AD-A192 269 INTEGRATED OPTOELECTRONIC CIRCUITS UTILIZING INGAASP GROWN BY MOLECULAR B.. (U) GEORGIA TECH RESEARCH INST ATLANTA E L MEEKS 1987 N00014-83-K-2017 1/1

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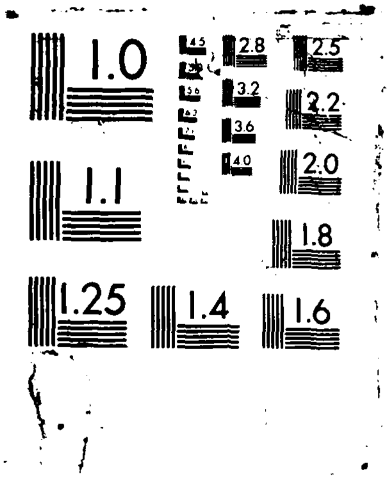
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INTEGRATED OPTOELECTRONIC CIRCUITS UTILIZING [illegible] GROWN BY MOLECULAR BEAM EPITAXY
(Silicon Doping of MBE GaAs)

By

E. L. Meeks

Georgia Tech Research Institute

Prepared for:

Naval Research Laboratory
Washington, D.C. 20375

Under

Contract No. N00014-83-K-2017

1 April 1985 - 31 December 1986

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INTEGRATED OPTOELECTRONIC CIRCUITS UTILIZING InGaAsP GROWN BY MOLECULAR BEAM EPITAXY

(Erbium Doping of MBE GaAs)

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Molecular Beam Epitaxy

INTRODUCTION

Erbium

The scope of the work on this program was changed in the final year. The investigation of InGaAsP MBE materials was stopped and a study of Er doping of MBE grown GaAs layers was initiated.

Gallium Arsenide

Investigations of the properties of rare-earth ions in past decades has concentrated on insulating crystal hosts as laser gain media[1]. More recently, investigations have been extended to covalent crystal hosts such as silicon[2,3] and the III-V compound semiconductors.[3-11] The rare earth doping has been investigated by ion implantation,[3,7,9] and by doping during growth.[4,5,12] At low temperature, this doping results in intense and sharply structured photoluminescence bands in the near infrared due to internal 4f-4f transitions of the rare earth ion Er^{3+}. The wavelength of these emissions is centered around 1.54 microns and does not depend on the band gap energy of the host material, but on the rare earth doping.[8] The emission associated with the Er ions at 1.54 microns is of particular interest for use with fiber optics.

In the present work, the low temperature photoluminescence of Er in GaAs was confirmed and the growth conditions necessary to produce Er doped layers were established.

EXPERIMENTAL PROCEDURE

The Er doped layers were all grown in a Georgia Tech built MBE system. A magnetic manipulator with substrate heater and thermocouple is part of the vacuum interlock through which the substrates are introduced into the growth chamber. The substrate heater thermocouple is mechanically attached to the Mo substrate carrier with a Ta screw. There is no substrate rotation, but the source to substrate spacing is 10 cm with sources 16.7° off axis for a 12% geometric flux variation across a 2 cm sample. The source flange is water cooled and holds six sources with a seventh central position that is used for viewing the substrate. Manually activated Ta shutters interrupt the source beams for flux control. The system is ion pumped and maintains a vacuum better than 1×10^{-9} Torr under idle conditions without LN$_2$. A

LN_2 cold trap surrounds the growth region and the substrate and heater are inserted through a hole in this trap with the magnetic manipulator.

The source materials used were Ga, As and Si of 6N purity and Be and Er of 4N purity. The source heater furnaces were also made at Georgia Tech with all parts constructed of tantalum or pyrolitic boron nitride, except the thermocouples. Tungsten rhenium thermocouples were used in all cases, except for the arsenic source and substrate heater where chromel alumel was used.

All of the epitaxial layers were grown on chromium doped semi-insulating substrates or silicon doped substrates with (100) orientation. The substrates were polished on one side and were chemically etched in the cleaning process to remove 25 microns of the surface to ensure removal of surface damage from polishing.

The growth procedure for each layer was initiated at the end of the growth of the previous layer by the start of the cleaning procedure. Many substrate cleaning procedures, some very elaborate, have been tried but none have produced better results than the simple one that was used for all layers grown. Each substrate was cleaned in boiling trichlorethylene, rinsed in methanol and DI water, and blown dry with nitrogen. The surface was then etched in 5:1:1 H_2SO_4: H_2O_2:H_2O, rinsed in DI water, blown dry with N_2, and kept covered until mounted with indium on molybdenum substrate blocks. The mounting procedure was done in a filtered air laminar flow station, and again, the substrates were kept covered until loaded in the vacuum interlock when the previous layer was removed. The samples were exchanged in the interlock with the growth chamber and sources at idle under ultra high vacuum.

Sorption pumps were used to evacuate the interlock after sample exchange and a small ion pump was also available to improve the vacuum before the isolating gate valve was opened to the growth chamber. The source fluxes were calibrated using an ion gauge in the growth position. By opening and closing the source shutters and adjusting the source temperature, the fluxes of the growth components were calibrated for the desired growth rate. A small tantalum ribbon was used to shield part of the substrate during growth creating a step edge and a moving stylus profiler was used at this edge to determine layer thickness. All substrates were heated to 680°C in an arsenic flux to clean the surface and growth was started by opening the gallium oven shutter as soon as the substrate temperature cooled to 580°C. In most cases, a one micron layer of unintentionally doped GaAs was grown before the erbium doped layers.

The substrate temperature for optimum GaAs growth was previously determined to be 580°C and all of the buffer layers and Er doped layers were grown at this temperature. The PL intensity is not a strong function of the growth temperature, but the maximum reported by Smith, et al.[6] corresponds to the optimum GaAs growth temperature at 580°C.

The flux from the Er source was measured by depositing Er on an unheated GaAs substrate in the normal growth position for 64:48 hours. Part of the substrate was masked and the thickness of the deposit was measured with a moving stylus profiler. Assuming a bulk density for the deposited film, a flux of 3.3×10^{12} $cm^{-2}s^{-1}$ was obtained for a source temperature of 1000°C.

RESULTS AND DISCUSSION

Secondary ion mass spectrometry (SIMS) data on the MBE grown GaAs:Er doped layers confirm the presence of Er in the layers and the increase in Er doping with source temperature. Figures 1, 2 and 3 show the SIMS profiles for layers grown with source temperatures of 790°C, 830°C and 870°C, respectively. The profiles were all run until the interface between Er doped layer and buffer layer was observed. An oxygen ion beam was used for maximum sensitivity and the GaO peak was monitored to confirm the stability of the ion beam current. Also, since the Ga concentration should be the same for all GaAs samples, the GaO peak was used to standardize the system sensitivity between layers so the Er count for the three layers could be compared. Figure 4 shows the adjusted Er signal intensity for these layers compared to the equilibrium vapor pressure[13] of Er. Figure 4 also shows Er signal intensity for five other layers grown with source temperatures 810°C, 830°C, 850°C, 870°C and 910°C. These five layers were profiled using an argon ion beam. The detection sensitivity of the SIMS is much better for the oxygen ion beam, thus, the count obtained with oxygen is higher for the same source temperature. Both sets of data show proportionality between Er incorporated in the grown layer and the equilibrium vapor pressure of Er. Thus, using the Er vapor pressure and the measured Er flux at 1000°C, the Er flux at the substrate was calculated. Figure 5 shows the Er flux at the substrate surface calculated over the source temperature range.

Photoluminescence measurements on three samples of MBE grown GaAs:Er layers were very interesting. There is no evidence of any broad background which is observed in ion-implanted samples. The signal goes to zero for energies less than 0.75 eV and greater than 0.81 eV as indicated in Figures 6-8.

Figures 6-8 show the photolumlinescence of layers grown with Er source temperatures of 790°C, 830°C and 870°C. The 830°C layer is the brightest of the three, showing a maximum intensity 10 times the 790°C layer and 2 times the 870°C layer. The optimum Er source temperature, therefore, seems to be broad and spanned by these samples and a more precise temperature for maximum intensity was not determined.

The measurements shown in Figures 9 and 10 were taken with narrower slit width and expanded scale and show considerable structure on the PL peak. Very narrow lines of less than 0.1 meV can be seen.

In the region of the bandgap, the 790°C sample shows a broad Zn donor-acceptor pair line (Figure 11) but the near-gap and acceptor related PL above 1.24 eV is weak. At higher Er doping, no PL was detected in this region, indicating the Er doping quenches the near-gap PL.

CONCLUSION

The intense and sharply structured photoluminescence of MBE grown Er doped GaAs centered around 1.54 microns has been confirmed. A broad intensity maximum corresponding to an optimum

Er flux of 3-4 x $10^{-10}cm^{-2}$ at the sample surface was measured. For our growth system, these conditions were obtained at an Er source temperature of 830°C. As requested, samples were delivered to NRL for further evaluation and processing.

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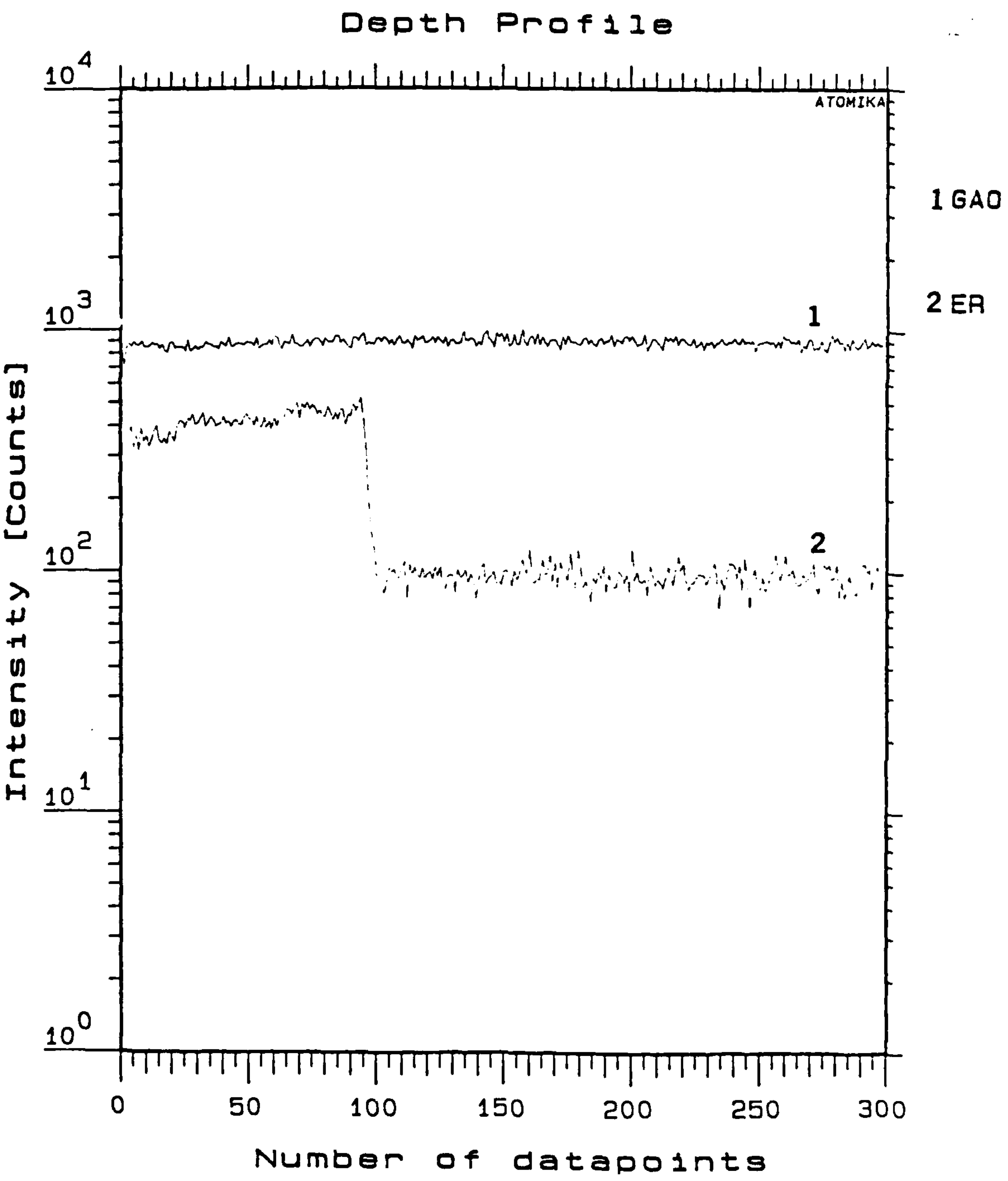

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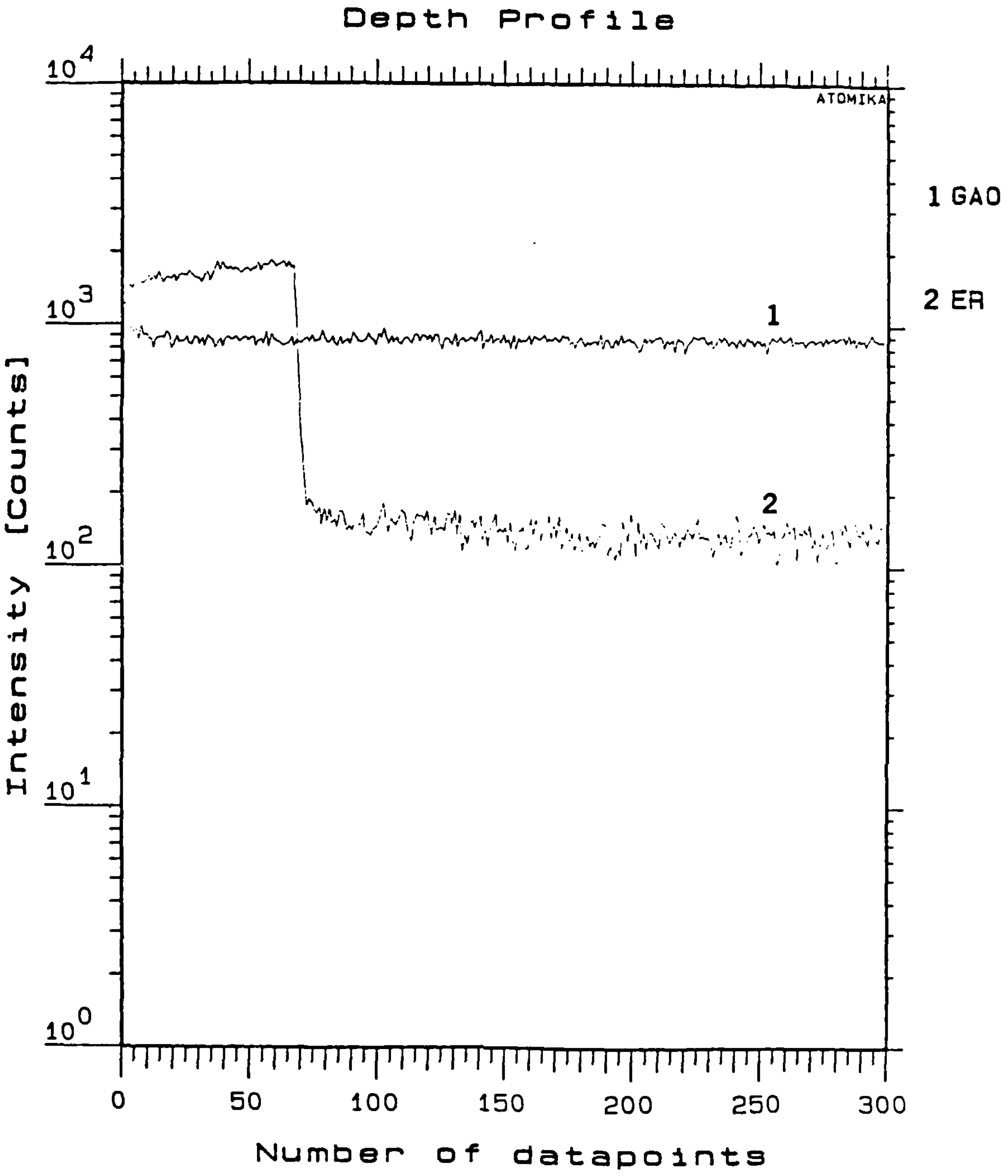
Scan Gate : 50 [%]

Label	Mass	Cycles	Energy
GAO	85.5	5	0.0
ER	166.0	6	0.0

Figure 1. SIMS Profile GaAs + Er 790°C.



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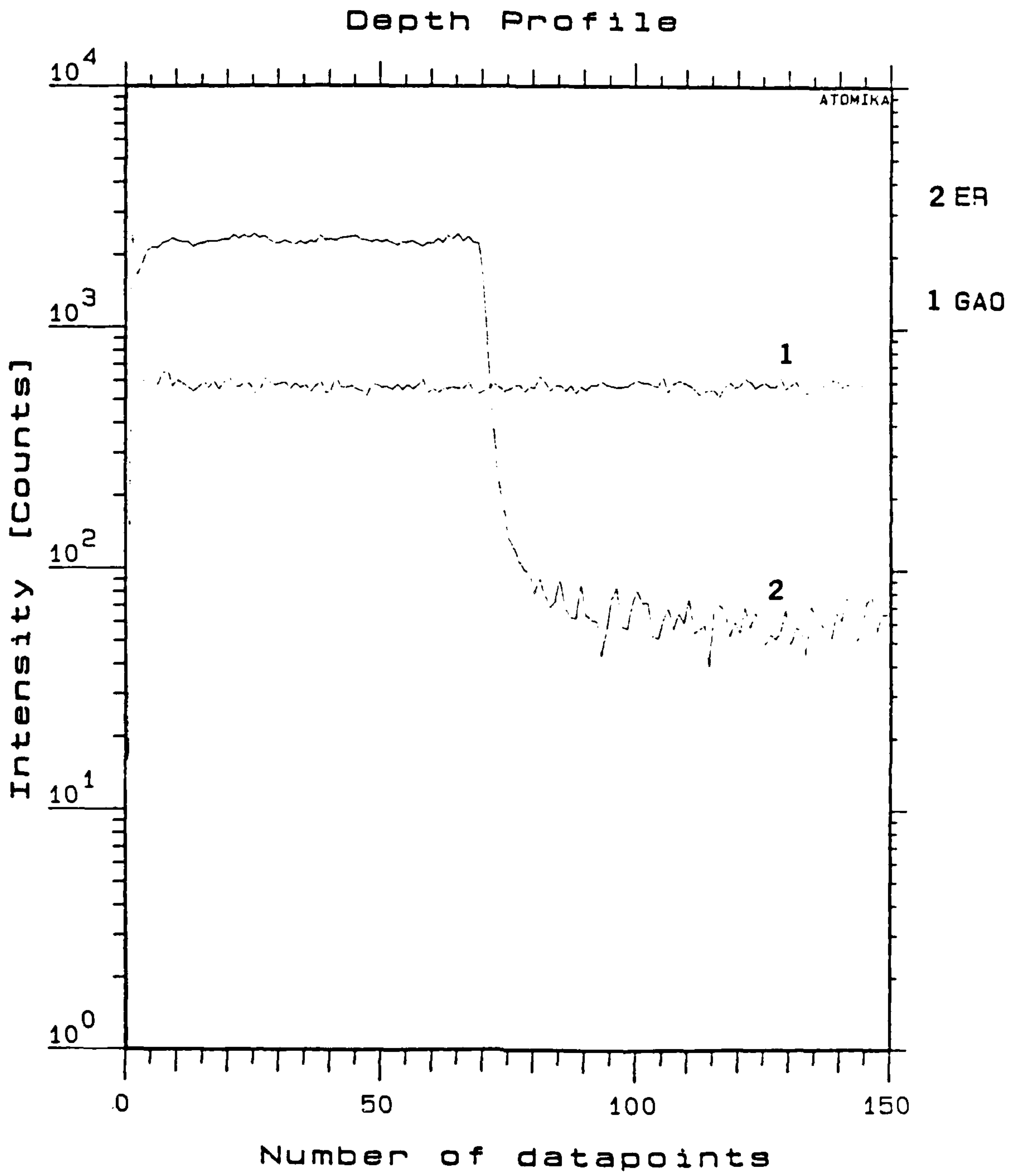
GAO	85.5	6	0.0
ER	166.0	6	-3.0

Figure 2. SIMS Profile GaAs + Er 830°C.



Primary Ions : Oxygen
Ion Energy : 12 [kev]
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Scan Gate : 50 [%]

Label	Mass	Cycles	Energy
ER GAAS 8	166.0	6	0.0
GAO	85.5	6	0.0

Figure 3. SIMS Profile GaAs + Er 870°C.

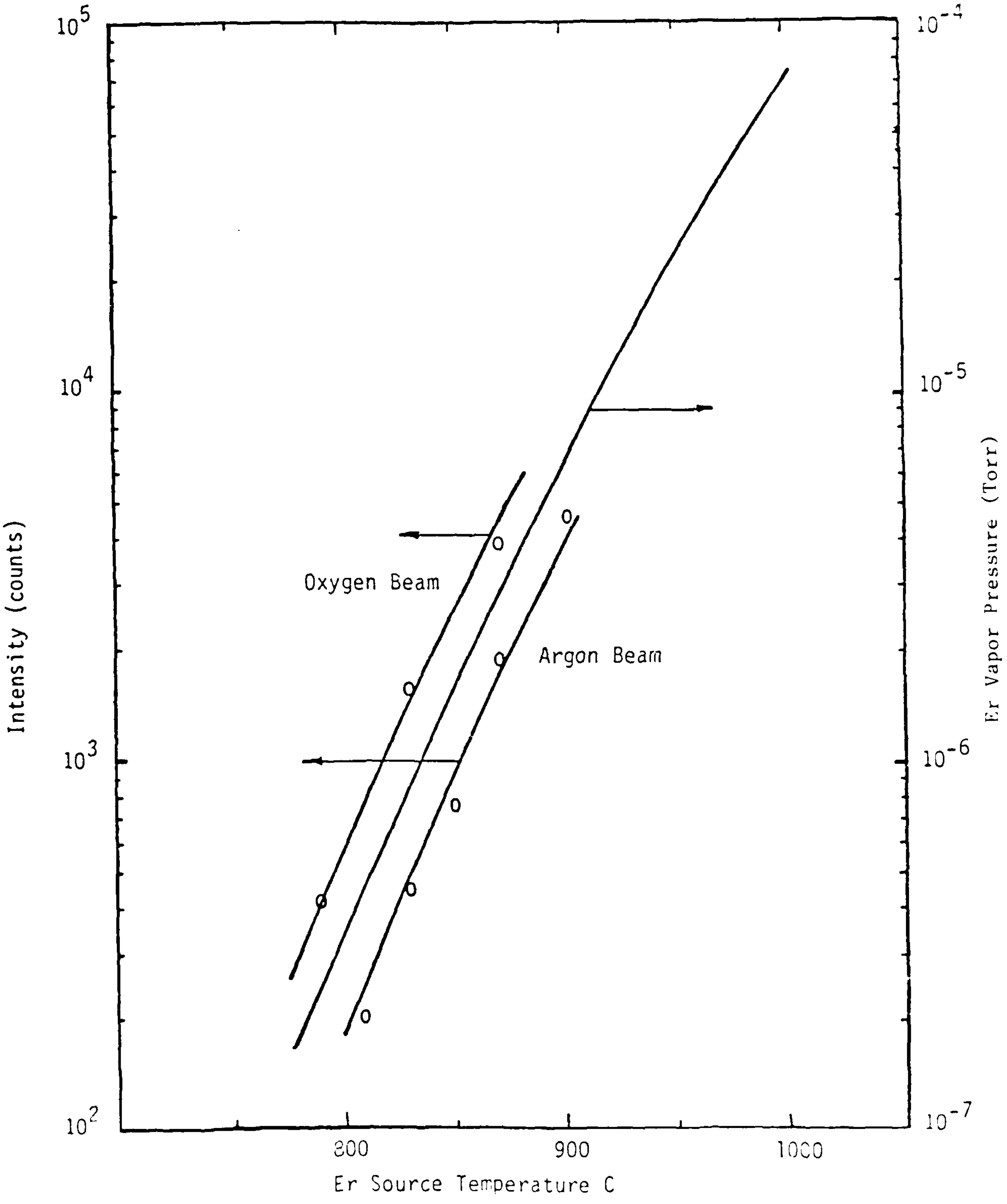

Figure 4. Er Doping vs. Source Temperature.

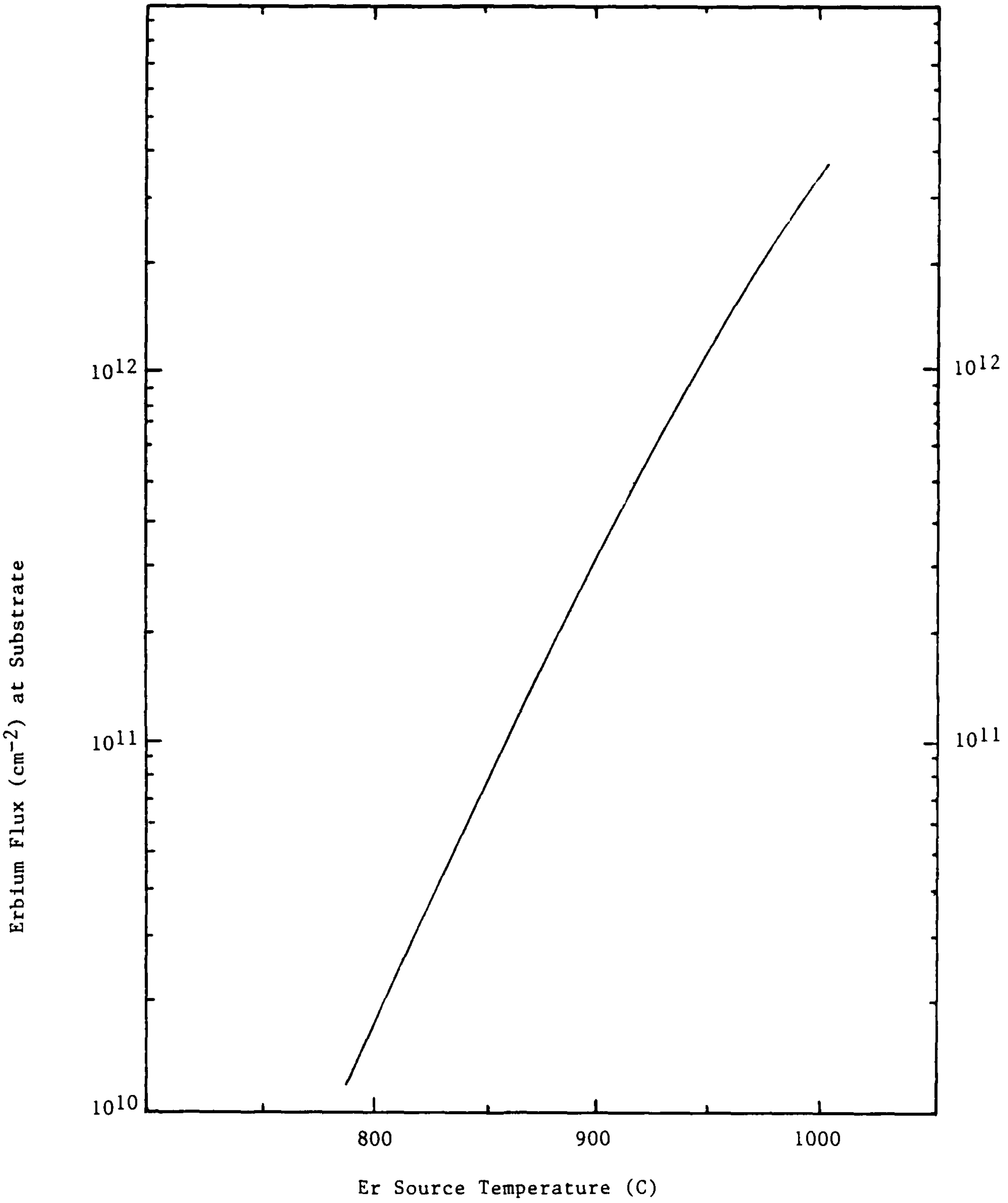

Figure 5. Erbium Flux at the Substrate.

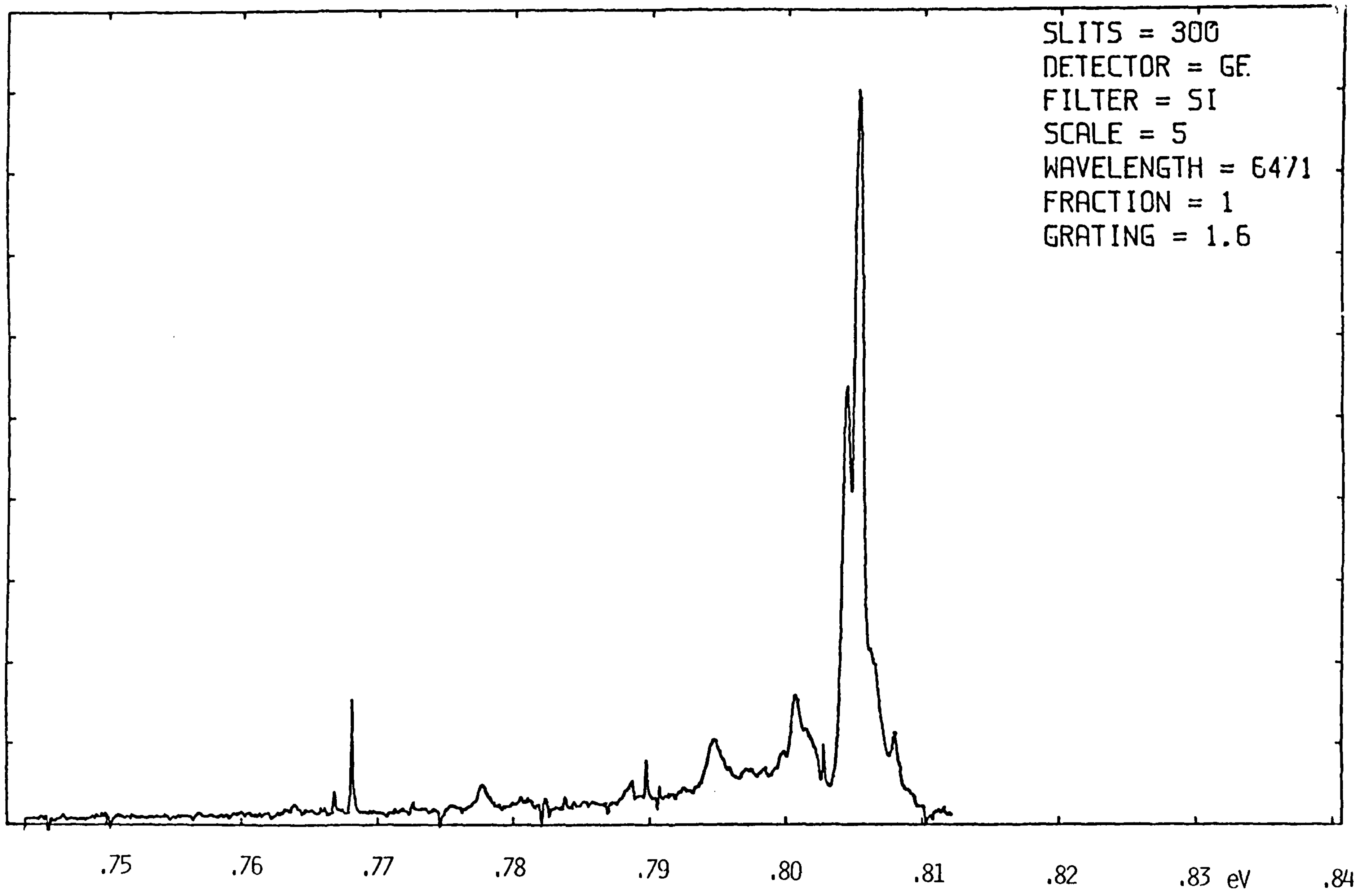

Figure 6. Photoluminescence of MBE GaAs + Er at 790°C. (PL by P. Klein NRL)

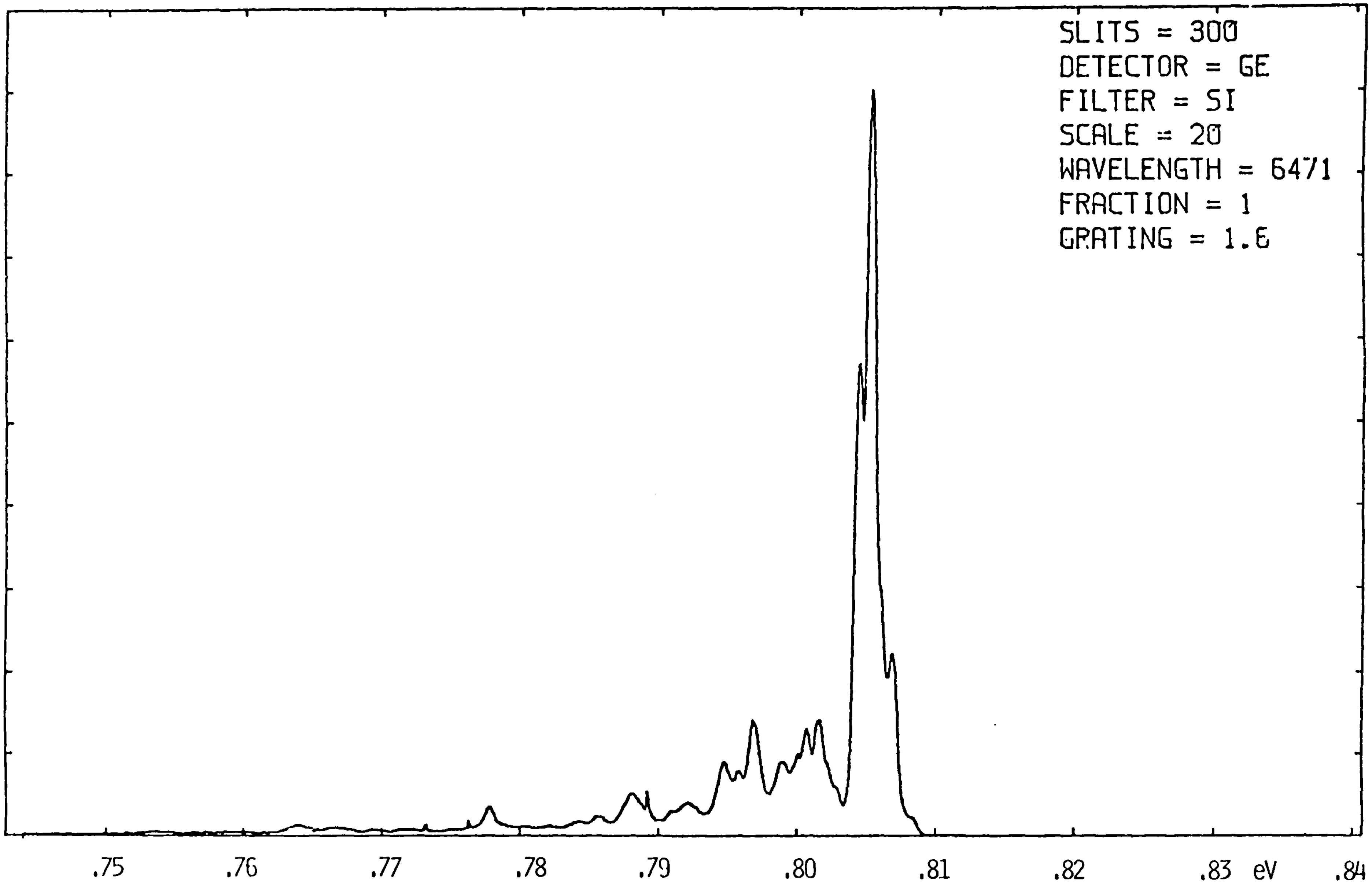

Figure 7. Photoluminescence of MBE + Er at 830°C. (PL by P. Klein NRL)

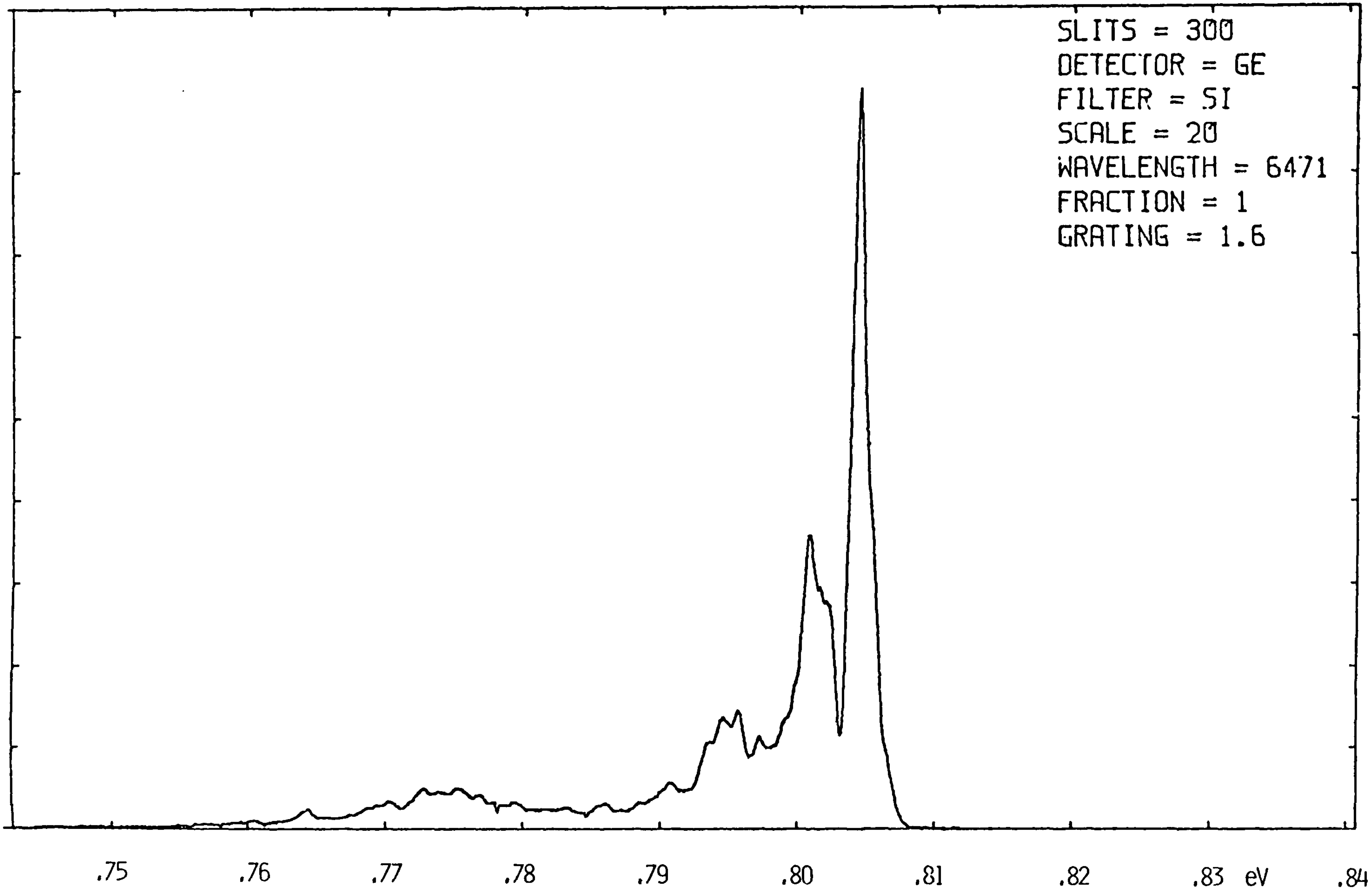

Figure 8. Photoluminescence of MBE GaAs + Er at 870°C. (PL by P. Klein NRL)

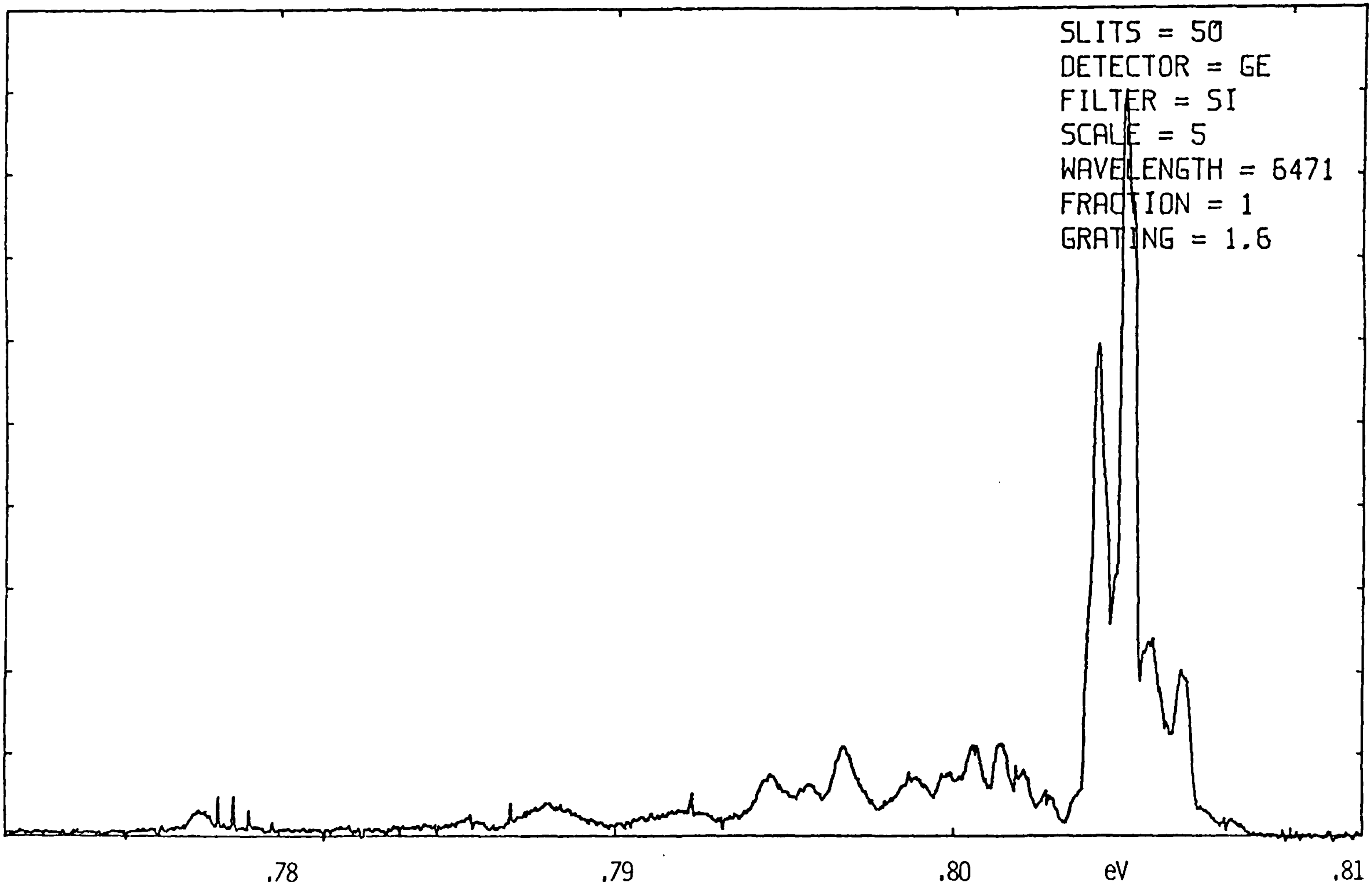

Figure 9. Photoluminescence of MBE GaAs + Er at 830°C. (PL by P. Klein NRL)

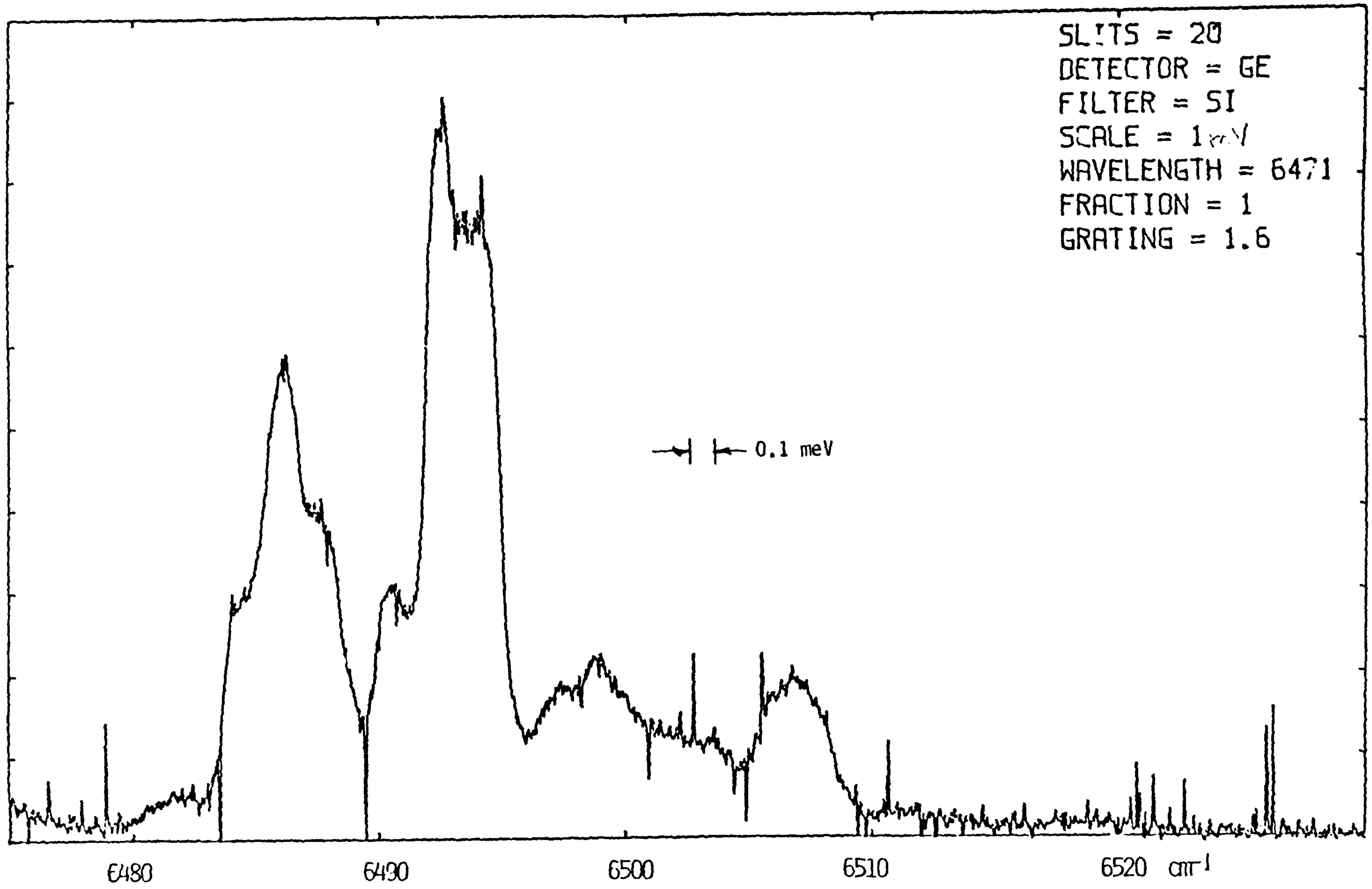

Figure 10. Expanded scale PL showing very narrow linewidths. (PL by P. Klein NRL)

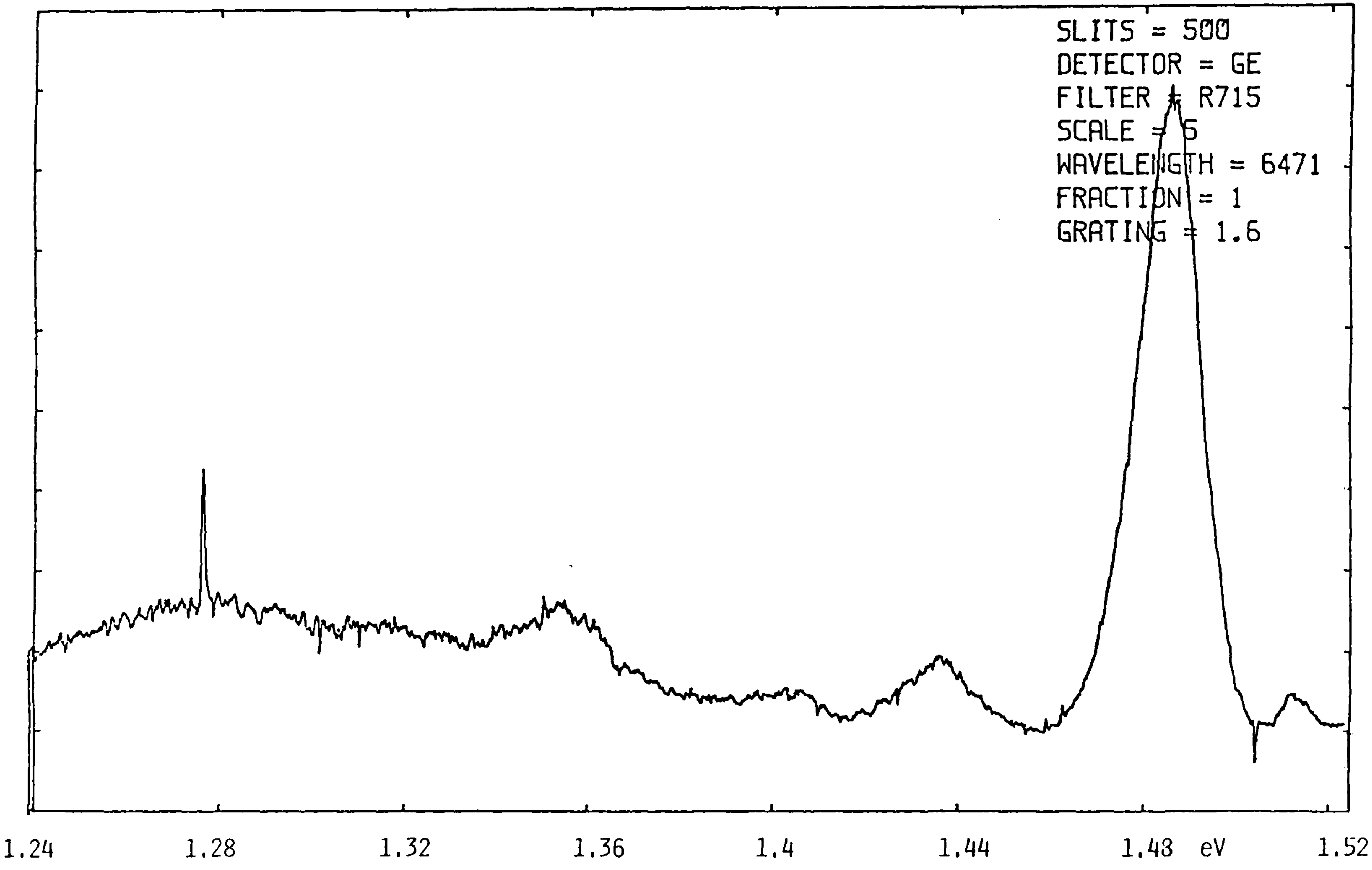

Figure 11. Photoluminescence of Bandgap Region for MBE GaAs + Er at 790°C. (PL by P. Klein, NRL)

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