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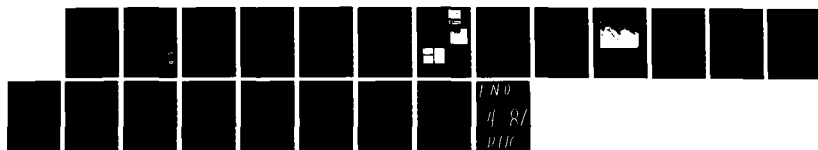
DEFECT REDUCTION IN EPITAXIAL GROWTH USING SUPERLATTICE 1/1
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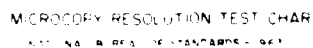
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NARR. -- PROG. -- FROM U) Several superlattice structures, in which the lattice constant has been used to reduce the defect concentration by eliminating the defects from the GaAs substrates, have been developed. The structures consist of a strained layer superlattice buffer, grown on the substrate, and the rest of the film is grown on silicon substrates. The structure of the strained layer is grown either as part of the substrate or buffer layers is being addressed. We are currently investigating the growth of the strained layer superlattice and its effect on the defect concentration without any other existing.				
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Progress Report

**DEFECT REDUCTION IN EPITAXIAL GROWTH
USING SUPERLATTICE BUFFER LAYERS**

Submitted to

U.S. Air Force Office of Scientific Research

AFOSR-85-0201

By

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December 1986

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During the last period activities have been carried out along the following directions.

1) DEFECT REDUCTION IN GaAs GROWN BY MOLECULAR BEAM EPITAXY USING DIFFERENT SUPERLATTICE STRUCTURES

Several superlattice structures, grown by molecular beam epitaxy, have been used to reduce the density of threading dislocations originating from the GaAs substrates. Results clearly indicate that compared to epitaxial layers grown directly on GaAs substrates, a GaAs-In_xGa_{1-x}As superlattice ($x < 0.12$) reduces the dislocations by approximately two orders of magnitude. Transmission electron microscopy, electron beam induced current, and etch pit density have been used to characterize the effectiveness of using superlattice buffer layers for the reduction of defects in GaAs epilayers. (APL 942, 49, 1986)

2) DEFECT REDUCTION IN GaAs EPILAYERS ON Si SUBSTRATES USING STRAINED LAYER SUPERLATTICES

Initial results indicate that GaAsP-InGaAs strained layer superlattice buffer layers are effective in reducing dislocation in GaAs grown on silicon substrates. Transmission electron microscope (TEM) studies (see attached results) show that a very large percentage of dislocations originating at GaAs/Si interfaces are stopped at these superlattice buffer layers. (Material Research Society, Symposium on Heteroepitaxy on Silicon Technology, accepted)

3) EFFECT OF STRAINED LAYER SUPERLATTICES (SLS) ON THE OUT DIFFUSION OF Ge

GaAs with and without SLS have been grown on Ge substrate. The effect of out diffusion of Ge into the GaAs epilayer is being studied at different annealing temperature and time.

4) STABILITY OF STRAINED LAYER SUPERLATTICE UNDER HIGH LEVELS OF CURRENT INJECTION

The stability of SLS in electronic devices, either as part of the active layers or buffer layers is being addressed. Successful long-term room temperature operation of InGaAs/GaAsP strained layer superlattice light emitting diodes under high constant current injection is reported. These LED's have been tested over 1000h with $3000\text{A}/\text{cm}^2$ with no observed degradation in the optical output. This SLS has an average lattice constant equal to that of GaAs, thus no defects are generated between this SLS and the GaAs substrate or epilayers. This is the first reported lifetime test of any strained layer superlattice devices. (Submitted to Electron Device Letters)

5) MOLECULAR STREAM EPITAXY

We are currently conducting pioneering work in new ways to deposit superlattices and atomic layer epitaxy without any gas switching. This is achieved by rotating the substrate to cut across stream of gases such as TMG, AsH₃, PH₃ and TEI. Thickness control on the atomic levels has been achieved. (Six papers on this subject have been already published)



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Defect reduction in GaAs grown by molecular beam epitaxy using different superlattice structures

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Several superlattice structures, grown by molecular beam epitaxy, have been used to reduce the density of threading dislocations originating from the GaAs substrate. Results clearly indicate that compared to epitaxial layers grown directly on GaAs substrates, a GaAs-In_xGa_{1-x}As superlattice ($x < 0.12$) reduces the dislocations by approximately two orders of magnitude. Transmission electron microscopy, electron beam induced current, and etch pit density have been used to characterize the effectiveness of using superlattice buffer layers for the reduction of defects in GaAs epilayers.

Compound semiconductor substrates typically have several types of defects, such as dislocations, which can degrade the operation of devices and circuits. For example, semi-insulating GaAs substrates have dislocations of the order of 10^4 cm^{-2} and greater. Moreover, the dislocation density which typically has a W-shaped distribution is nonuniform across the sample surface.¹ The presence of such defects greatly influences device parameters such as source-drain currents and threshold voltages resulting in a dramatic degradation of performance.² We have recently reported the use of GaAsP-InGaAs strained-layer superlattices (SLS's) to reduce threading dislocations originating from the GaAs substrate. Furthermore, the GaAs epilayers grown on this SLS buffer were found to be almost dislocation-free.³ This SLS structure, grown by metalorganic chemical vapor deposition, was constructed from alternating layers under tension (GaAsP) and compression (InGaAs). The compositions of the two ternary alloys are adjusted such that the SLS is lattice matched to GaAs.⁴

Superlattice structures composed of phosphorus and arsenic compounds are difficult to grow using molecular beam epitaxy (MBE). Consequently, most MBE superlattice buffer layers are based on Al_xGa_{1-x}As-GaAs SLS structures ($0 < x < 1$).⁵ Since this SLS structure is nearly lattice matched, the built-in strain is insufficient to suppress the propagation of threading dislocations originating from the substrate. However, it has been reported in the literature⁵ that using Al_{0.3}Ga_{0.7}As-GaAs and AlAs-GaAs SL's, the dislocation density can be reduced by factors of 3 and 20, respectively. This is in contrast to the three to four orders of magnitude reduction when GaAsP-InGaAs SLS's are used.³ In this letter we report the use of GaAs-In_xGa_{1-x}As ($0 < x < 0.2$) SLS buffer layers reducing dislocations in the GaAs epilayers.

The GaAs-InGaAs SLS's were grown by MBE at 550 °C on both (100) Cr- and Si-doped substrates. For comparison, a similar structure made of Al_{0.3}Ga_{0.7}As-GaAs superlattice was grown at 620 °C. A 2-μm GaAs epitaxial layer was grown on the SL and directly on the substrate for etch pit density (EPD) determination and comparison. All the

grown layers were Si doped to the mid $10^{16}/\text{cm}^3$ range. The superlattice has five periods, each layer being 100 Å thick. Since the individual layers are sufficiently thin, the lattice mismatch is elastically accommodated by the uniform strain. This strain can be present as a compressive strain in the In_xGa_{1-x}As layers only, thus maintaining the GaAs lattice constant in the growth plane. However, the strain can also be accommodated by tensile and compressive strains in the GaAs and InGaAs films, respectively. In this case the superlattice will have a lattice constant corresponding to InGaAs with an InAs mole fraction of $x/2$. If the total thickness of the SL structure made of these five periods exceeds the critical thickness⁶ h_c , misfit dislocations will be generated at the SLS-GaAs interfaces. The generation of misfit dislocations has been observed in our present study and will be discussed later. Consequently, in order to prevent the generation of dislocations at the SLS-GaAs interfaces we must limit the total thickness of the SLS to a value below the critical thickness, h_c . However, by incorporating intermediate thick layers of GaAs we can extend the number of sets of five-period GaAs-InGaAs SLS's. In Fig. 1 we illustrate a structure incorporating a 2000-Å GaAs buffer layer between

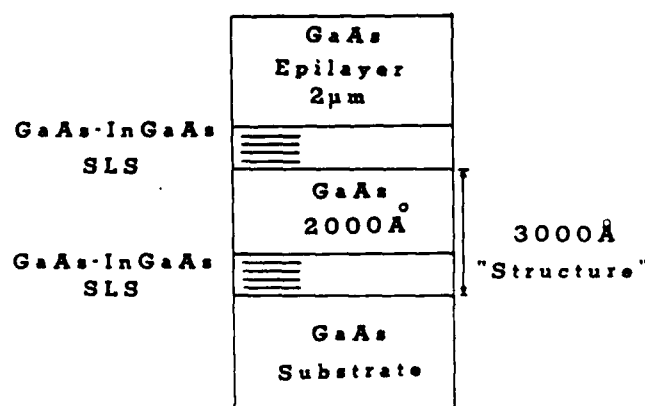


FIG. 1. Schematic of GaAs-InGaAs SLS with 2000-Å GaAs intermediate layer used to reduce defects in the GaAs epilayer.

TABLE I. Results of etch pit density (EPD) measurements^a

Run	Five-period SLS structure	Number of repeat "structures" with 3000-Å period	EPD (cm ⁻²)
A	GaAs-Al _{0.3} Ga _{0.7} As	2	1-5 × 10 ⁴
B	GaAs-In _{0.06} Ga _{0.94} As	2	~5 × 10 ³
C	GaAs-In _{0.1} Ga _{0.9} As	2	9 × 10 ² -5 × 10 ³
D	GaAs-In _{0.06} Ga _{0.94} As	5	~5 × 10 ²
E ^b	GaAs-In _{0.06} Ga _{0.94} As	5	less than 10 ²
F ^c	GaAs-In _{0.14} Ga _{0.86} As	2	~2 × 10 ⁴

^aSubstrate EPD for both Si- and Cr-doped samples are of the order of 10⁵ cm⁻². EPD for epilayers grown directly on the substrate are in the 10⁴/cm⁻² range.

^bSi-doped substrates.

^cThe total thickness of the SLS is 1200 Å, which exceeds the critical thickness.

two successive GaAs-InGaAs SLS's. This structure, with a 3000-Å period, can be repeated to any desired number. Consequently, this will introduce more strained interfaces thereby blocking the threading dislocations originating from the substrate. In our study we have investigated samples composed of a 3000-Å period "structure," repeated two and five times with an InAs percentage of 6 and 10%.

Molten KOH was employed to reveal the dislocation density on the GaAs substrates and on the epitaxially grown layers. The etching time for each sample was 2-3 min. Etch pit densities for the substrates and the epitaxially grown GaAs cap layer on the Al_{0.3}Ga_{0.7}As-GaAs and In_xGa_{1-x}As-GaAs ($x = 0.06$ and 0.14) superlattices are compiled in Table I. The Al_{0.3}Ga_{0.7}As-GaAs SL reduces the EPD by one-third to one-fifth of that of the substrate. Similar studies on the GaAs-In_xGa_{1-x}As SLS indicate a two to three orders of magnitude reduction. This trend is clearly illustrated in Table I for both semi-insulating and n -type

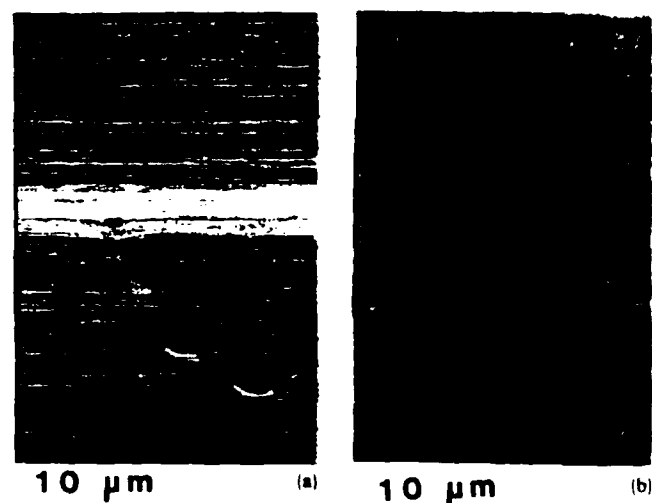
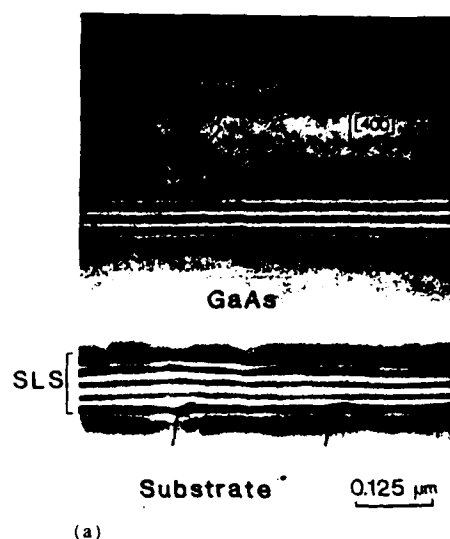
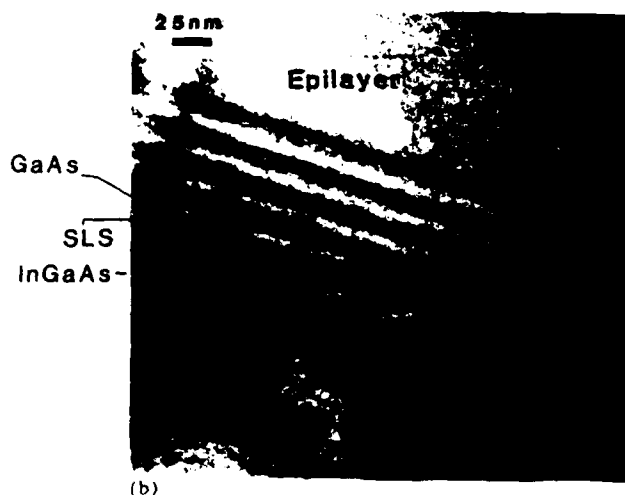


FIG. 2. (a) Y-modulation EBIC micrograph of recombination sites detected by surveying a 1-mm² device area. No other electrically active defects were observed in this device. The Y-modulation mode is used for enhancement of the contrast from small defects to facilitate survey scanning when covering large device areas. (b) Conventional (Z modulation) EBIC micrograph of the same area as above. Here dark areas represent lower induced current or higher recombination.



(a)



(b)

FIG. 3. TEM micrograph of GaAs-InGaAs superlattice. (a) Total thickness of the GaAs-In_{0.14}Ga_{0.86}As five-period SLS exceeds the critical thickness resulting in the generation of dislocations shown by arrows. (b) GaAs-In_{0.06}Ga_{0.94}As SLS composed of five periods of the 3000-Å "structures." Threading dislocations, shown by arrows, do not penetrate the SLS structure.

GaAs substrates. Increasing the number of five-period SLS and GaAs intermediate layers reduces the EPD, as shown for samples D and E in Table I. Samples D and E correspond to a 3000-Å period structure repeated five times.

Dislocation densities were also characterized using the electron beam induced current (EBIC) technique. 300-Å-thick gold Schottky diodes with an area of approximately 2×10^{-2} cm² were evaporated on the GaAs epilayers. An electron beam with an energy in the range 10-20 keV was injected through these gold Schottky diodes. Dislocations appear as dark images in the EBIC mode since they act as nonradiative recombination centers which reduce the electric current compared to the surrounding area. Figure 2 shows the EBIC image of sample D. Over the entire metalized area ($\sim 10^{-2}$ cm²) only four dark spots were observed. The corresponding dislocation density was approximately 4×10^2 /cm². This result compares favorably with the average value obtained from EPD data using a KOH etch (Table I).

Transmission electron microscopy (TEM) was also used to investigate threading dislocations for several SLS samples. The TEM samples were prepared by lapping and ion milling two wafers bonded face to face. They are viewed in cross section with the electron beam parallel to the $\langle 110 \rangle$ axis. Figure 3(a) shows a TEM cross section of the GaAs-In_{0.14}Ga_{0.86}As five-period SLS, with a total thickness of 1200 Å, which is larger than h_c . Misfit dislocations are generated at the SLS-GaAs interfaces as shown in Fig. 3(a). The presence of threading dislocations is consistent with the results shown in Table I for sample F, where the EPD is approximately one order of magnitude higher than those of samples B and C whose values of x are in the range of 6–10% and whose total thickness is less than 1000 Å. The GaAs and In_{0.14}Ga_{0.86}As layers in Fig. 3(a) do not appear to be strained. The strain and associated defects appear to be localized at the SLS and GaAs interfaces. This result is consistent with previous observations obtained from Raman spectroscopy⁷ for the accommodation of strain between GaAs-InGaAs SLS and the GaAs substrate. Figure 3(b) shows a GaAs-In_{0.06}Ga_{0.94}As SLS, with the 3000-Å structure, repeated five times. Dislocations originated from the substrate are stopped by the SLS structure.

In conclusion, GaAs-InGaAs SLS's have been successfully used as buffer layers to reduce dislocations originating

from GaAs substrates. Employing EPD and EBIC a reproducible set of defect densities has been recorded. An analysis of the propagation of these defects has been investigated using TEM. It is therefore expected that devices on these low defect density epilayers will exhibit less variation in electrical parameters than those fabricated directly on GaAs substrates.

The authors are grateful to M. Tischler for helpful discussions and assistance. This work is supported by the Air Force Office of Research.

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Defect Reduction in GaAs Epilayers on Si Substrates Using Strained Layer Superlattices

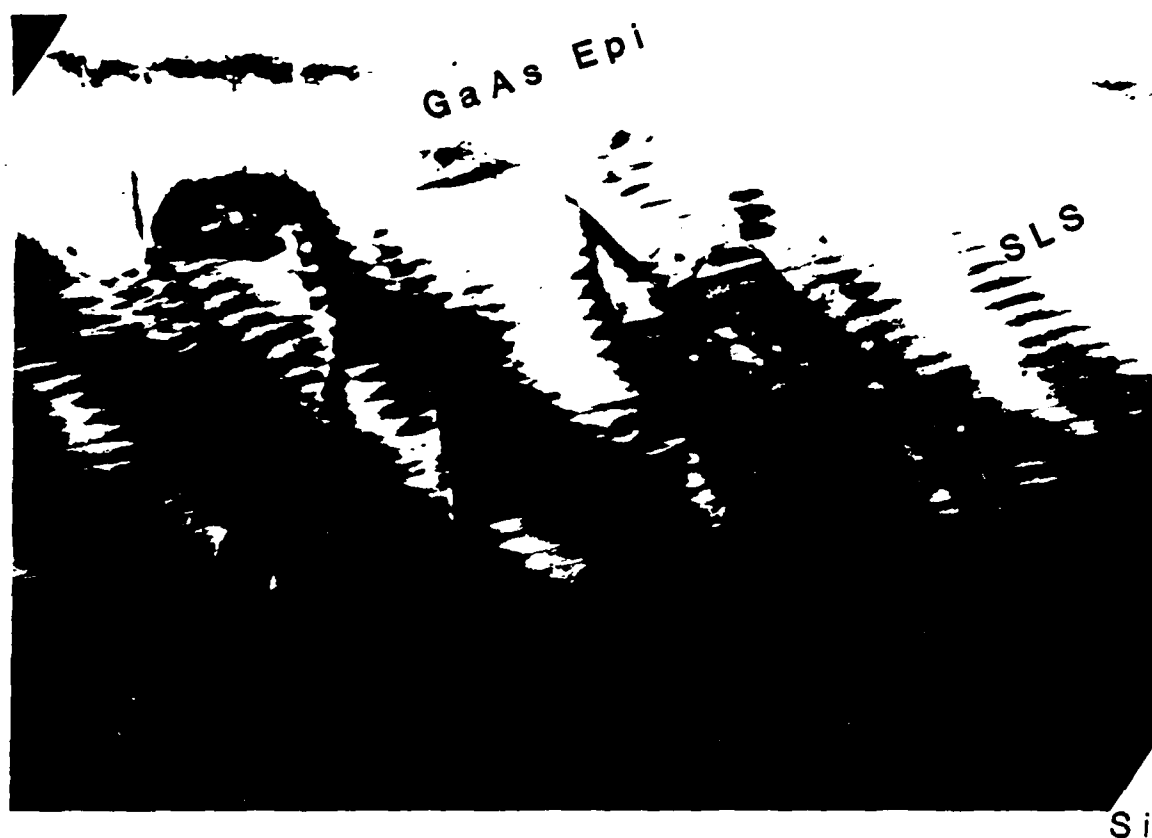
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The $\text{GaAs}_{1-y}\text{P}_y - \text{In}_x\text{Ga}_{1-x}\text{As}$ strained-layer superlattices "SLS" with $y = 2x$ has an average lattice constant equal to that of GaAs. This allows strained-layer heterostructures to be grown lattice matched on GaAs substrates. These superlattices, used as a buffer layer, have been found to significantly reduce the etch pit density on subsequently grown GaAs epitaxial layers compared to similar layers grown directly on GaAs substrates.

Initial results indicate that these two ternaries, SLS, is also effective in reducing dislocations in GaAs grown on silicon substrates. For example five periods, 300 Å, $\text{GaAs}_{0.8}\text{P}_{0.2} - \text{In}_{0.1}\text{Ga}_{0.9}\text{As}$ SLS buffer layers was found to reduce dislocation density by about two orders of magnitude as being indicated from TEM studies. We will report on the effect of layer compositions and thicknesses, strain, and the number SLS periods on the reduction of dislocations in GaAs films grown on silicon substrates.



**Use of GaAsP/InGaAs SLS Buffer to
Reduce Dislocations Originating from
GaAs Grown on Si Substrate**

LIFETIME TEST FOR HIGH CURRENT INJECTION
STRAINED LAYER SUPERLATTICE LIGHT EMITTING DIODE

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ABSTRACT

Successful long term room temperature operation of InGaAs/GaAsP strained layer superlattice light emitting diodes under high constant current injection is reported. The devices have been tested up to 1000h with 830 A/cm^2 , 500h with 3000 A/cm^2 and 450h with 4000 A/cm^2 with no observed degradation in the optical output. These results indicate that a SLS whose lattice constant is well matched to that of the substrate is stable under high-level current injection.

Recently, strained layer superlattices (SLSs) which consist of alternating thin layers of two semiconductors having different lattice constants in bulk crystal form have received considerable attention in electronic and optical device applications. The basic electronic and optical properties of the SLSs can be modified over a wide range by a proper choice of the material and geometrical parameters¹. A number of applications using those SLS structures in lasers²⁻⁶, LEDs⁷⁻⁹, photodetectors¹⁰⁻¹² and FETs¹³ has been reported. Although these SLSs have great flexibility in device design, their reliability has been questioned. In particular, it has been observed that under conditions where constant high-level excitation or rapid thermal cycling is required, the SLS devices are unstable⁴. It is noted, however, that many of SLS structures are not lattice matched to the substrate. Consequently, misfit dislocations are always generated at the interface between the SLS and the substrate when a total thickness of the SLS exceeds a strain-dependent critical thickness¹⁴. In order to avoid the generation of these misfit dislocations, device structures normally incorporate a buffer layer whose lattice constant is equal to the average lattice constant of the SLS. However misfit dislocations are still generated between the buffer layer and the substrate and propagate toward the SLS. This probably explains why SLS devices such as photopumped lasers are unstable especially under high-level excitation.

In this letter, we report a series of lifetime tests for InGaAs/GaAsP SLS light emitting diodes to investigate the reliability of these structures. Since the InGaAs/GaAsP SLS structure consists of alternating InGaAs and GaAsP layers with equal and opposite lattice mismatch with respect to the GaAs substrate, the average lattice constant can be matched to that of GaAs. Therefore, this SLS can be incorporated with GaAs and AlGaAs device struc-

tures without the formation of misfit dislocations at the heterointerface. Several potential applications of this SLS including LEDs⁷, defect reduction¹⁵ and solar cell¹⁶ have been reported.

The LED structure was grown by metalorganic chemical vapor deposition (MOCVD) at atmospheric pressure. Trimethylgallium (TMG), Triethylindium (TEI), AsH₃ and PH₃ were used as Ga, In, As and P sources, respectively. H₂Se and dimethylzinc (DMZ) were used as the n-type and p-type dopant, respectively. A schematic cross-section of the device is shown in Fig.1. First, a Se-doped 0.3 μ m GaAs ($n \sim 1 \times 10^{18} \text{ cm}^{-3}$) was grown on a (100) Si-doped GaAs substrate. Then an undoped 10 period In_{0.1}Ga_{0.9}As/GaAs_{0.8}P_{0.2} SLS active region was grown by injecting TEI and PH₃ alternately during the growth of GaAs. The thickness of each layer is about 100Å (total active region is about 2000Å). The mismatch between GaAs and the two compounds in bulk crystal form is $\pm 0.79\%$. Finally, a Zn-doped 0.5 μ m GaAs layer ($p \sim 1 \times 10^{18} \text{ cm}^{-3}$) was grown on the SLS. The growth temperature was 630°C for all these layers. A SiO₂ layer (2000~3000 Å) was deposited by plasma assisted CVD to make a 6 μ m-wide stripe structure. The wafer was thinned down to about 70 μ m and polished, then ohmic contacts were made by depositing Au-Sn-Au (100-200-1000Å) followed by annealing at 400°C for few minutes for n-type and Au-Cr-Au (100-200-1000Å) for p-type. Finally, the wafer was cleaved and sawed into individual diodes with typical dimensions of 250 x 300 μ m. The ideality factor for these diodes ranged between 2 and 3 over three orders of magnitude on a current scale.

Diodes were mounted to a gold plated copper block with the p-type face down and held by a spring-clip. The optical output was detected by a Si-photocell. Three current injection

levels, namely, 830, 3000 and 4000 A/cm² were used in our lifetime tests. Since the junction of the diode is formed less than 1 μm below the p-type electrode, the current spreading effect is not significant. It has been noted that the leakage current across the edges of the diode and through the SiO₂ isolating layer is less than a fraction of a percent of the total current flowing through the junction. Moreover, the optical output for a given input current was found to be independent of the size of the diode for the same 6 μm -wide stripe structure. Consequently, the current density is determined by assuming the effective current injection area of 10 x 250 μm for a stripe structure whose contact area is 6 x 250 μm .

Fig.2 shows the optical outputs as a function of operating time with constant current injection at room temperature. No degradation in the optical outputs has been observed. For instance, for a 830 A/cm² current injection, the diode (no stripe structure, full-surface metallization 125 x 125 μm) operates more than 1000 hours without degradation. It has also been observed that there is no degradation up to 500 hours and 450 hours for corresponding current densities of 3000 A/cm² and 4000 A/cm², respectively. By comparison, at 4000 A/cm², some of the devices failed after 6 to 10 hours operation. Although not shown in Fig.2, there are slight reductions (less than a few percent) in the optical outputs within the first hour. This reduction is somewhat larger for a diode which has no stripe structure than that of a stripe structure diode. This is probably because of a larger surface leakage current due to the structure which has no SiO₂ isolating layer. For stripe structure diode, such an initial reduction in the optical output is very small. Under a high level injection condition at which the optical output almost saturates, a slight reduction has been observed. This is mainly because of junction heating caused by the high current injection. In fact, above 4000 A/cm² the optical

output decreases gradually as the current density increases because of the heating effect at the junction.

Fig.3 shows typical emission spectra at 77 K and 290 K both before and after 500h operation with 3000 A/cm^2 current injection. The measurement were done with the current density of 200 A/cm^2 both before and after the aging test. After the aging test, the spectral peak position remained unchanged. However, we have observed a slight broadening of the emission spectra for each temperature measurement as illustrated in Fig.3. This broadening may be the result of some interdiffusion at the SLS interface. These preliminary observations suggest that a SLS which is lattice matched to the substrate is stable under high current injection condition and such a SLS structure has a great potential for practical device applications.

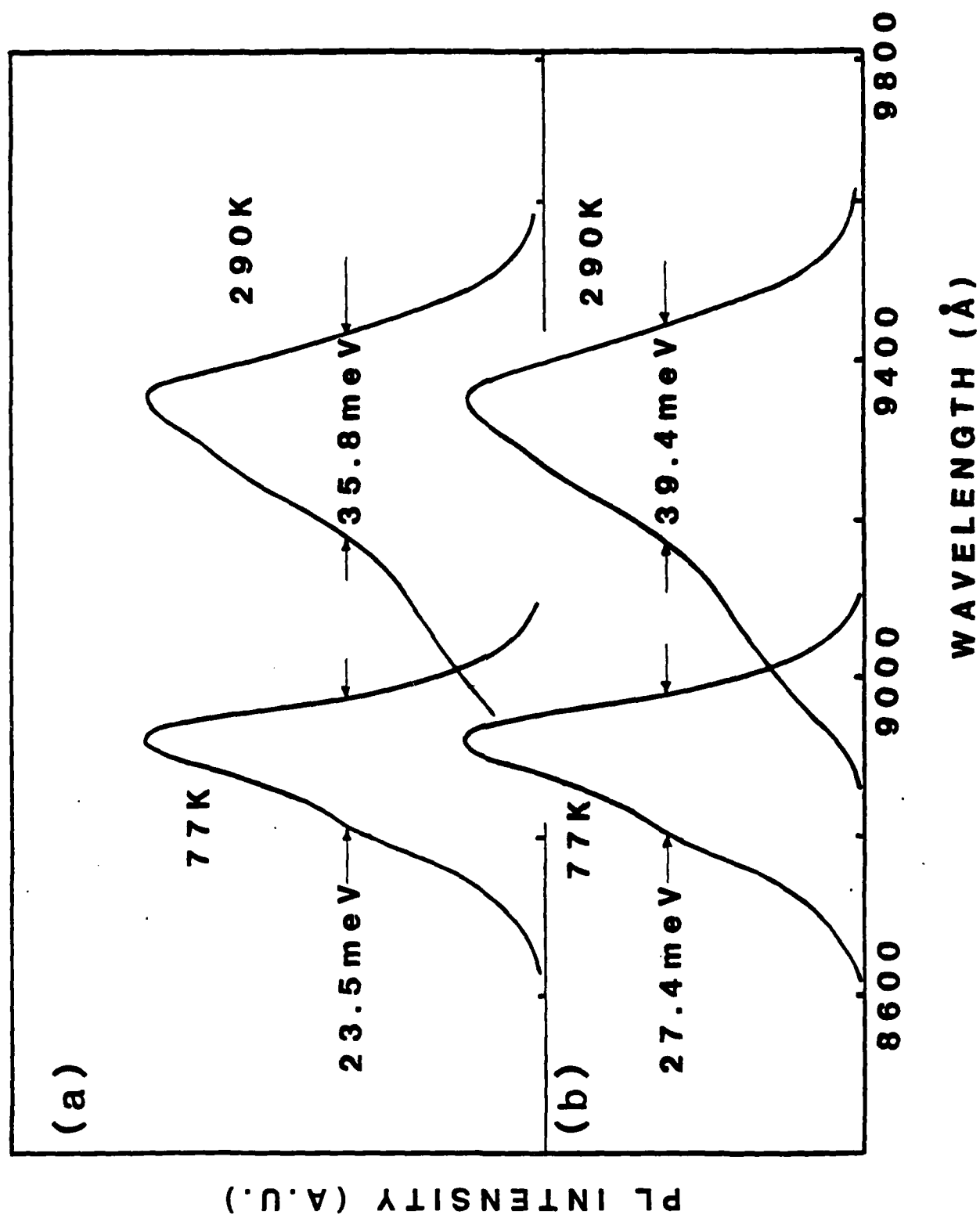
In conclusion, we have presented the lifetime test of InGaAs/GaAsP SLS LEDs to investigate the reliability of the SLS structure. No degradation has been observed up to 1000, 500 and 450 hours with 830, 3000 and 4000 A/cm^2 , respectively. This result indicates that the SLS which has a equal lattice constant to that of the substrate is stable under high current injection and has a great potential for practical SLS device applications.

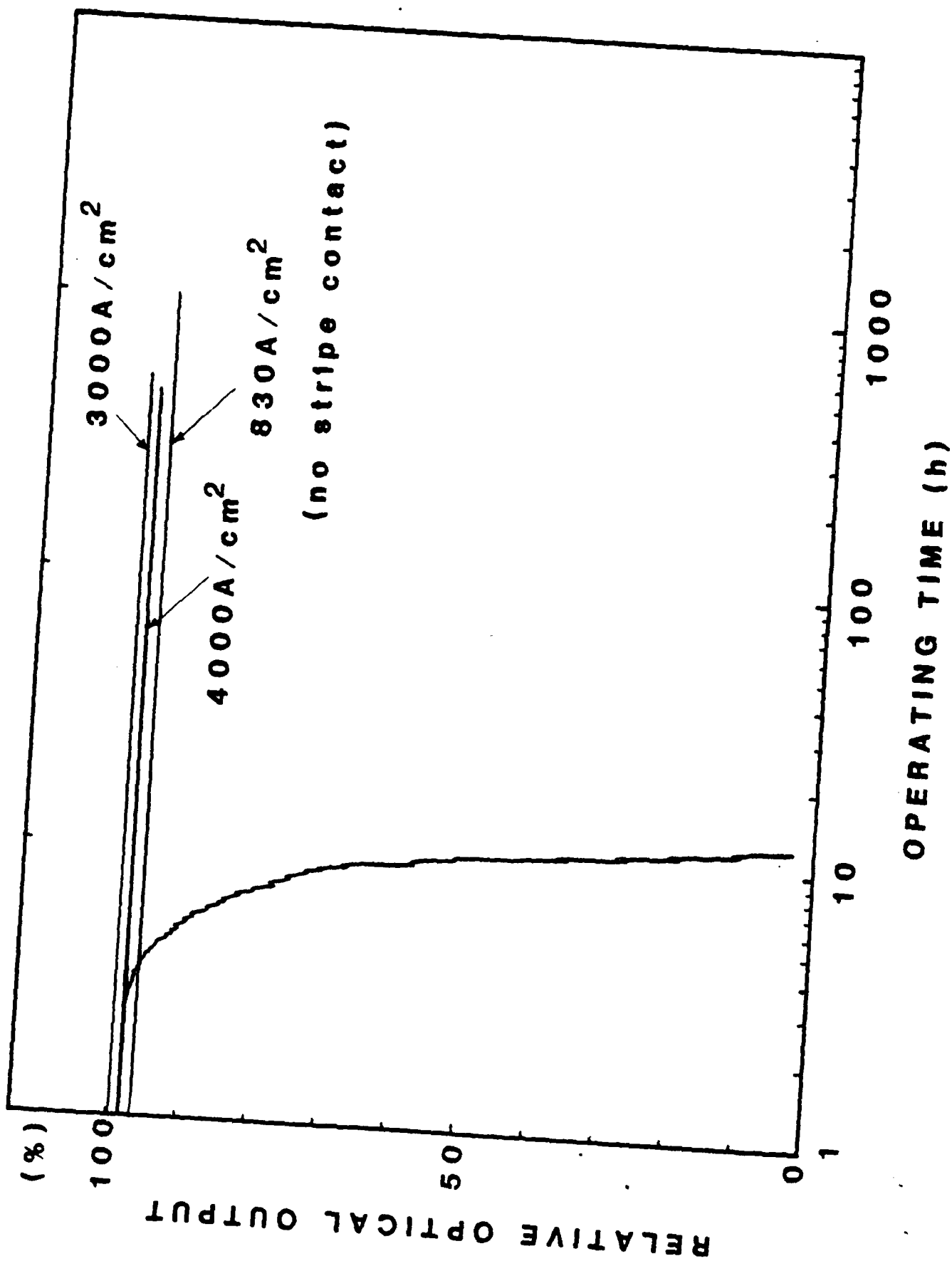
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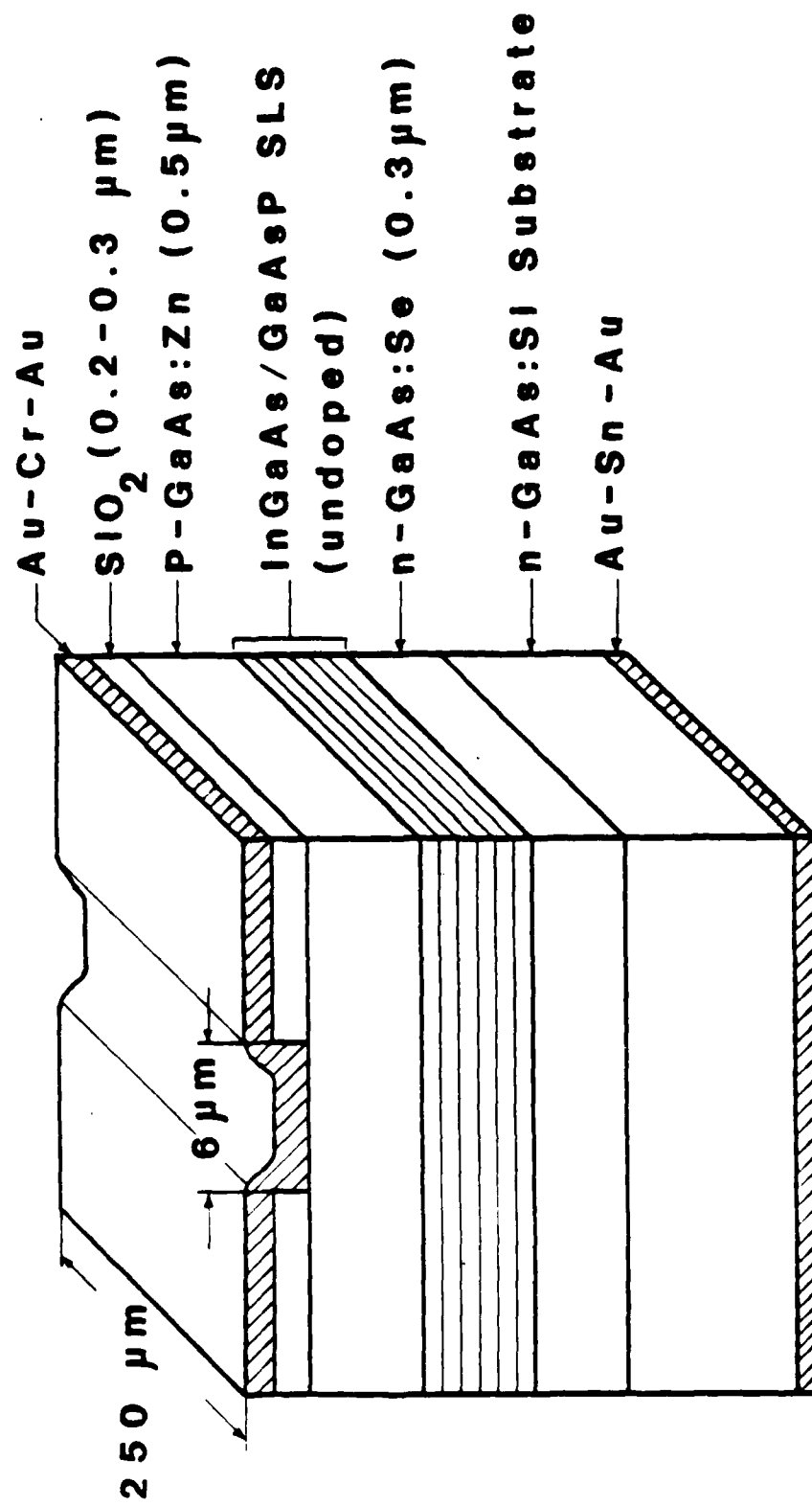


Fig.1 Schematic drawing of strained layer superlattice light emitting diode structure. The undoped active region consists of 10 periods $\text{In}_{0.1}\text{Ga}_{0.9}\text{As}/\text{GaAs}_{0.8}\text{P}_{0.2}$ SLS (each layer $\sim 100\text{\AA}$) whose lattice constant is equal to that of GaAs.

Fig.2 Optical outputs as a function of operating time. After the initial reduction (less than a few percent) no degradation has been observed.

Fig.3 Typical emission spectra of LED at 77K and 290K before (a) and after 500 hours operation with 3000 A/cm^2 current injection (b). The spectral peak position remained unchanged, while the spectral width caused slight broadening.

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