

[54] **MULTIPLE ANODIZATION SCHEME FOR PRODUCING GAAS LAYERS OF NONUNIFORM THICKNESS**

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[58] Field of Search **204/15, 42, 56 R, 57, 14 N**

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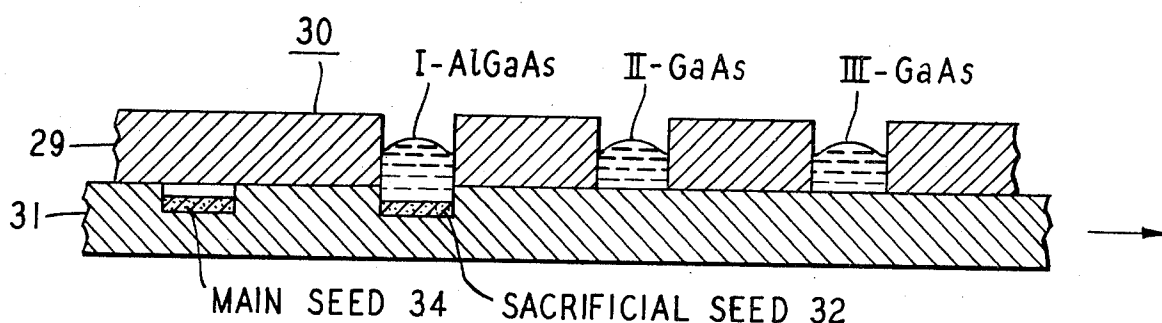
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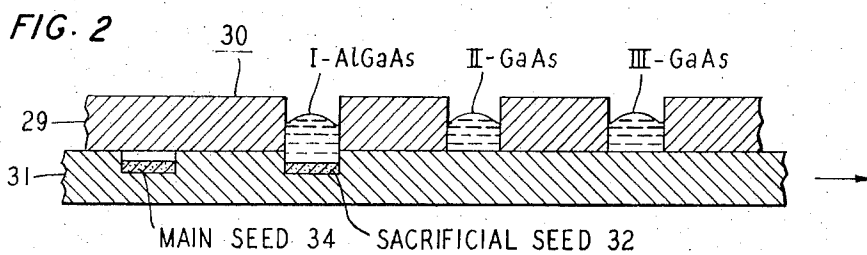
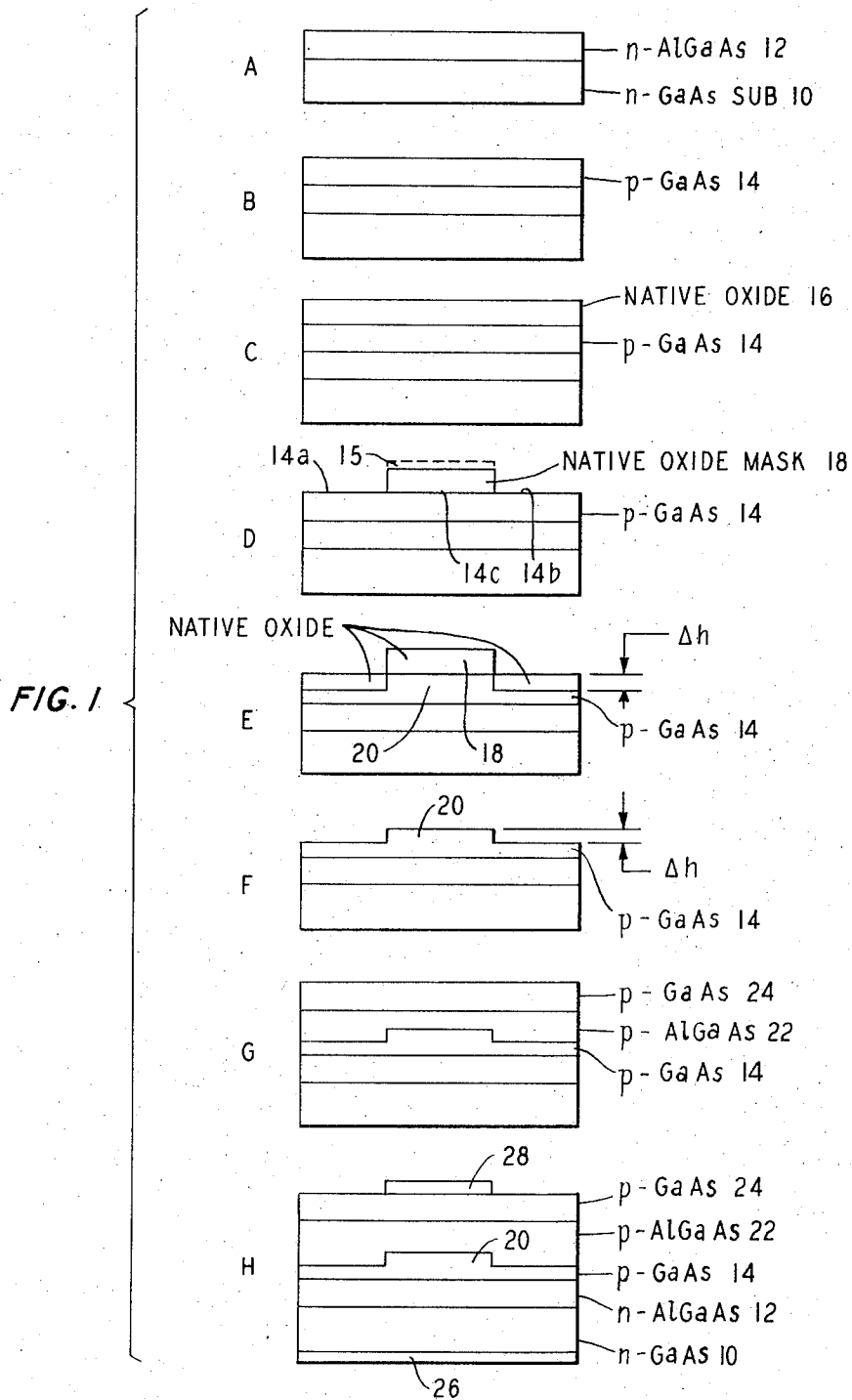
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[57] **ABSTRACT**

A multiple anodization technique is described for producing GaAs layers of nonuniform thickness; that is, GaAs layers having substantially rectangular steps or grooves of the type capable of guiding light. The technique includes the following steps: (1) growing a native oxide layer on a major surface of the GaAs layer by submersing the GaAs layer in an anodization bath of concentrated H_2O_2 having a pH less than 6; (2) removing selected portions of the oxide layer so as to expose the GaAs layer adjacent to the desired step region; and (3) immersing the surface again in an anodization bath of concentrated H_2O_2 having a pH less than 6. Also described are techniques for growing by liquid phase epitaxy an AlGaAs layer over the resultant GaAs step structure in a manner which alleviates two problems: the formation of deleterious oxides on the GaAs layer and the dissolving of the step configuration while in contact with the AlGaAs growth solution.

15 Claims, 2 Drawing Figures





MULTIPLE ANODIZATION SCHEME FOR PRODUCING GAAS LAYERS OF NONUNIFORM THICKNESS

CROSS-REFERENCE TO RELATED APPLICATIONS

This application was concurrently filed with application Ser. No. 434,181 (S. E. Miller) entitled "Single Transverse Mode Operation in Double Heterostructure Junction Lasers Having an Active Layer of Nonuniform Thickness."

BACKGROUND OF THE INVENTION

This invention relates to the fabrication of dielectric optical waveguides and, more particularly, to the fabrication of such waveguides by a multiple or repetitive anodization procedure applied to GaAs based semiconductor layers.

In copending application Ser. No. 308,833, filed Nov. 22, 1972 now U.S. Pat. No. 3,813,141, S. E. Miller teaches that a properly constructed rectangular step in an otherwise planar layer can be made to guide light in a single transverse mode. In this regard, solution of the boundary value problem associated with the structure for single mode operation gives rise to a family of curves which relate the maximum width of the rectangular step or rib to the step height. Although S. E. Miller proposed that his structure be fabricated of glass for use as an optical fiber (now referred to as a single-material fiber), he also teaches in the above-identified, concurrently filed application that a similar structure fabricated from GaAs can be incorporated into a double heterostructure (DH) junction laser to effect fundamental transverse mode operation parallel to the junction plane. Hereinafter such a DH laser will be referred to as "Miller" laser.

In order to produce the desired step in the GaAs layers, a step which might typically be in the range of 100 to 1000 Angstroms, one skilled in the art would probably first consider etching the GaAs layer. However, known etching procedures provide inadequate control of etching depth and layer uniformity to reproducibly form such small steps.

SUMMARY OF THE INVENTION

We have found, however, that more than adequate uniformity, control, and reproducibility of such small steps in GaAs based layers can be attained by a procedure which utilizes the following steps: (1) growing a native oxide layer on a major surface of the GaAs layer by submersing the GaAs layer in an anodization bath of concentrated H_2O_2 having a pH less than 6; (2) removing selected portions of the oxide layer so as to expose the GaAs layer adjacent to the desired step region; and (3) immersing the surface again in an anodization bath comprising concentrated H_2O_2 and having a pH less than 6. The anodization procedure is of the type described by B. Schwartz in application Ser. No. 292,127 filed on Sept. 25, 1972 now U.S. Pat. No. 3,798,139 but takes advantage of the fact that when ingot grown GaAs is subjected to a concentrated H_2O_2 anodization bath having a pH of 2.0, for example, 18.6 Angstroms of native oxide is grown and simultaneously 12.5 Angstroms of GaAs is consumed for each volt of applied anodization voltage. The consumed GaAs is incorporated into the grown oxide. On the other hand, for GaAs epitaxially grown from Ga solution (LPE), we

have found that the corresponding rates are 11 Angstroms/V of oxide formed and 7.4 Angstroms/V of GaAs consumed. Thus, a particular step height can be accurately fabricated by choosing an appropriate anodization voltage. For example, if step (1) utilizes an anodization voltage of 100 V., about 1,860 Angstroms of oxide is grown and about 1,250 Angstroms of GaAs is consumed, the latter being incorporated into the grown oxide. After suitable masking, as defined by step (2), a 100 Angstrom step can be fabricated in step (3) by anodization about 8 V. for ingot grown GaAs and at about 13.5 V. for LPE grown GaAs.

In the foregoing procedure, the step height was determined by the second anodization voltage applied in step (3) because it was lower than the first anodization voltage applied in step (1). However, if the converse were true, then the step height would be determined by the first anodization voltage.

The structure resulting after step (3) is a passive dielectric optical waveguide capable of guiding light in the step region, and, in addition, capable of restricting oscillation to a single transverse mode for appropriate step widths and heights. This structure has been referred to as a rib-waveguide which may be used as a passive device or which may be incorporated into an active device such as a Miller laser. In order to complete the fabrication of such a laser, a layer of AlGaAs would be grown by LPE, for example, over the stepped GaAs layer, it being assumed that a first AlGaAs layer underlies the stepped GaAs layer. That is, the stepped GaAs is sandwiched between AlGaAs layers of opposite conductivity type whereas the stepped GaAs layer may be either n-type, p-type, unintentionally doped or compensated. Care should be exercised in the growth of the previously mentioned GaAs layer to alleviate the formation of deleterious oxides on the GaAs layer when exposed to air for the anodization procedure. In addition care should be exercised in the growth of the last AlGaAs layer to prevent the dissolution of the step configuration while in contact with the AlGaAs growth solution. Techniques are described hereinafter for alleviating both of these problems.

BRIEF DESCRIPTION OF THE DRAWING

Our invention, together with its various features and advantages, can be easily understood from the following more detailed description taken in conjunction with the accompanying drawing, in which:

FIG. 1, parts A-H, show schematically the fabrication of a Miller laser at various stages of the fabrication process in accordance with an illustrative embodiment of our invention; and

FIG. 2 is a schematic of apparatus for growing the layers of FIG. 1 by liquid phase epitaxy in accordance with another aspect of our invention.

DETAILED DESCRIPTION

Turning now to FIG. 1, parts A-H show sequential structural changes after each principal step in our inventive technique used to fabricate a Miller laser or a rib-waveguide. Of course, for simplicity and clarity of explanation FIG. 1 is not necessarily drawn to scale.

In FIG. 1, part A, an n-AlGaAs layer 12 has been epitaxially grown on an n-GaAs substrate 10. Illustratively, layer 12, as well as other GaAs or AlGaAs layers described hereinafter, is grown by a liquid phase epitaxy (LPE) technique of the type described by H. C. Casey,

Jr., et al., in *Journal of Applied Physics*, Vol. 45, page 322 (January, 1974). Thus, the next step in the procedure, as shown in FIG. 1, part B, is to grow by LPE a p-GaAs layer 14 on layer 12. Next, as shown in FIG. 1, part C, a native oxide layer 16 is formed on the GaAs layer 14.

We have found that oxide layer 16 is preferably formed by an anodization scheme of the type described by B. Schwartz in U.S. Pat. No. 3,798,139 supra. Briefly, in this technique the structure of FIG. 1, part B is placed in an electrolyte bath illustratively comprising concentrated H_2O_2 (30 percent) and H_2O (70 percent), a commercially available solution. This structure is made the anode whereas a noble metal such as platinum is made the cathode. The electrolyte bath is typically buffered with phosphoric acid to decrease the pH to a value of 2.0 and a source of about 100 V. d.c. is connected between the anode and cathode. After about 10 minutes a native oxide layer is grown having a thickness of about 1,100 Angstroms. Simultaneously, about 740 Angstroms of GaAs layer 14 is consumed and is incorporated into the grown oxide layer 16. Alternatively, the anodization could be performed using a constant current source instead of the constant voltage source, in which case the current is caused to flow through the oxide and sample (typically at about $10\text{ma}/\text{cm}^2$) until the desired voltage drop across the oxide is obtained, in this case 100 V. Note that the amount of oxide grown and GaAs consumed per volt may be a function of the GaAs growth procedure and/or the surface preparation. However, these parameters can be readily measured in advance for the particular GaAs material being anodized. For example, we have found that for ingot grown GaAs, the foregoing bath at 100 V. d.c. produces an oxide layer having a thickness of about 1,860 Angstroms and consumes about 1,250 Angstroms of GaAs.

In general, a suitable pH range is about 1-6. The relationship between oxide thickness and anodization voltage varies with the pH of the H_2O_2 bath. While buffering with phosphoric acid decreases the pH, buffering with aqueous ammonium hydroxide increases the pH. A suitable voltage range is about 5 to 225 V. As described hereinafter, however, the maximum voltage utilized in the growth of subsequent native oxide layers may be limited to about 150 V. by considerations related to surface roughness.

Next, the structure of FIG. 1, part C, is removed from the bath and air dried by heating, for example, at 110°C for $\frac{1}{2}$ hour.

After drying is completed, portions of the native oxide layer 16 are removed by standard photolithographic techniques in order to define an elongated oxide stripe 18 as shown in FIG. 1, part D. Briefly, a photoresist layer (not shown) of any suitable photoresist (e.g., AZ1350) is deposited on oxide layer 16 and, after masking to shade the desired stripe, is exposed to ultraviolet radiation. The exposed photoresist, and the oxide directly beneath, is then removed in a caustic developer leaving a photoresist layer 15 (shown in FIG. 1, part D in phantom) on oxide stripe 18. The remaining photoresist layer 15 may be removed by submersing in acetone thereby leaving only oxide stripe 18 on GaAs layer 14. As described hereinafter, this stripe will be utilized as a mask against subsequent oxidation in order to form a rectangular step in the GaAs layer 14.

In order now to fabricate a rib-waveguide or a Miller laser, it is necessary to remove a preselected amount of GaAs, typically 100 to 1,000 Angstroms, from the exposed portions 14a and 14b of layer 14. Adequate uniformity and control of this process is achieved by repeating the above-described anodization procedure in which utilization is made of the fact that LPE grown GaAs about 7.4 Angstroms of GaAs is consumed per volt in a bath having a pH = 2.0. Thus, as shown in FIG. 1, part E, to form a step having a height $\Delta h = 100$ Angstroms, the structure of FIG. 1, part D is anodized at about 13.5 V. Similarly, to form a step having a height $\Delta h = 500$ Angstroms, the structure is anodized at about 67.5 V. Because the second anodization voltage (i.e., that used to form the step 20 of FIG. 1, part E) is less than the first anodization voltage (i.e., that used to form the oxide stripe 18) the second anodization does not grow more oxide on the previously oxidized areas lying beneath stripe 18. However, if the converse is true, that is, if the second anodization voltage is higher than the first anodization voltage, then the exposed portions 14a and 14b of GaAs layer 14 (FIG. 1, part D) will be oxidized as well as the unexposed portion 14c of layer 14 lying beneath oxide stripe 18. However, the higher resistance associated with portion 14c (due to the resistance of stripe 18) results in less additional GaAs being consumed in unexposed portion 14c than in exposed portions 14a and 14b. In this case the step height Δh is determined by the first anodization voltage rather than the second anodization voltage.

In either case, the next step in the procedure is to remove all of the grown native oxide by immersing the structure of FIG. 1, part E in a suitable acid or base, for example HCl or NH_4OH . The resultant structure, shown in FIG. 1, part F, exhibits a GaAs layer 14 having a centrally located rectangular step 20. As mentioned previously, for use as a rib-waveguide or as a Miller laser, the step height Δh is typically in the range of 100-1,000 Angstroms for fundamental mode operation. Consequently, the above-described anodization bath would require anodization voltages in the range of about 13.5 V. to 135 V. For other applications, however, larger step heights might be desirable so that, without more, larger anodization voltages would be required. However, we have found that anodization voltages larger than about 150 V. produce a relatively rough surface on the top of the GaAs layer 14. Such roughness has made it difficult to reproducibly grow by LPE good quality AlGaAs on the top of layer 14. (Such layers are utilized to form the Miller laser as will be described hereinafter with reference to FIG. 1, parts G and H.) In addition, even if surface roughness were not a problem in a particular device application, the maximum anode-to-cathode voltage which can be used in the anodization apparatus is about 225 V. Thus, in a single anodization step at 225 V. the maximum step height Δh that can be produced on LPE grown GaAs is about 1,660 Angstroms.

To produce larger steps either of two procedures can be followed. First, it is possible to repeat the procedure beginning with the growth of another native oxide layer over the entire surface of the structure of FIG. 1, part F and subsequently performing the steps associated with FIG. 1, parts D and E. However, this procedure would require critical realignment of the photolithographic mask with the rectangular step 20. Alternatively, we have found that larger step heights can be

readily attained by a preferred technique in accordance with another embodiment of our invention. That is, the photoresist layer 15 of FIG. 1, part D can be specially treated and left on the structure during the second anodization. The native oxide formed on portions 14a and 14b by the second anodization step is dissolved in the caustic developer solution whereas the treated photoresist layer 15 is unaffected. This technique defines a composite mask as shown in FIG. 1, part D, which comprises the native oxide stripe 18 and the overlying treated photoresist layer 15. Consequently, the structure of FIG. 1, part D can be subjected to a plurality of sequential anodization and oxide-stripping steps while maintaining the integrity of the composite mask. In each step the native oxide is grown on the exposed portions of layer 14 and is then stripped away. This procedure is repeated a requisite number of times to achieve the desired step height.

More specifically, an illustrative set of steps in this procedure entails the structure of FIG. 1, part B, as previously described, to form native oxide layer 16. This structure is then air dried at about 110°C for 30 minutes. Photoresist AZ1350 is then applied and a stripe pattern is formed on the surface of the oxide in conventional fashion. Note that the conventional caustic developer solution dissolves the exposed photoresist and the exposed native oxide thereunder. The unexposed photoresist, layer 15, is essentially insoluble in the developer. Next, photoresist layer 15 is hardened by air drying at 110°C for about 30 minutes. This step forms the composite mask comprising native oxide stripe 18 and the hardened photoresist layer 15. Next, the sample may be reanodized to oxidize the exposed portions 14a and 14b of the GaAs layer 14b. The structure is then submersed in the developer solution or other suitable solvent to remove the grown oxide. The last two steps can now be repeated to remove as much GaAs as desired from the exposed portions 14a and 14b. When the desired step height is finally attained, the hardened photoresist layer 15 may be removed by submersing in acetone and the native oxide mask 18 may be removed as previously described.

Note that we have found that use of the photoresist directly on the GaAs layer 14 without the underlying native oxide strip 18 was unsuccessful because of undercutting of the photoresist in the anodization bath.

Returning now to FIG. 1, part F, there will now be described a procedure for growing an AlGaAs layer 22 over the stepped GaAs layer 14 in order to complete a Miller laser. In order to achieve good quality growth of AlGaAs layer 22, we have found that there should be as little oxide as possible on the surface of stepped GaAs layer 14. We have identified two sources of such oxide which can be independently removed as follows. First, in stripping the native oxide from the stepped GaAs layer 14 the structure is typically submersed in concentrated HCl or HF acid and subsequently rinsed in water. The water rinse causes an oxide to grow on the GaAs surface. However, if the water rinse is followed by a step in which the surface is exposed to a bromine methanol solution for about 1-3 minutes, the oxide is removed without any deleterious effects to the rib structure and the subsequent growth of a good quality AlGaAs layer 22 can be effected. A suitable range of bromine content in the solution is approximately 0.01 to 0.1 percent by volume. The bromine methanol etching is terminated by rinsing the sample in methanol,

and the sample is then inserted into the epitaxial layer growth apparatus. Secondly, during the growth of GaAs layer 14 some of the solution used to first grow AlGaAs layer 12 may be dragged over into the solution used to grow GaAs layer 14. The mixing of the two causes layer 14 to contain a small amount of Al. That is, layer 14 instead of being pure GaAs may be $\text{Al}_y\text{Ga}_{1-y}\text{As}$ with y between 0.01 and 0.02 approximately. Consequently, if layer 14 is exposed to air aluminum oxide will form thereon. Even though this oxide may be removed by the aforementioned bromine methanol etching, if the surface is again exposed to air the oxide will reform. We have found, however, that it is possible to obtain good quality AlGaAs layers 22 if the amount of solution drag-over is minimized to the extent that y is less than about 0.008. One approach to this problem is to perform the final bromine methanol etching prior to growth of AlGaAs layer 22 in a well-known oxygen-free glove-box. However, this procedure entails handling of the LPE apparatus (i.d., the solution holder and seed slider) in an oxygen-free atmosphere. Alternatively, we have found that use of the LPE apparatus shown in FIG. 2 enabled us to grow the GaAs layer 14 so that it was substantially free of Al. Consequently, either no oxidation of Al formed on the surface of layer 14 or the amount formed, if any, was insignificant. This procedure is as follows. The wells I, II and III of solution holder 29 are filled, respectively, with Ga solutions of AlGaAs with Sn or an n-dopant, GaAs with Ge or a p-dopant and GaAs with Ge or a p-dopant. After the n-AlGaAs layer 12 is grown on the main seed 34 of FIG. 2 (which corresponds to substrate 10 of FIG. 1, part A), the slider 31 of FIG. 2 is moved so that the main seed 34 is under well II which contains a p-GaAs solution. The growth rate is adjusted to about 0.05 μm per minute so that while the main seed 34 is under well II, only about 0.3 μm of GaAs is grown thereon. This step allows any solution dragged over from well I to be diluted. Thus, any subsequent drag-over will be diluted even more. Meanwhile the sacrificial seed 32 equilibrates the p-GaAs solution of well III. Finally, 0.4 μm GaAs (i.e., layer 14 of FIG. 1, part B) is grown on the already existing 0.3 μm GaAs layer. The 0.4 μm GaAs layer will be almost free of AlAs, typically $y \sim 0.003-0.005$.

In addition to the oxide problem, it is important to initiate the growth of AlGaAs layer 22 so that the integrity of the step 20 in GaAs layer 14 is maintained. If the solution used to grow the layer 22 dissolves a portion of the layer 14, the step which is typically only 1,000 Angstroms or less in height may be obliterated. This problem may be alleviated as follows. The wells are emptied and wells I and II are refilled with Ga solutions containing, respectively, AlGaAs with Ge or a p-dopant and GaAs with Ge or a p-dopant. The AlGaAs solution I is allowed to equilibrate for about 2 hours at 800°C. Then a fresh sacrificial GaAs seed 32 is slid under it and allowed to equilibrate for about 15-30 minutes. This step insures that the solution I is at equilibrium at the bottom where growth occurs. Then the solution and seeds (i.e., the oven, not shown) are cooled at a rate of about 0.25°C per minute until the temperature decreases by about 3-4°C before the main seed 34 is slid under the solution I. This temperature drop insures that growth has already started on the sacrificial seed 32 and will continue smoothly on to the main seed. We have found that this procedure provides sudden growth

of the p-AlGaAs layer 22 without rounding of the edges of the step 20 of GaAs layer 14.

After the p-AlGaAs layer 22 is grown by the aforementioned technique, a similar technique is used to grow the p-GaAs layer 24. The latter layer is utilized to facilitate forming electrical contacts to the Miller laser. By techniques well known in the art contact 26 is formed on the substrate 10 and a stripe geometry contact 28 is formed on the GaAs layer 24. Stripe contact 28 is substantially in registration with the rectangular step 20 in layer 14.

It is to be understood that the above-described arrangements are merely illustrative of the many possible specific embodiments which can be devised to represent application of the principles of the invention. Numerous and varied other arrangements can be devised in accordance with these principles by those skilled in the art without departing from the spirit and scope of the invention. In particular, although the foregoing technique was specifically directed to the fabrication of a rectangular step in a GaAs layer, it would be obvious to one skilled in the art that a substantially rectangular groove in a GaAs layer can readily be formed in substantially the same way. For example, in the photolithographic masking step, a photoresist mask, which is the negative of that utilized to form the step, could be utilized to form a groove. Alternatively, a photoresist mask defining a pair of parallel stripes could be utilized and a groove formed therebetween by anodization and oxide stripping. It should be noted, however, that the active region of the laser formed by a step functions in a way different from the active region of a laser formed by a groove.

Moreover, whereas the foregoing fabrication procedures employed a stripe mask, it would be obvious to one skilled in the art that the mask could have any planar shape (in top view), but the cross-section at any particular point will still be a substantially rectangular step.

What is claimed is:

1. A method of fabricating a substantially rectangular step in a preselected region of a GaAs layer comprising the steps of:

- a. growing a native oxide layer on a major surface of said GaAs layer by submersing said GaAs layer in an anodization bath comprising H_2O_2 having a pH less than about 6;
- b. removing selected portions of said oxide layer so as to expose said GaAs layer adjacent said preselected region, a native oxide mask remaining over said region; and
- c. immersing said GaAs layer in an anodization bath comprising H_2O_2 having a pH less than about 6 so that the exposed portions of said GaAs layer are consumed in the formation of a native oxide layer thereon, thereby to form said step in said GaAs layer.

2. The method of claim 1 wherein a first anodization voltage is applied in step (c) which is less than or equal to a second anodization voltage applied in step (a), so that the height of said step is proportional to said second anodization voltage.

3. The method of claim 1 wherein a second anodization voltage is applied in step (c) which is greater than a first anodization voltage applied in step (a), so that during step (c) GaAs is consumed not only in the exposed portions of said GaAs layer but also beneath said

oxide mask, the height of said step being proportional to said first anodization voltage.

4. The method of claim 1 wherein the anodization baths of steps (a) and (c) each have a pH of about 2.

5. The method of claim 4 wherein said GaAs layer is grown by liquid phase epitaxy and wherein during said anodization steps (a) and (c) about 11 Angstroms of native oxide is grown and about 7 Angstroms of GaAs is consumed per volt.

6. The method of claim 4 wherein said GaAs layer is ingot grown and wherein during said anodization steps (a) and (c) about 18 Angstroms of native oxide is grown and about 12 Angstroms of said GaAs is consumed per volt.

7. The method of claim 1 where step (b) includes the additional steps of: applying a layer of photoresist over said oxide layer, irradiating with ultraviolet radiation the portions of said photoresist layer adjacent the desired oxide mask; submersing said photoresist layer in a caustic developer solution so as to dissolve the irradiated portions thereof and, in addition, to dissolve the native oxide layers under said irradiated portions, the unirradiated portion of said photoresist layer over said oxide mask remaining thereon, heating said remaining photoresist layer at an elevated temperature for a time period effective to harden said photoresist layer and to render it more chemically inert, said hardened photoresist layer and the oxide mask thereunder forming a composite mask for subsequent processing steps, performing anodization step (c) to grow a native oxide layer on the exposed portions of said GaAs layer and to consume GaAs in said portions so as to form said step, submersing said last grown native oxide layer in a solvent which removes said oxide layer but not said composite mask, and repeating the last mentioned two steps so as to increase the height of said step without the need for remasking said step.

8. The method of claim 1 wherein anodization voltages are applied in steps (a) and (c) which are less than approximately 150 volts so as to maintain within tolerable limits the roughness produced in the surface of said GaAs layer during the growth of the native oxide layers.

9. The method of claim 1 wherein all native oxide layers grown on said GaAs layer are removed.

10. The method of claim 9 including the additional step of growing by liquid phase epitaxy of AlGaAs on said GaAs layer.

11. The method of claim 10 including prior to the growth of said AlGaAs layer the additional step of submersing said GaAs layer in a solution of bromine methanol containing bromine in the approximate range of 0.01 to 0.1 percent.

12. The method of claim 10 wherein the solution used to grow said AlGaAs layer is allowed to equilibrate at about 800°C for several hours, bringing said solution into contact with a sacrificial GaAs seed to allow said solution to equilibrate for a time in the range of approximately 15 to 30 minutes, cooling said solution at a relatively slow rate to reduce its temperature by several degrees and then bringing said solution into contact with said GaAs layer.

13. The method of claim 10 including the steps of growing on a GaAs substrate a first AlGaAs layer by liquid phase epitaxy, growing said GaAs layer on said first AlGaAs layer by liquid phase epitaxy, controlling the amount of solution dragged over from the solution

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used to grow said first AlGaAs layer to the solution used to grow said GaAs layer so that the mole fraction of AlAs in said GaAs layer is less than approximately 0.008.

14. The method of claim 13 performed in conjunction with apparatus including a solution holder having at least three wells, a slider having at least two recesses, one for carrying said substrate and one for carrying a sacrificial GaAs seed, placing Ga solutions in each well, a first one of which contains AlGaAs, and the second and third of which contain GaAs, instituting a controlled cooling program, bringing said first solution into contact with said substrate to grow said first AlGaAs layer thereon, bringing said substrate into contact with

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said second solution of GaAs in order to dilute any solution dragged over from the well containing AlGaAs and concurrently bringing the third solution of GaAs into contact with said sacrificial seed so as to equilibrate said third solution, and bringing said substrate into contact with said third solution, thereby to grow a GaAs layer having a mole fraction of AlAs which is less than 0.008.

15. The method of claim 14 wherein the temperature of said second solution is decreasing at a rate of about 0.05°C per minute while it is in contact with said substrate.

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