



ADA034078 DEVELOPMENT OF LARGE AREA MULTI-LAYER LIQUID PHASE EPITAXY FOR LARGE OPTICAL CAVITY LASER STRUCTURES 10 Robert GITT Laser Diode Laboratories, Inc. A Subsidiary of United Corp. 205 Forrest Street Metuchen, New Jersey 08840 fechnical Rep Final Dt. 120 18 M. U. S. Army Mobility Equipment Research and Development Center Night Vision Laboratory, USAECOM Contract No DAAK02-73-C-9254 Fort Belvoir, Virginia 22060 NER DISTRIBUTION STATEMENT: "Approved for Public Release; Distribution Unlimited: 1473 dn 405626-

FOREWARD

This Final Technical Report covers the work performed under Contract No. DAAK02-73-C-0254 between the period March 30, 1973 thru July 31, 1974.

This contract with Laser Diode Labs., was to fabricate by liquid phase epitaxy, large optical cavity GaAs laser structures having a minimum area of two (2) square centimeters of usable material. Program technical direction was provided by Mr. Richard R. Schurtz, Night Vision Laboratory, Fort Belvoir, Virginia. The work was performed at Laser Diode Laboratories, Metuchen, New Jersey under the direction of R. B. Gill.

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ABSTRACT

The development of a liquid phase eptixaial process capable of yielding GaAlAs-GaAs multiple layer structures from which efficient lasers can be fabricated is discussed. The furnace facility is described in detail and the development of the LPE process is described. The application of various LPE processes to the growth of both large optical cavity and symmetrical optical cavity lasers is presented. Data is shown which depicts performance characteristics and emission patterns for the resultant devices.

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SECTION I

OBJECTIVE

The objective of this program is to fabricate, by liquid phase epitaxy, large optical cavity laser structures having a minimum cross-sectional area of two square centimeters and to demonstrate the quality of these structures by the characterization of injection lasers constructed from these wafers. To accomplish this objective the technical effort was divided into several parallel programs:

A. <u>Laser Structure Specification</u>. To identify the multiheterojunction laser configuration required for the fabrication of injection lasers meeting the technical requirements described under DAAK02-73-C-0254 dated 30 March, 1973.

B. <u>Liquid Phase Epitaxial Technology.</u> To develop a liquid phase epitaxial facility and epitaxial process required for the growth of large optical cavity laser structures.

C. <u>Device Evaluation</u>. To continuously evaluate laser diodes fabricated from the epitaxial layers and to correlate the lasing characteristics of these diodes to the laser structure and growth conditions.

SECTION II

RESULTS AND DISCUSSION

A. General Requirements

The overall objective of this program was to fabricate, by liquid phase epitaxy, large optical cavity injection laser structures having a minimum cross-sectional area of two square centimeters and to demonstrate the quality and uniformity of these epitaxial wafers by the characterization of injection lasers constructed from these wafers. The objective performance characteristics of the injectic lasers are summarized in Table 1. Two types of laser structures were investigated during this program to achieve the desired device performance. Initially laser diodes which utilized the multi-heterojunction laser cavity structure described by Lockwood et al' in 1970 were fabricated. An outline of this type of laser structure is shown schematically in Fig. 1. The structure itself is formed in a single thermal cycle by sequential growth of layers of GaAlAs and GaAs onto (100) single crystal GaAs substrate by liquid phase epitaxy. The first grown layer (1) is n-type GaAs which serves as an interface between the following (GaAl)As layer (2) and the substrate material. While this first layer is not necessary to the active laser structure, it generally absorbs submicron nonuniformities in the first growth interface and provided a uniform flat interface for the growth of the following layer.





Layer 2 consists of n-type (GaAl)As doped with either Te or Sn and serves to confine the stimulated radiation and lasing mode to the optical waveguide D_c through its heterojunction interface with layer 3.

Layer 3, which forms the bulk of the waveguide region, consists of either lightly doped or undoped n-type GaAs. Its thickness (D_C-D) strongly effects the laser threshold current density, the primary propagating mode order, the emitted optical beam pattern and catastrophic damage level, while its free electron concentration effects both the lasing threshold and quantum efficiency. Layer 5 consists of P-type (GaAl)As whose interface with Layer 4 serves both as an optical barrier to confine the radiation and lasing mode to the waveguide region and as a potential barrier to confine the electrons injected from layer 3 into layer 4 within layer 4 where the radiative recombination occurs.

Finally, layer 6, consisting of p-type GaAs is grown to facilitate ohmic contacting to the P side of the structure. While not essential to the structure it does provide a surface that can be contacted relatively easily if a thin (GaAl)As layer is desired for layer 5.

As a general rule, for this structure, the energy gaps and refractive indices of the layers have the following relationship:

Egl	2	Eg ₃	~	Eg4	a	Eg6	2	Egsul
n 1	a	n ₃	۲	n4	2	n ₆	2	nsub
		Eg5	>	Eg2	>	Eg3		

 $n_5 < n_2 < n_3$ and

for an asymmetrical LOC laser:

After considerable effort directed toward optimizing the epitaxial process required for the generation of the large optical cavity laser a parallel effort was established toward evaluation of a laser structure in which the injected carriers and optical wave would be separately confined.

This structure which is schematically represented in Figure 2 was first demonstrated by Panish² at Bell Telephone Laboratories in 1973. In this structure the injected carriers are confined to the p-type GaAs (region 3) by the heterojunctions located at its interface with the P and N type GaAlAs layers. On the other hand the optical wave is confined 1-x xby the GaAlAs - GaAlAs heterojunctions which defines then a 1-y yheterostructural optical cavity consisting of regions 3,4 & 5.



TABLE I

27°C OBJECTIVE PERFORMANCE CHARACTERISTICS OF LOC GAAS INJECTION LASERS AT

Threshold Current Density	7KAcm ⁻² (Max.)
Differential Quantrum Efficiency	35%
Emission Wavelength	$8950 \pm 50^{\circ}_{\rm A}$
Peak Power Output A 200ns pulse width and 2KHS	1.5 watts/mil
Beam Spread, full angle at 10% points	35°

laser facet **

of

Without use of an antireflective coating **

This configuration results in a symmetrical optical cavity (SOC) GaAs laser structure. Results on devices of this type as reported by Panish² indicated the possibility to fabricate high power lasers operating in the first order transverse mode with lasing threshold current densities less than 5KAcm⁻² at 27°C.

Materials Technology

B.

1. Substrate Requirements

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The Gallas-Gals multi-heterojunction laser structure required for both LOC and SOC injection lasers is formed by multiple liquid phase epitaxial growth onto Gals substrates. All of the substrate wafers used in this program were selected from high quanlity Gals single crystal ingots grown at LDL by the horizontal gradient freeze technique. These substrates were typically n-type with a net donor concentration lying between 1.5 and 2.5 x 10^{18} cm⁻³, contained dislocation densities less than 1500cm⁻² and were doped with either Te, Sn or Si. Surface orientation was maintained at 100 ± 0.3 degrees to minimize growth defects due to misorientation. All wafers were polished prior to growth using standard commercial techniques.

2. Liquid Phase Epitaxy

a.

Furnace Design

The multi-layer GaAlAs-Gaas structures required for the fabrication of either LOC or SOC GaAs injection lasers are generated by the growth of the structure by liquid phase epitaxy onto GaAs substrate in a single growth operation using a multiple melt epitaxial boat. Since best results are obtained when horizontal thermal gradients across the boat are minimized, the epitaxial furnace must have a long flat zone which can be programmed to change uniformily at rates ranging from 0.1°C to 2°C per minute at about 850°C. Since most commercial furnaces which meet this requirement are both large and complex, (multiple zone), a special roller mounted furnace system utilizing a heat pipe core was designed and constructed. The major elements of the growth system developed under this contract were patterned after a system already in use at the Night Vision Laboratory, which included a heat pipe core furnace, a Vac-Sorb pump and check valve evacuation system, a slow cool temperature regulation equipment, and a single end supported growth chamber to allow furnace removal for fact cycling. This furnace system which was used throughout the program is shown in Figure 3.

The furnace chamber which is critical to uniform epitaxy was constructed at LDL. The furnace core consists of #12 Kanthal A-L wire wound onto a 24 ich 1000 25 2.5 theb. Dece Cyntchere Inschurant



Figure 3. Furnace system used for multi-layer liquid phase epitaxy.

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inch long by 2.5 inch bore Dynathern Isothermal Furnace liner. Electrical isolation between the heater element and the metallic core was achieved by using eight 1/4 inch diameter alumina thermocouple sleeves mounted to the outside diameter of the core as insulating stand-offs between the core and the winding. After winding, the furnace core was insulated with one layer of 100 series zircar This element was then mounted into a Marshall felt. clam shell furnace frame which had been stripped of its original heater element and insulation. Three shaped firebricks were used to position the furnace core at the center of the clam shell enclosure. The entire enclosure was then packed with fiber frax to reduce heat loss. With the furnace growth tube inserted into the core and a heat reflector positioned at one end of the core this chamber provides a thermal flat zone at 900°C)+ 0.2°C) of at least 20 inches.

Temperature controlled during epitaxy is provided by a continuously programmable furnace power supply. Using a modified Data Trak Controllerprogrammer combination, a full scale programmable interval of 15°C lying between 800°C and 900°C can be selected for epitaxy.

Initially two platinum-platinum plus 13% rhodium thermocouples were used to control and sense furnace temperature. One was placed within the furnace core and the second within the quartz growth tube. When the furnace was in the standby position the furnace thermocouple was used to maintain the furnace core at 850°C, when the growth tube with the epitaxial boat positioned within it was inserted into the furnace, furnace thermal monitoring and control was switched to the second thermocouple which was mounted within the tube. This set up proved to be unsatisfactory due to frequent damage to the tube thermocouple during growth and lack of thermal reproducibility and stability. The dual thermocouple arrangements was replaced by a single thermocouple mounted on the isothermal furnace liner wall. Results with this system have been very satisfactory with current reproducibility estimated to be approximately 0.3°C at 850°C and stability better than 0.1°C.

The epitaxial growth chamber consists of a quartz tube secured with an "O" ring seal to the stainless steel furnace head. Initially viton

"O" rings were used but these proved unsatisfactory after a number of growth cycles. Currently silicone "O: rings are used at the glass to metal seal.

The furnace head contains the ambient gas input and exhaust lines, a thermocouple guage for monitoring system gas pressure and a vacuum line for exhausting the system during pre-growth purging. The furnace head is sealed at the loading end with a stainless steel flange which contains the quartz push rod used to propel the epitaxial boat slide and cause transport of the wafer from one bin to another during epitaxy.

A Vac Sorp cryogenic pump is used to evacuate the growth tube, input gas lines, and hydrogen purifier to insure removal of both O_2 and H_2O from the system and to check the integrity of the system's joints and seals prior to each growth cycle. This pump system provides for the rapid exhausting of the system to less than 10^{-2} torr without risk of contamination due to backstreaming, a problem frequently encountered in systems employing oil based mechanical vacuum pumps.

The gas outlet line contains a high vacuum spring loaded check valve to provide a positive

system pressure during epitaxy and sealing of the lines during the vacuum exhaust cycles. Initially the valve was mounted directly onto the furnace head but due to rapid failure caused by thermal cycling and comtamination of the sealing ball it was subsequently installed in the output line about 12 inches downstream from the furnace head. A Matheson #401X stainless steel valve which is regularly disassembled and cleaned to provide uncontaminated sealing surfaces is used for this function.

b.

Epitaxial Boat Design

All of the epitaxy conducted during this program utilized multiple bin graphite epitaxial boat which incorporated a sliding wafer holder to transport the substrate wafer from one melt to another during growth to achieve multiple layered growth within a single thermal cycle. A typical LPE boat used in this program is shown in Figure 4. The basic boat consists of three components, a four walled support frame, the multiple bin melt container and the substrate holder. In this design the bin assembly is placed into the support frame where its base rests totally on the sliding substrate holder. Direct broad area contact is therefore maintained between the

boat LPE Multi-bin graphite 4. F1g.

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bin assembly and slide at all times to assure proper melt wiping during transport of the substrate from one melt to another during growth. In addition, this construction causes the contacted surfaces which wear during repeated processes to self compensate for the minor deviations in mechanical tolerances which naturally occur. In addition, the number of melts or melt cross-section to be utilized in any experiment can be economically varied since a substitute or experimental bin assembly can be easily inserted into the support frame as required without replacement of or modification to the remainder of the boat. In practice this approach was used to modify the initial boats from five bin to seven bin assemblies. Finally the graphite plugs also shown in Figure 4 are placed over the growth melts prior to epitaxy to permin use of minimal melt charges while assuring uniform distribution of the melts across the substrate surface.

Epitaxial Process and Results

3.

A typical epitaxial growth sequence is initiated by loading each boat melt bin with the required (5 gm.) charge of 69 or 79 purity Gallium. After loading the boat is inserted into the quartz reactor tube which is then sealed and purged with dry nitrogen. After purging the entire furnace system including gas feed lines are evacuated to at least 10^{-2} torr to remove residual H₂O and O₂ and to insure the integrity of the systems joints and seals. Next high purity paladium diffused hydrogen is bled into the system until its pressure releases the check valve on the exhaust lines permiting normal gas flow through the furnace system. At this point, the preheated furnace chamber is rolled over the furnace tube and the Gallium baked at 850°C for 20 minutes to reduce the surface oxides which form on the Gallium metal. After bake out the furnace chamber is rolled back away from the growth tube and the melts and boat permitted to slowly cool to ambient temperatures. After cooling the system is once again flushed with dry nitrogen and opened. The pre-weighed melt dopants including sufficient Gallium Arsenide to saturate the melts at the growth temperatures are then added to the melts. After inserting a freshly etched and polished substrate wafer into the substrate holder the

boat is inserted into the tube, the tube sealed and the furnace evacuation and purging process previously described repeated. The furnace chamber is then rolled over the growth tube and the boat heated to 850°C to insure melt saturation. Following the saturation cycle, the furnace temperature control is switched to the thermal program and the growth cycle initiated. Using a quartz push rod in contact with the sliding substrate holder the substrate wafer is pushed under the first growth melt and growth initiated at a stabilized cooling rate of 0.22°C per minute. After the prescribed growth interval the substrate holder is again pushed to slide the wafer from the first melt to the second. This process is repeated for each melt to generate the required epitaxial structure. After growth of the final layer the substrate is pushed clear of the boat to terminate growth and wipe the residual gallium from its surface. The furnace chamber is then removed from the furnace tube, the tube cooled, nitrogen purged and opened. After removal of boat from the furnace tube the epitaxial wafer is cleaned in hot HCL, cross-sectioned, etched to delineate the metalurgical interfaces and inspected using a differential interference microscope Typical results of successful multi-layer at 1000X.

epitaxy are shown in Figure 5 for a large optical cavity laser and in Figure 6 for a symmetrical optical cavity device. The specific details of epitaxial processes used to generate these structures which resulted in efficient multi-heterojunction GaAs lasers are shown in Table 2 and 3. During the development of the two processes just described numerous growth conditions and substrate preparation techniques were evaluated to improve the uniformity of the structures and repeatability of the growth process. It was found that the thickness and surface quality of the substrate were critical to epitaxial layer uniformity and flatness. Using a .016 inch recess in the slide to hold the substrate during epitaxy, best results are obtained with a substrate 0.014 inches thick. A thinner substrate results in layer non-uniformities due to melt carry over from one bin to the next during transfer of the seed wafer, while thicker substrates cause line growth defects in growth layers which are parallel to the sliding direction. This is primarily due to mechanical abrasion of the layer surface during the substrate transfer from one bin to the next. Surface defects or contaimination generally result in non-uniform wetting of the substrate during the growth of the first GaAs or transition layer.





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Actual Cross-section of epitaxial wafer used for the fabrication of GaAs SOC lasers (1000X). Figure 6.



TABLE 2

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LPE Process for LOC Laser Fabrication

	Melt C	omposit:	ion		Growth Time
ga.	GaAs mg.	Dope	ants mg.	Al mg.	Minutes
ß	400	15	Sn		15
S	425	15	Sn	ß	15
2	450				Э
2	450	10	Ge		.25
2	425	50	Ge	2	2
S	440	25	Ge		26

Initial Growth Time 845°C

Growth Rate 0.22°C/minute

TABLE 3

Same and the second

LPE Process for SOC Laser Fabrication

	Growth Time Minutes	17	18	4	.25	4	18	2
	Al mg.		7	1.2		1.0	10	
Composition	Dopants mg.	10 Sn	10 Sn	5 Sn	15 Si, 10 Ge	25 Ge	50 Ge	25 Ge
Melt	GaAs mg.	400	400	435	450	450	425	440
	Ga gm.	ŝ	ŝ	S	ŝ	S	S	5

•

These growth defects are then propagated through all subsequent layers rendering the structure useless for device fabrication.

The effect of substrate meltback during growth of the transition layer on the uniformity of the total structure was also determined. Melt composition was adjusted to vary substrate meltback from 0 to about 0.002 inches. Best results were obtained for layers grown after an initial 0.001 inch substrate meltback. Excessive meltback again produced melt carry-over while insufficient or zero meltback caused a non-uniform. uneven first metallurgical interface that affected all subsequent layers.

Several methods of saturating the melts with GaAs at the growth temperature were also evaluated. Initially single crystal GaAs slugs which were shaped to bin cross-section were placed over the Gallium to both uniformity distribute the Gallium across the bin and provide source of Gallium Arsenide for melt saturation.

This approach was abandoned after several growth attempts when it became evident that uniform melt distribution across the bin base was not achieved and the grown layers were highly irregular and contained discontinuities. Improved results were obtained by saturing the melts with weighed GaAs chunks and using a graphite melt cover to distribute a thin(~.100 inch) melt across the base of the bin.

Several methods were evaluated in attempts to improve the surface quality of the last grown layer. Ideally if all the Gallium could be removed from the substrate surface after growth of the last layer but prior to furnace cool down a final surface that could be contacted directly for laser processing would result. This would eliminate several processing steps as well as positioning the junction closer to the heat sink for improved thermal conduction during device operation. Initially, all runs were pushed completely clear of the last bin after growth and the tube removed from the furnace chamber to permit rapid cooling to ambient. However, this initial attempts were frustrated due to lifting of the boat bin assembly during the sliding motion which resulted in substantial melt carry over and a large surface residue after the final wiping action. The addition of two graphite screws which hold the bin assembly against the slide during the growth

process eliminated this problem and permitted growth of final layers as thin as 2 microns with nearly Gallium free surfaces. Several attempts to fabricate lasers from these structures yielded only shorted devices. Examination of these failures indicated that the metalization process was incompatible with a 2 micron surface layer. Contact sintering as required by the existing process appeared to cause alloying beyond the depth of the junction thereby either shorting or damaging it. To eliminate this problem the substrate slice was permitted to remain under the last melt until the system cooled to ambient. This resulted in a final layer thickness of 50+ microns. Unfortunately these layers were characterized by both Gallium inclusions after about 25 microns growth, as well as an extremely poor surface due to Germanium growth on the surface during the final stages of cooling. A cross-section of one of these structures in shown in Figure 7.

Toward the end of the program several additional attempts were made to grow a top layer with a contactable surface without further processing. In these latter growths the top layer thickness was maintained at between 6 and 10 microns with removal of the substrate from the



Figure 7. Cross-section of an epitaxial slice cooled in last bin to room temperature. Top layer is very thick with Ga inclusions and Ge on

the surface. Run FR2-88 100X

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last melt used to terminate growth. Effective melt wiping was achieved and the structures were contacted without apparent detrimental effects to fabricated devices. Other parameters which affected growth quality included optimization of melt saturation and development of process conditions and controls which assured an oxygen free growth environment. Examples of defects caused by improper melt saturation or O₂ contamination of the growth environment are shown in Figures 8 and 9. During the course of this work over ninty epitaxial structures were generated. Of these nearly 55% were of insufficient quality to justify device fabrication. Many of the defective layers resulted from process experimentation necessary to develop an understanding of the furnace system and the process sequence required for reproducible epitaxy. Toward the second half of the program epitaxial problems were largely resolved, therefore systematic device optimization and evaluation using uniform 2.7cm² epitaxial wafers became feasible.



Figure 8. Excess GaAs in last bin - damaged surface as wafer was pushed out of last bin. Run FR2-8 1000X.



Figure 9. Non-uniform growth resulting from oxygen contamination of growth ambient. Run FR2-44 1000X.

Device Results

C.

The multi-heterojunction GaAlAs-GaAs epitaxial structure grown during this program were continuously processed into laser die which were then evaluated to determine the suitability of the materials for laser fabrication. Initially device results were quite eratic with most wafers containing sufficiently large growth irregularities to render further processing and evaluation unmeaningful. As experience was gained in understanding the characteristics of the growth furnace and epitaxial boat, epitaxial structure quality improved making laser evaluation and characterization possible. Two types of device structures were evaluated, the large optical cavity laser and the symmetrical optical cavity device. Most of the effort was directed to the former wafers which yielded satisfactiry devices of each type. The growth experience gained in LOC laser structure generation made possible the rapid successful fabrication of SOC laser structures. Performance results for both types of lasers are shown in Figures 10 and 11. LOC lasers were fabricated having optical cavities ranging from 1.5 to 40µm and recombination regions restricted to less of 0.5µm. The lasing threshold currents for these devices ranged from 6KAcm⁻² to 18KAcm⁻² at 27°C. Differential quantum efficiencies up to 0.59 W/A were observed on non-reflective coated lasers and up to 0.44 W/A on reflective coated devices. Emitted beam







patterns were highly dependent on optical cavity width. Typical results are shown in Figures 12 and 13. Best results were obtained for optical cavities of about 2.2µm. Devices having wider cavities were characterized by high order transverse optical modes which resulted in dispersed non-uniform far field emission patterns.

Far field emission patterns of the SOC lasers were generally more uniform than those of the LOC devices. Typical data taken during the course of structure optimization for the SOC devices are shown in Figures 14, 15 and 16 for the far field beam pattern perpendicular to the plane of the junction. Initially beams having 25 to 30 degrees full angle divergence at the 50% intensity points were consistently observed, however, by reducing the Aluminum content at the interior heterojunction beam divergence was reduced to about 18° degrees for devices fabricated near the end of the program. Additional reductions in beam divergence would be expected by reducing the Al concentration at the exterior heretojunctions which define the boundaries of the optical cavity. A second but no less important result of the reduced Al content at the interior heterojunction has been a marked increase in the threshold for catistrophic facet damage during laser operation. Initial SOC devices were damaged at emission intensities of about 0.5 watts per mil when driven with 60-75 nanosecond current pulses at 5 KH2. Recent devices exhibiting improved far field patterns have been operated at emission intensities up to 1.4 watts per mil at 60-75 nanoseconds and 15 KHZ. Further improvements in this area could also be anticipated by continued structure optimization. A typical









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emission pattern for a SOC laser in the plane of the junction is shown in Figure 17.

All of the laser structures generated during this program utilized either Ge or Ge:Si as the P dopants. Typical 27°C emission wavelengths ranged from 886.0 to 899.5 nm for the Si:Ge doped material and from 883.5 to 885.5 for the Ge doped wafers. No evidence of Al carry-over from the Al doped melts to the GaAs melts was observed.

CONCLUSIONS

The primary task of the program namely the development of a liquid phase epitaxial growth environmental and process suitable for the reproducible growth of large area (>2cm²), GaAlAs-GaAs multiple heterojunction structures suitable for the generation of efficient large optical cavity GaAs lasers has been achieved. A furnace system in which this epitaxy can be conducted has been demonstrated and suitable epitaxial processes for the growth of both LOC and SOC GaAs lasers developed. Many approaches to process optimization have been evaluated including methods of melt saturation, effect of surface substrate preparation, various growth rates and melt purification by vacuum or hydrogen firing on growth uniformity. Efficient LOC and SOC devices have been fabricated with the latter offering the potential of achieving all device performance goals including a high catistrophic failure level. In addition further structure and growth optimization for the SOC lasers also seem to offer continued reduction in emission pattern. Both LOC and SOC have achieved the desired threshold current density and efficiency at 27°C. However, SOC devices appear to posess lower lasing thresholds than comparable LOC devices offering the possibility of high peak power emission at low (20-30 KAcm⁻²) drive current densities.

SECTION IV

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