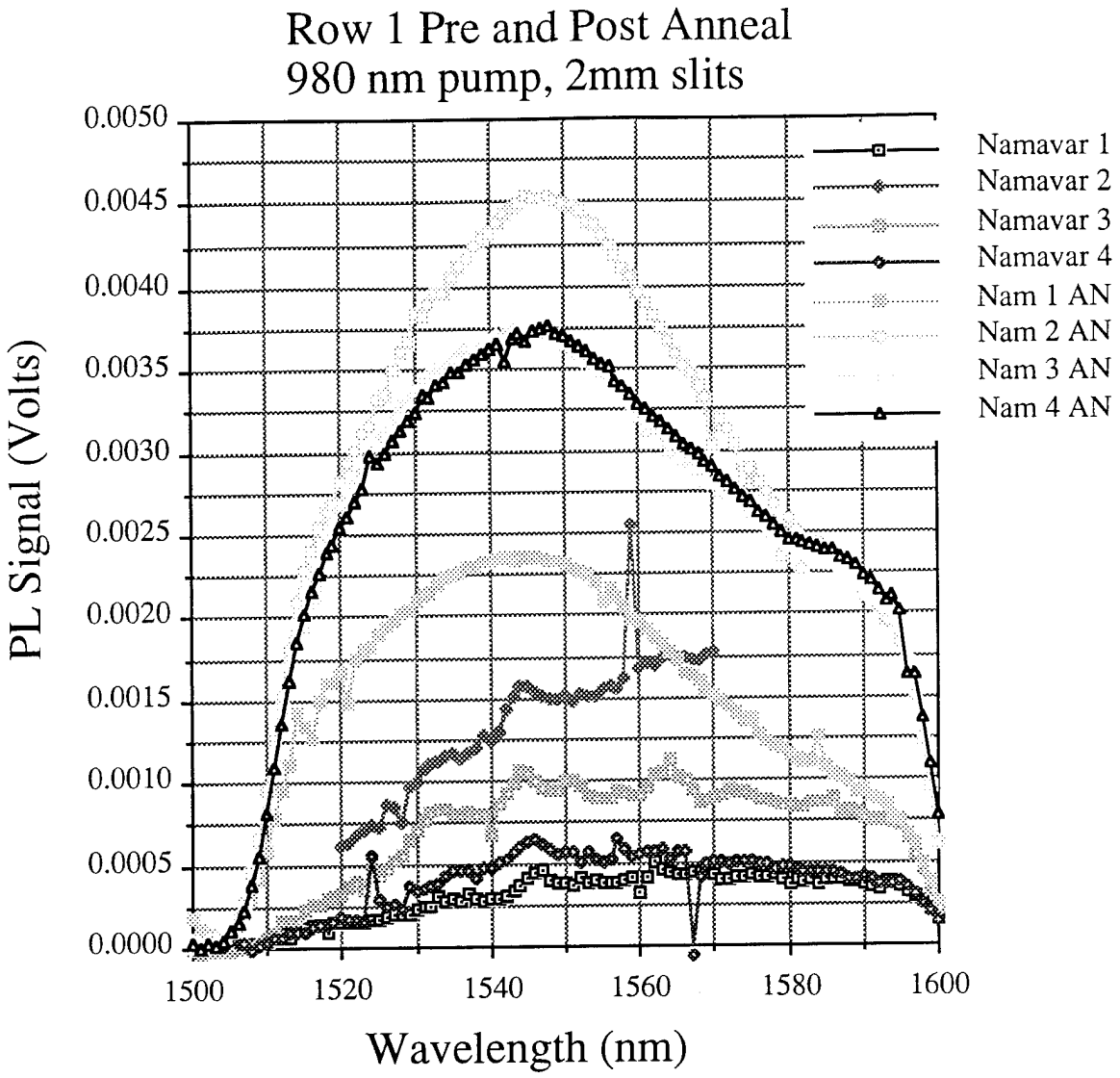


Figure 8. Row 1 PL with 980 nm pump laser, before and after annealing.

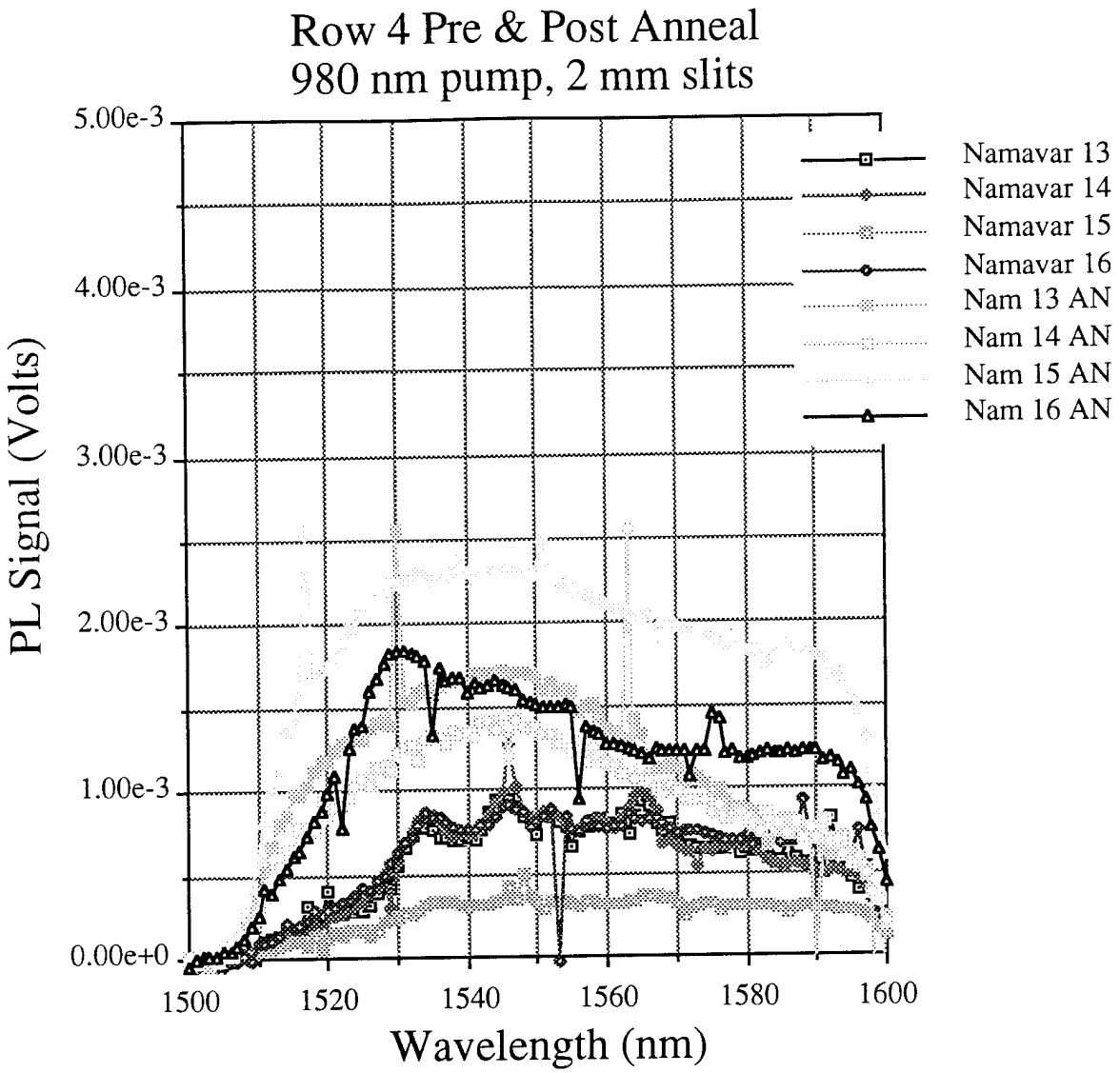


Figure 9. Row 4 PL with 980 nm pump laser, before and after annealing.

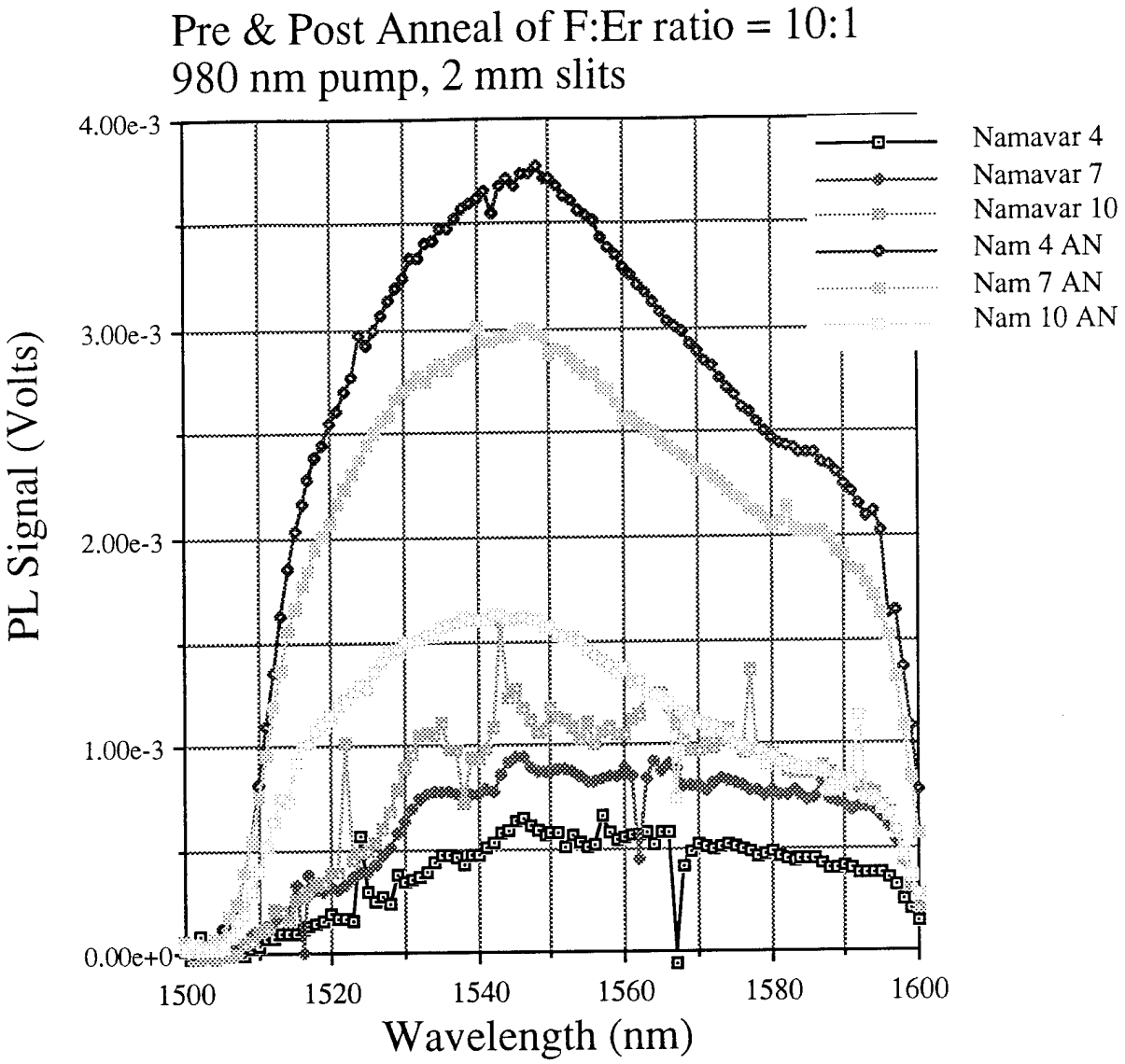


Figure 10. Samples with F:Er ratio = 10:1, samples 4, 7, and 10. PL with 980 nm pump laser, before and after annealing.

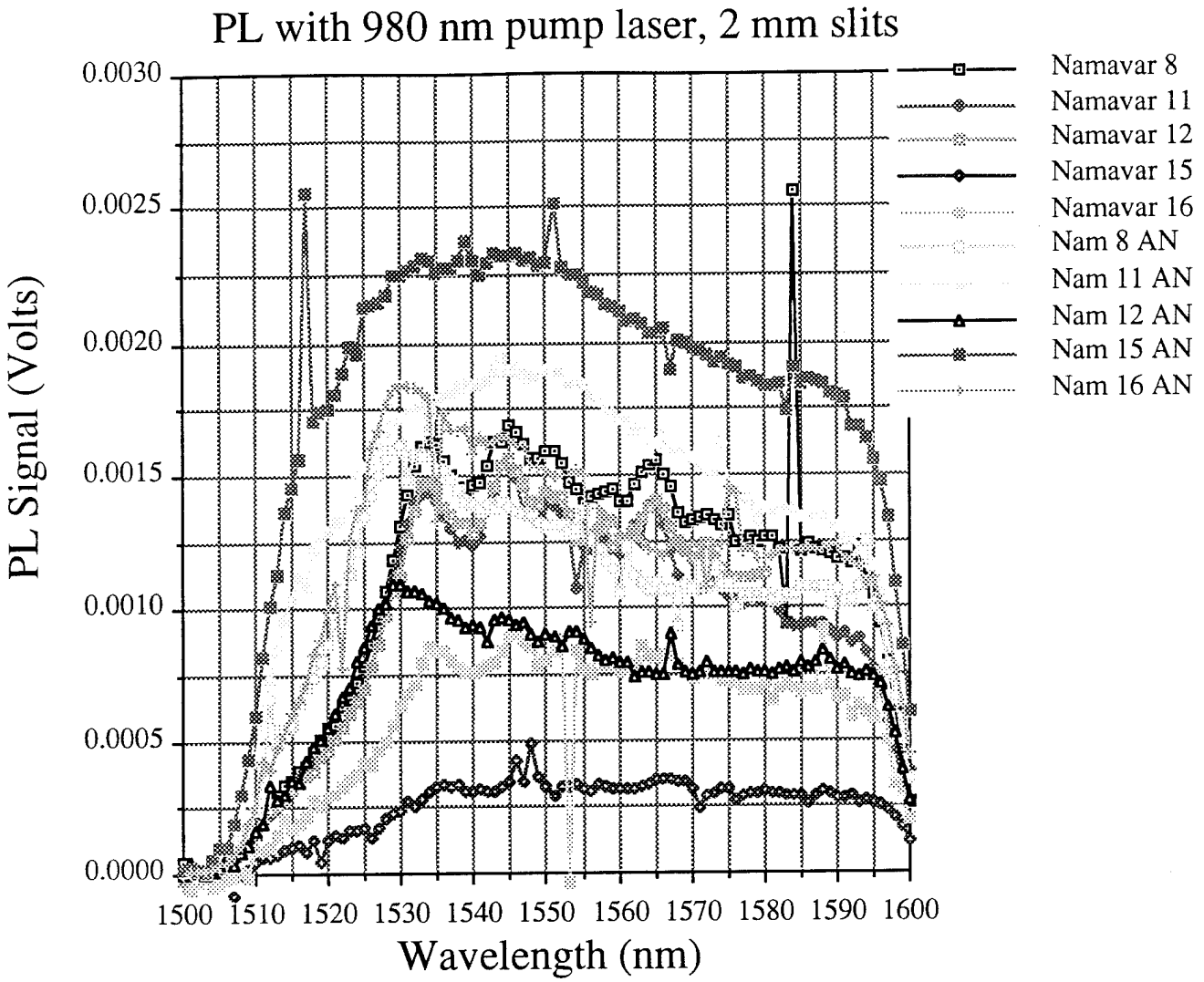


Figure 11. Samples with F:Er ratio nearly = 1:1. Samples 8, 11, 12, 15, and 16. PL with 980 nm pump laser, before and after annealing.

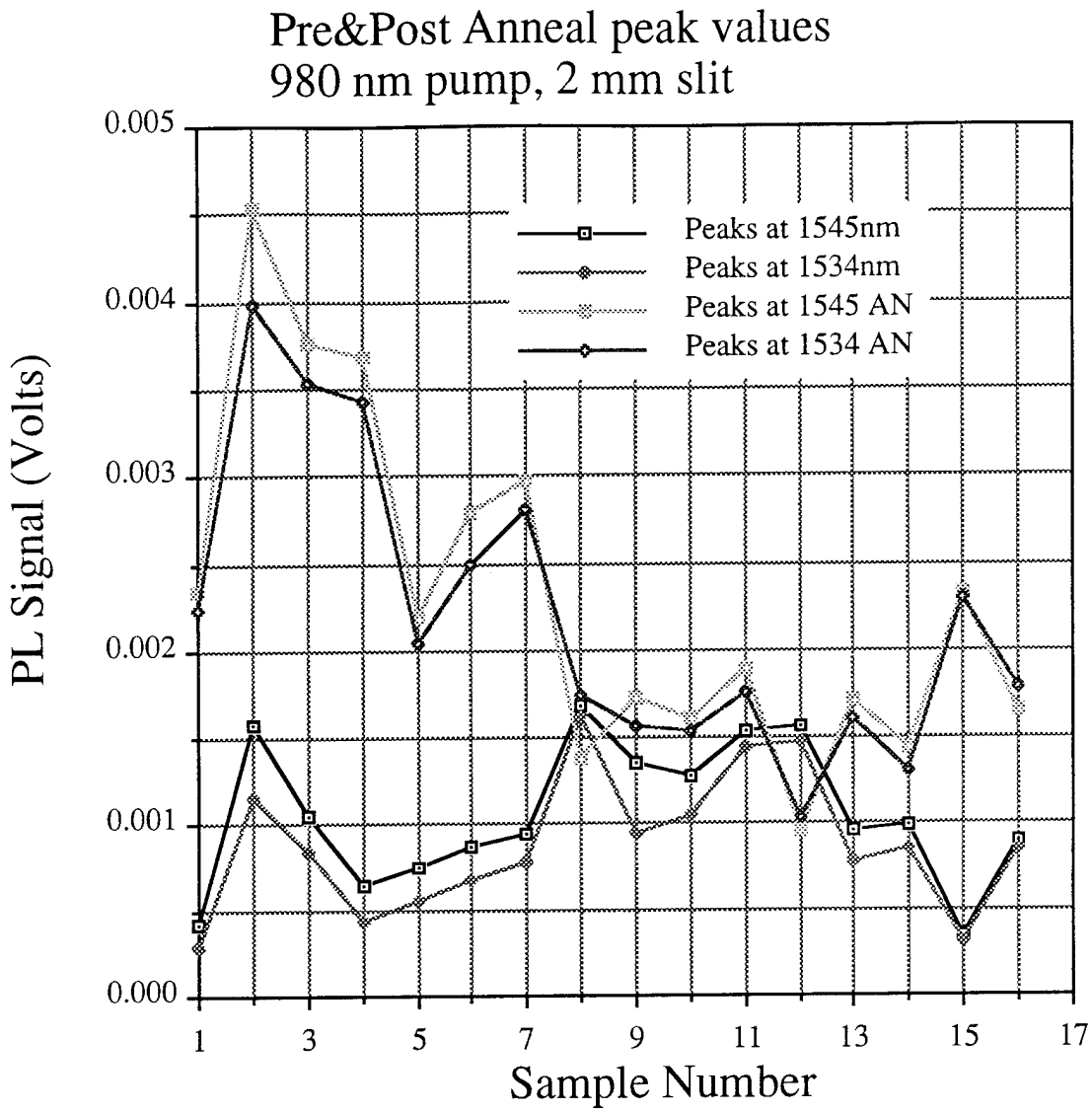


Figure 12. Peak PL signals (at 1545 nm and 1534 nm) versus sample number for all 16 samples, pre and post anneal. AN indicates annealed samples. PL with 980 nm pump laser.

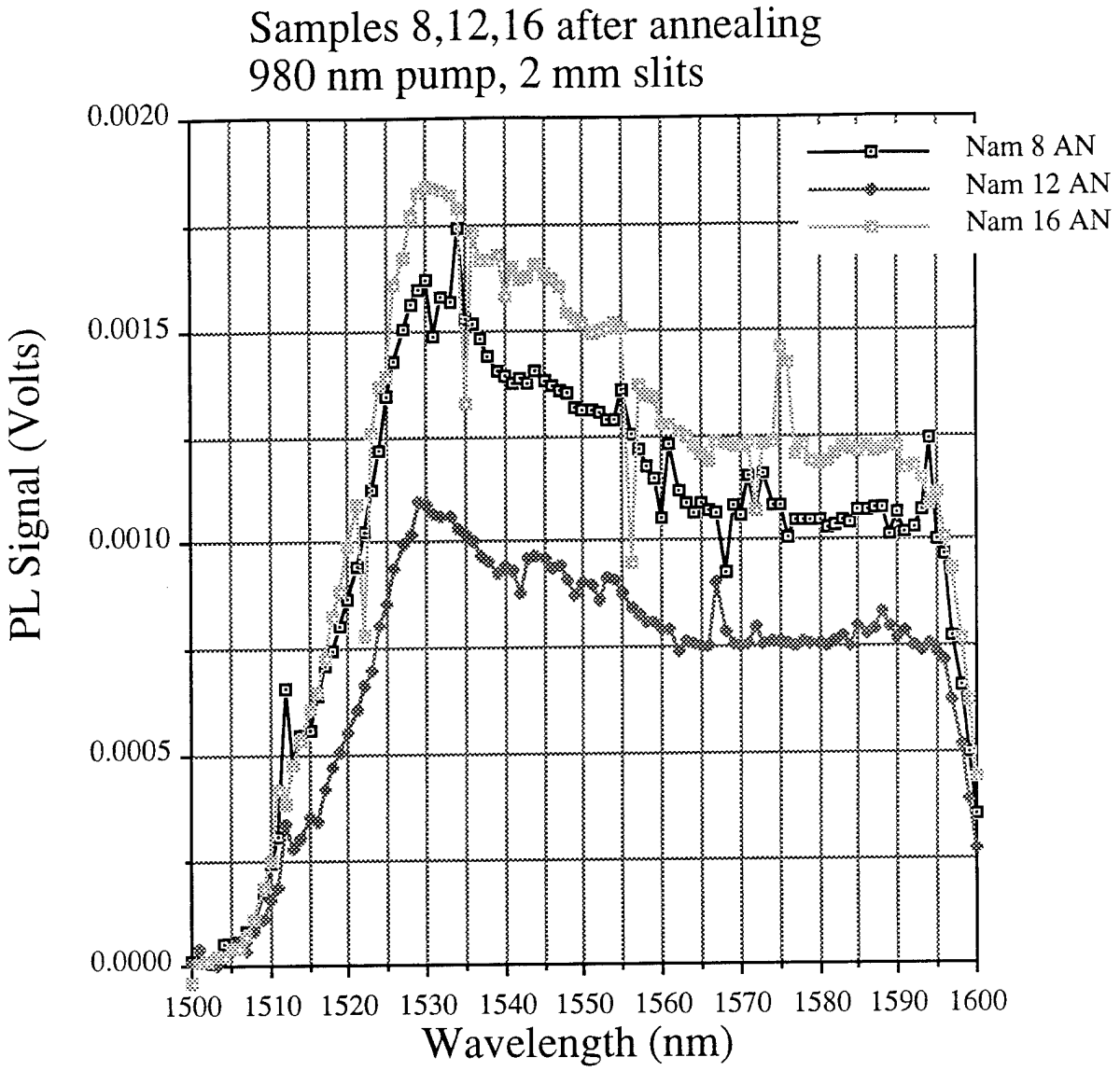


Figure 13. PL spectra of samples 8, 12, and 16, after annealing.

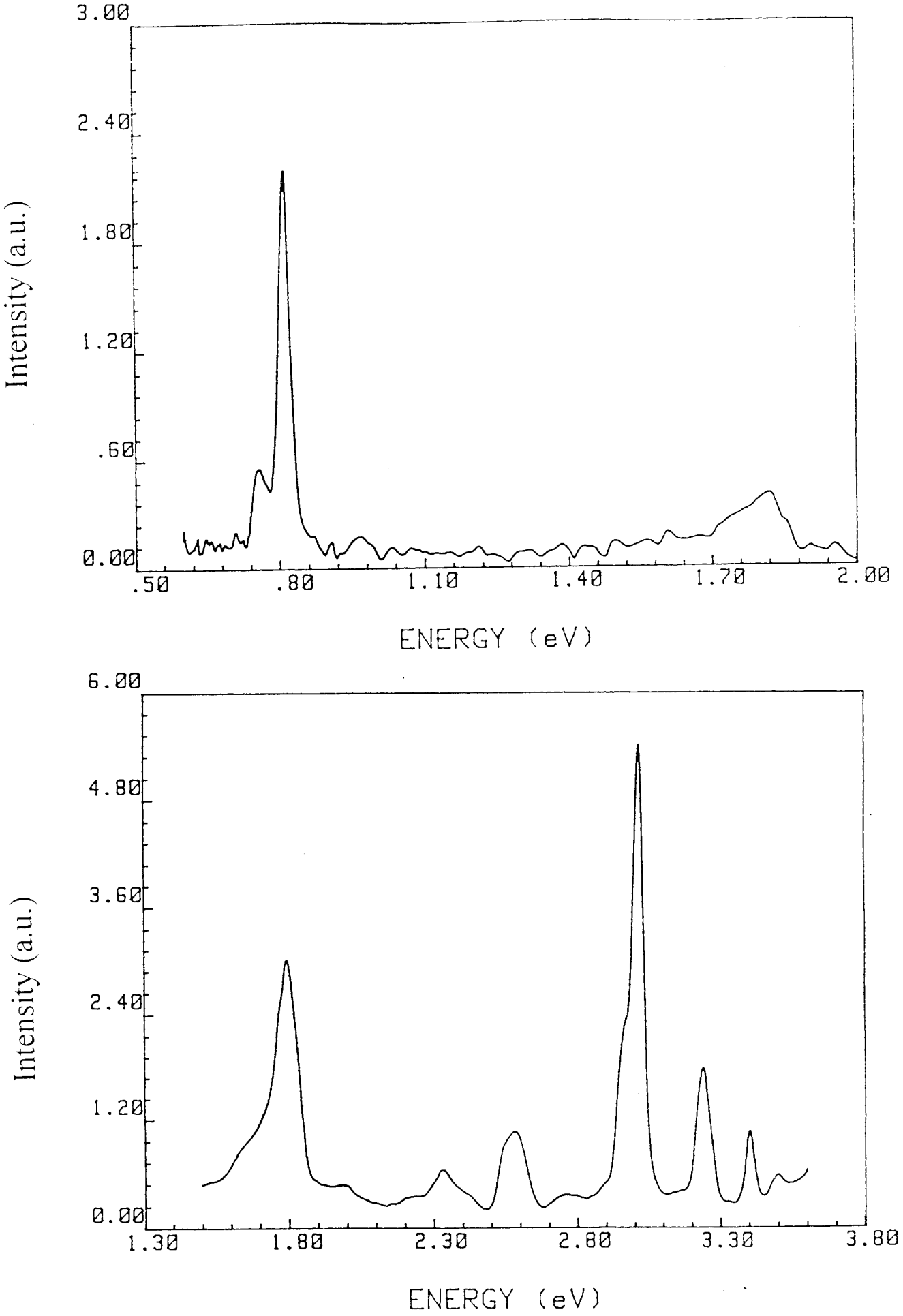


Fig. 14 - Cathodoluminescence (15 KV and 20 μA) of GN 166.

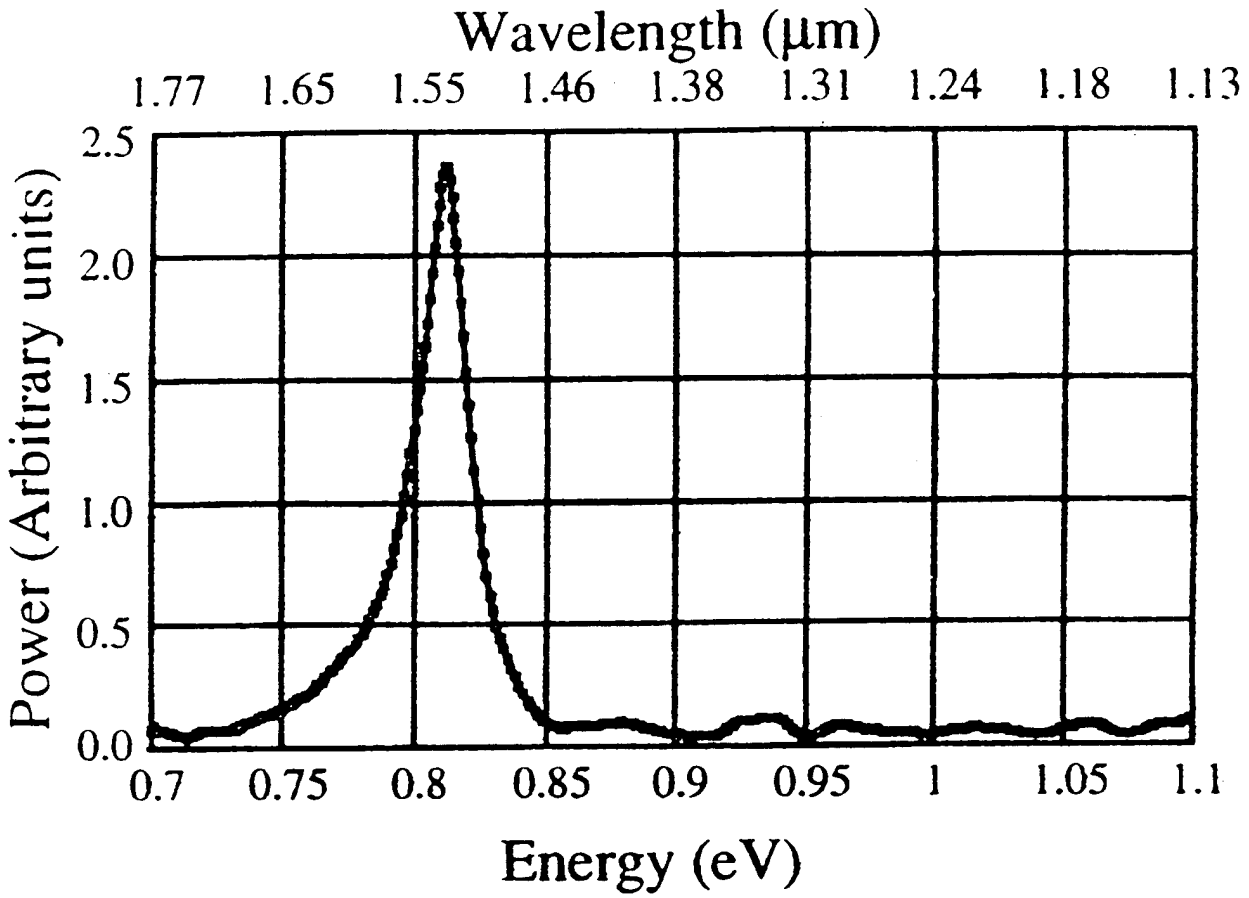


Fig. 15

Room-temperature CL spectra measured from erbium and oxygen coimplanted GaN annealed in flowing ammonia.

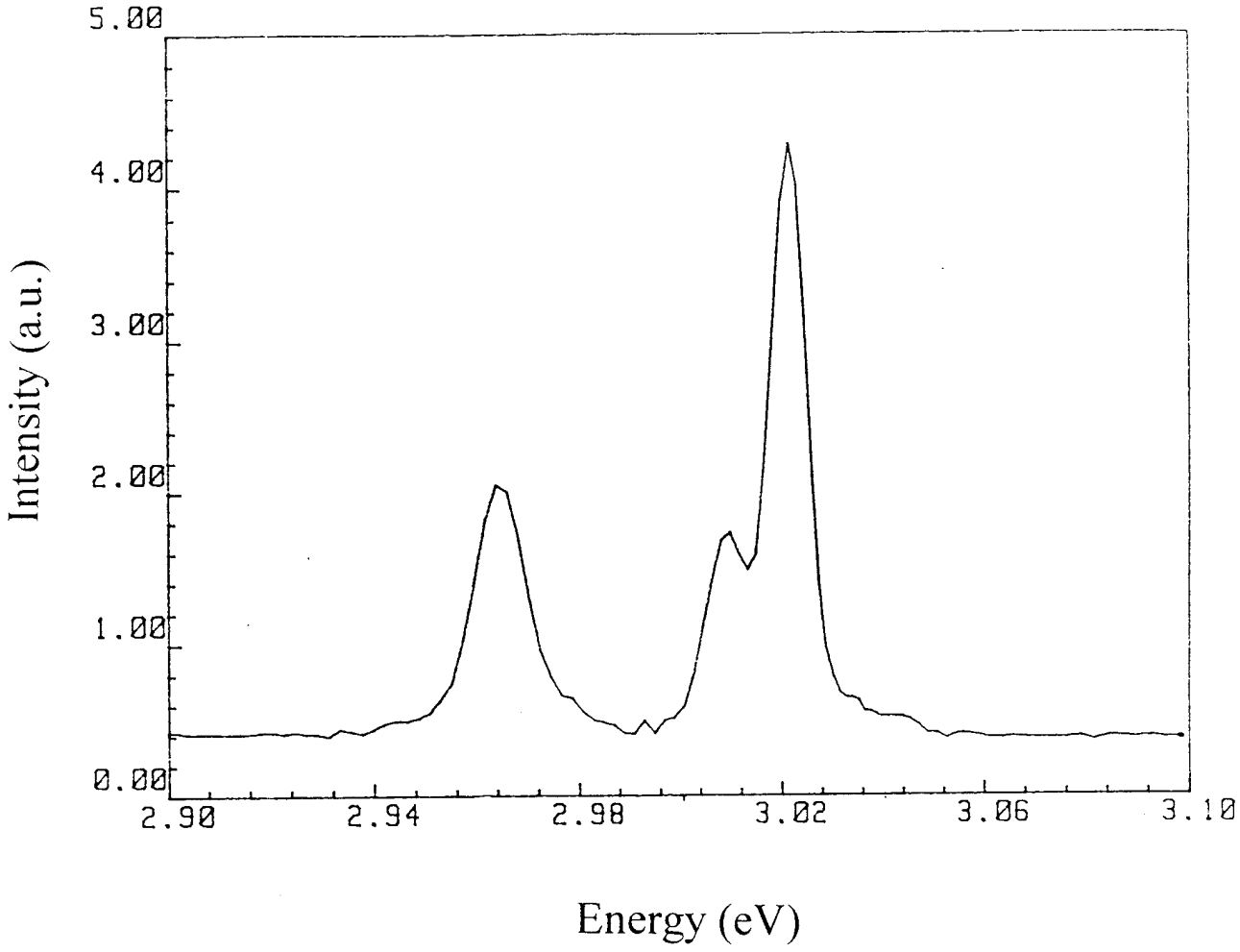


Fig. 16 - High resolution CL spectrum of GN 166 around 3 eV .

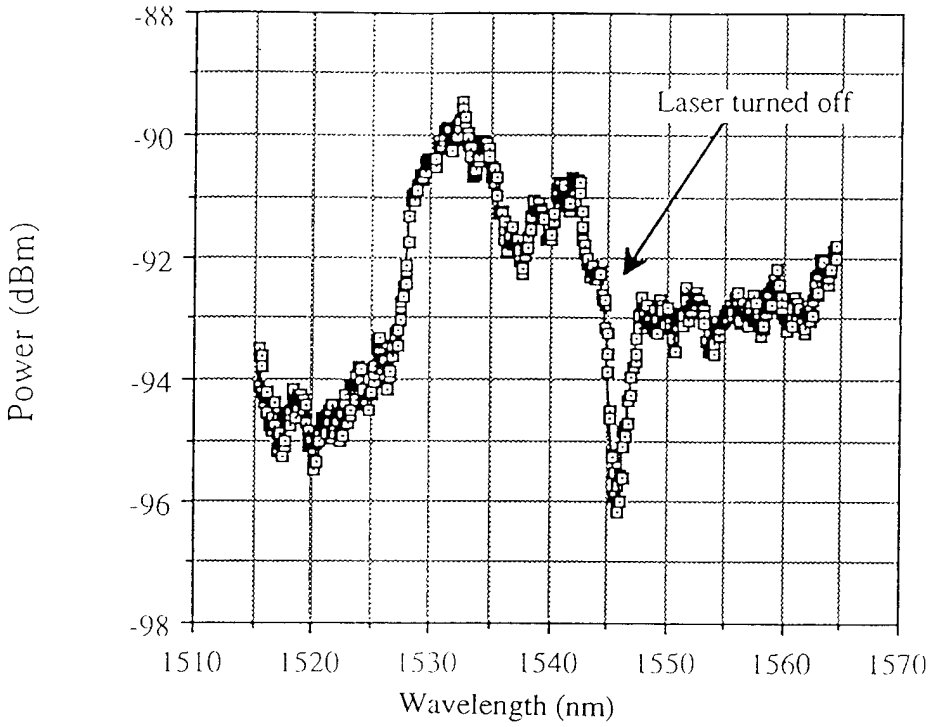


Fig. 17 - GN 166 pumped at 514 nm with 4.3 W at room temperature

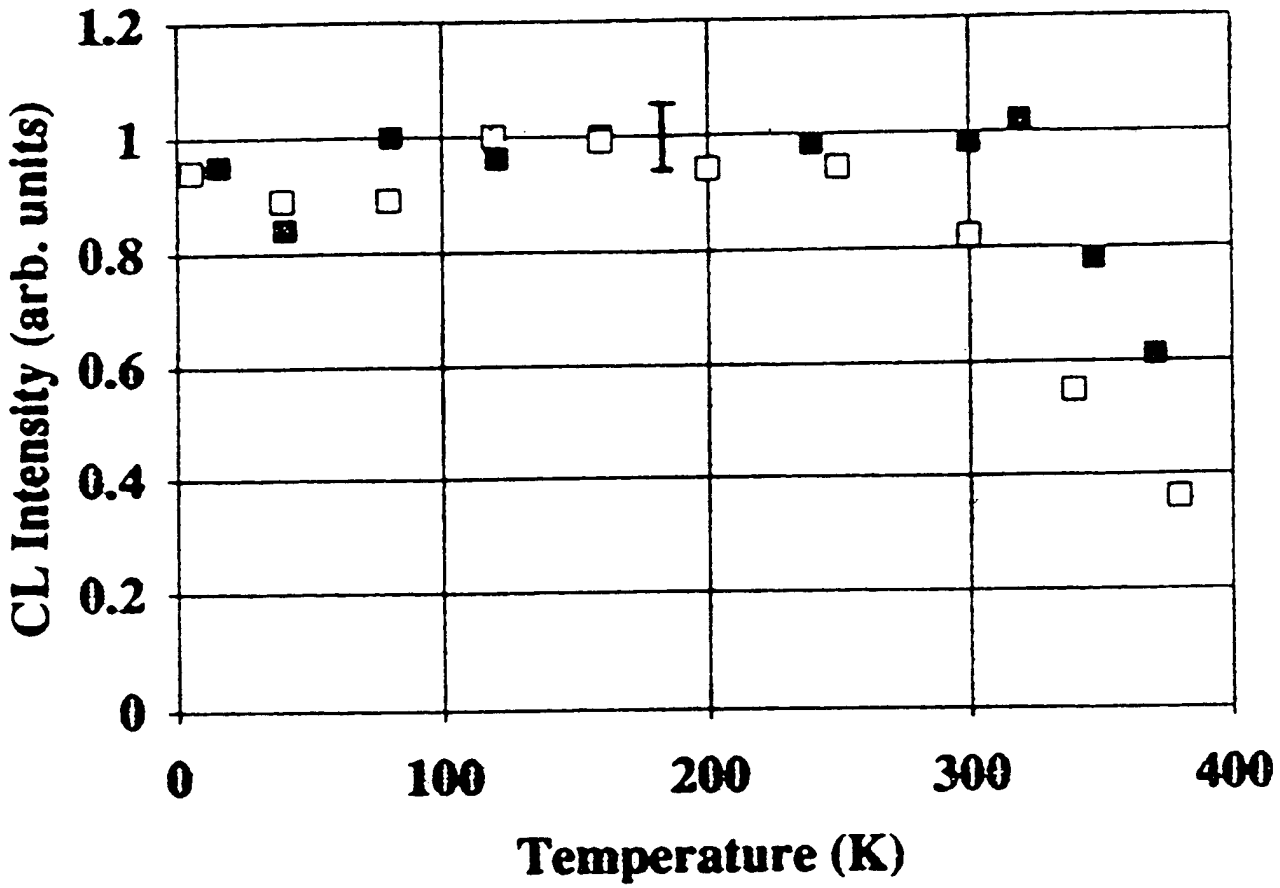
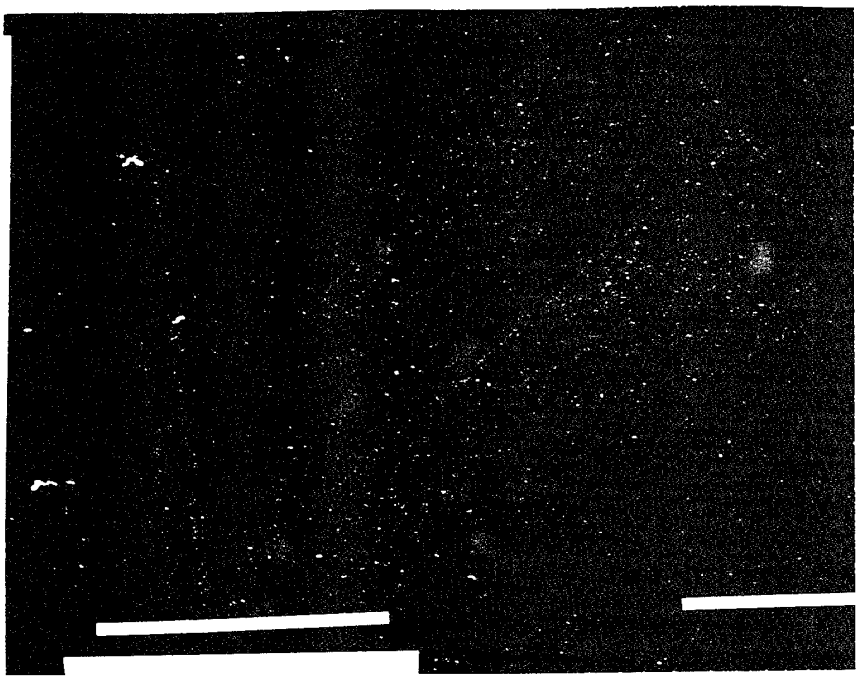
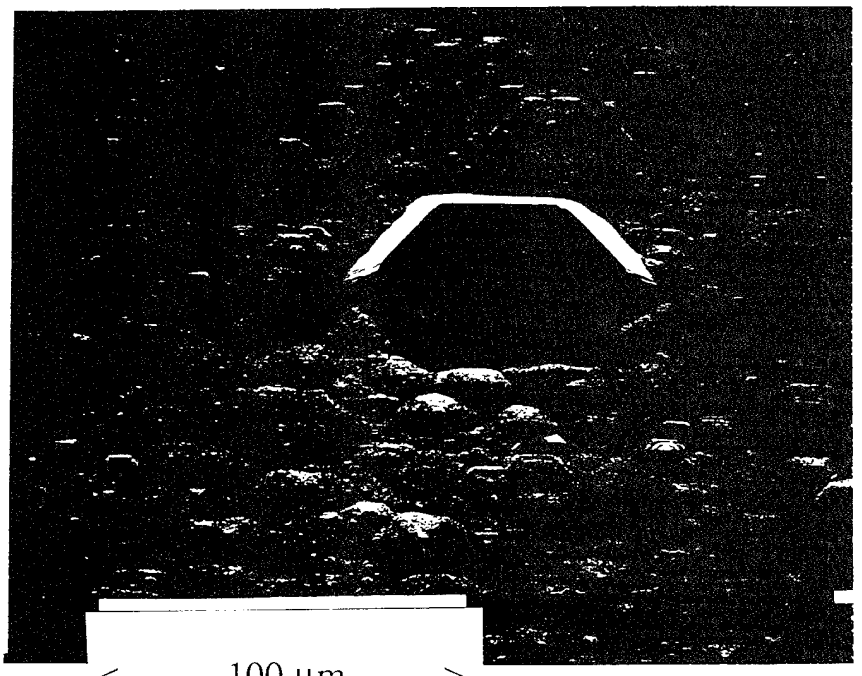


Fig. 18 Temperature dependence of the integrated intensity of the 0.81 eV peak in annealed GaN:Er:O (solid square), compared to data obtained from similarly Er and O coimplanted and annealed sapphire (open square).



<----- 10 μm ----->

GaN sample after polishing



<----- 100 μm ----->

Fig. 19 GaN sample before polishing

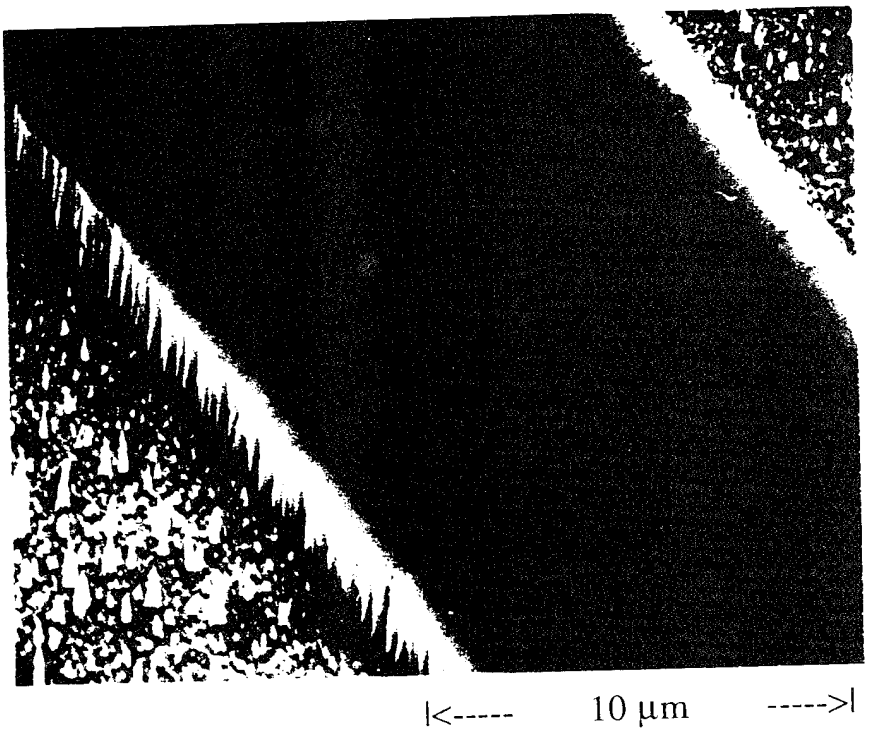
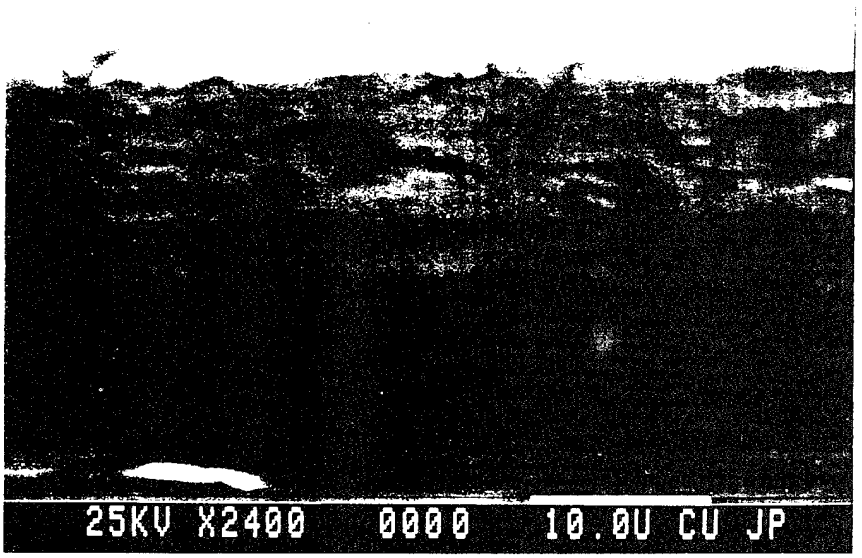


Fig. 20 RIE of GaN rib waveguide

Top Surface

GaN

Sapphire



GaN

Sapphire

Fig. 21 SEM picture of GaN on sapphire cut with a wafer saw.

Analytical Model Used in "rib2"

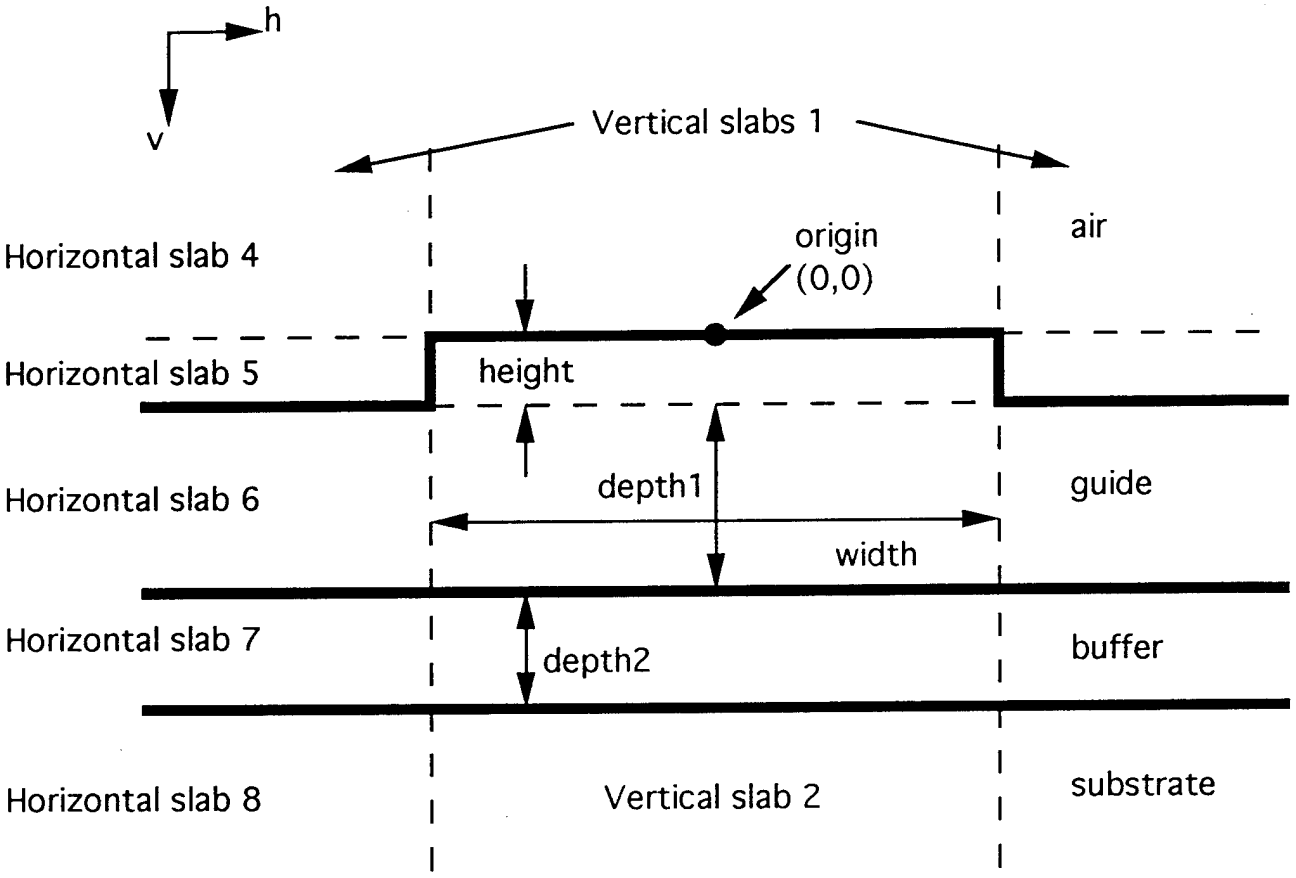
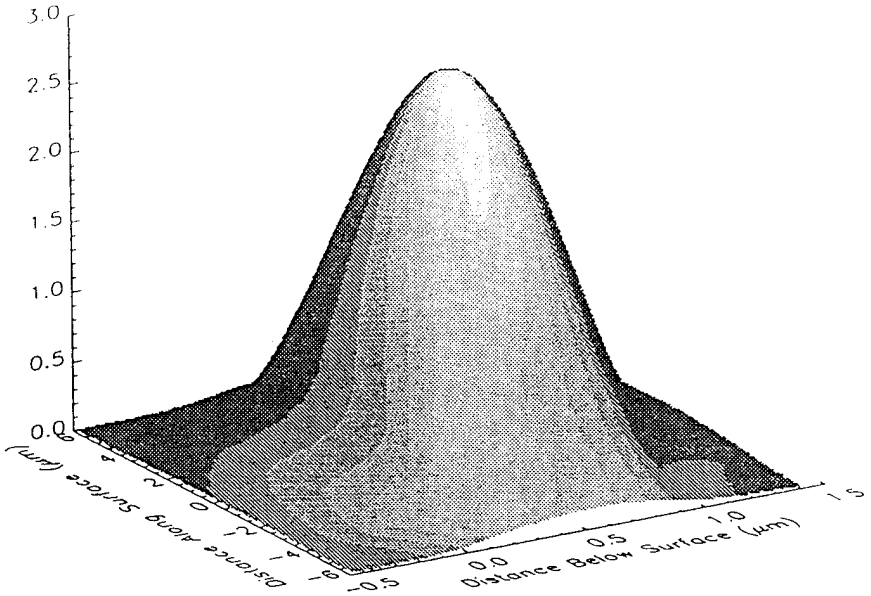


Fig. 22

WAVEGUIDE SIMULATIONS



width = 8.0 μm height = 0.1 μm

depth1 = 0.6 μm depth2 = 0.5 μm

guide index = 2.24 buffer index = 2.29

substrate index = 1.7462 mode = (0,0)

horizontal modes = 2 vertical modes = 2

W	8	8	8	8	8
H	0.115	0.12	0.11	0.11	0.11
D	0	0	0	0.01	0.005
Vmode	1	1	0	1	1
Hmode	1	2	0	2	1

All dimensions are in μm

Fig. 23

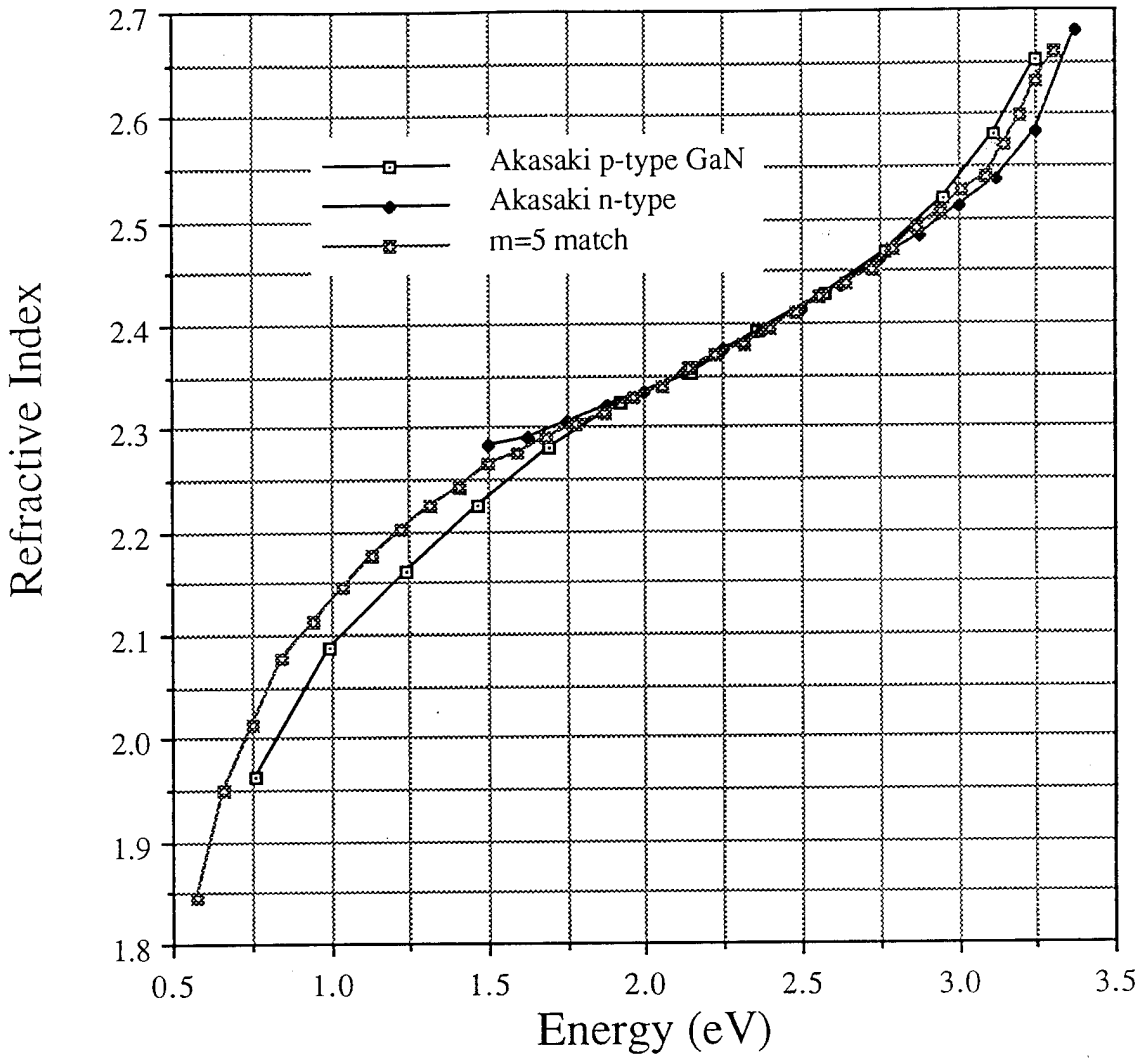


Figure 24. Refractive index data for GaN. The m=5 match is our measurement of an n-type sample, matched to Akasaki published data at 2.5 eV.