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61/559,430 14 November 2011 (14.11.2011) US(71) Applicant (for all designated States except US): **THE GOVERNMENT OF THE UNITED STATES OF AMERICA, AS REPRESENTED BY THE SECRETARY OF THE NAVY** [US/US]; Naval Research Laboratory, 4555 Overlook Ave, SW, Code 1008.2, Washington, DC 20375 (US).

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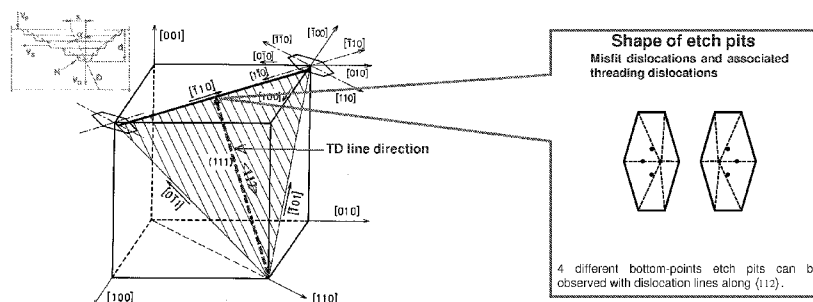
AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

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**Published:**

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- before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))

(54) Title: ETCHING AGENT FOR TYPE II INAS/GALNSB SUPERLATTICE EPITAXIAL MATERIALS



(57) Abstract: This disclosure involves a formula, mixing procedure, etching technique and application of an etchant for revealing defects in T2SL's grown lattice matched to (100) GaSb. The etching agent comprises a (2.5:4.5:16.5:280) solution by volume or (1%:2%:9%:88%) by weight, of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O. The etchant is made by mixing (49%) hydrofluoric aqueous solution with (30%) water-based peroxide, followed by sulfuric acid, and diluted with de-ionized H<sub>2</sub>O (DI-water).

## TITLE OF THE INVENTION

Etching Agent for Type II InAs/GaInSb Superlattice Epitaxial Materials

## 5 CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a non-provisional application of and claims priority to and benefits of United States Patent Application Number 61/559,430 filed 11/14/2011.

## BACKGROUND OF THE INVENTION

## 10 (1) Technical Field of the Invention

The disclosure involves a formula, mixing procedure, etching technique and application of an etchant for revealing defects in a Type II superlattice matched to (100) GaSb.

## (2) Description of Related Art

15 One aspect of this disclosure involves a method for revealing crystallographic defects on the (100) face of Type II InAs/GaInSb based superlattice structures by preferential wet chemical etching.

Type II InAs/Ga<sub>1-x</sub>In<sub>x</sub>Sb superlattices (T2SLs) lattice matched to GaSb substrates (x typically ≤ 20%) are an important semiconductor heterostructure material systems for applications in  
20 mid-, long- and very long-wave infrared detectors. They have theoretical performance limits well beyond those of incumbent technologies based on InSb and HgCdTe.

T2SLs were first introduced in the 1970s by Sai-Halasz, Tsu and Esaki, and then a decade later, were proposed by Smith and Mailhiot to be used for infrared detection. Presently, state-of-the-art T2SL material is grown using molecular beam epitaxy (MBE) on (100) GaSb  
25 substrates.

The period structure of the basic binary form of T2SL consists of a pair of 1 to 5 nm-thick InAs and GaSb layers. Approximately one additional monolayer (ML) of InSb ( $a = 6.4794\text{\AA}$ ) is also required in each period to balance the strain with respect to the GaSb substrate ( $a = 6.0959\text{\AA}$ ) resulting from the shorter lattice constant of InAs ( $a = 6.0583\text{\AA}$ ).

30 The ternary T2SL is an alternate form in which GaSb is replaced by Ga(1-x)In(x)Sb in the superlattice, and strain is balanced by adjusting the InSb-alloy fraction “x” in the ternary

layer and dispensing with the half-ML of InSb at each interface. These and several other types of T2SLs may be readily doped *n*- or *p*-type, and have been used to construct an enormous variety of infrared sensor structures, with epitaxial thicknesses of up to ~15  $\mu\text{m}$ 's.

Despite more than 30 years of study, however, T2SL technology has not yet achieved its full theoretical promise, largely due to the presence of bulk and surface crystallographic defects generated during MBE growth and device fabrication that promote excess dark current.

Progress in understanding the nature of these defects and how to remediate them has been hindered by the difficulty in identifying specific defect structures in this material system.

One widely used technique that has been used successfully to identify defects with densities of up to  $\sim 10^5 \text{ cm}^{-2}$  on substrates and epitaxial material, is preferential chemical etching. Here, defect structures are identified by subjecting the material to a wet chemical etch that has the characteristic of being amplified in the presence of crystallographic defects, and then analyzing the density, location and physical structure of resulting etch pits.

Though a number of techniques exist to preferentially etch GaSb and InAs on different crystallographic planes, no such techniques have heretofore existed for superlattices composed of periodic combinations of thin layers of these materials. This is due to the fact that though InAs and GaInSb belong to the same  $-43m$  point symmetry group, the difference in chemical composition yields a large contrast in preferential etch rates of these materials.

For etch pit defect delineation in (100) GaSb for example, a two-step etch process was used by J. Doershel, and U. Geissler where the surface was first chemically polished in a 2:18:40 (volume ratio) solution of  $\text{HF}:\text{HNO}_3:\text{CH}_3\text{COOH}$ , followed by etching in 2:1 solution of  $\text{HCl}:\text{H}_2\text{O}_2$ . Constant vigorous agitation was necessary to obtain reliable results.

Costa et al. used a (5:1) solution of  $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$  as well as (5:1)  $\text{CrO}_3$  (5 M aqua. solution.): $\text{HF}$  for etch pit delineation on GaSb (100). For preferential etching of InAs (111) orientation, Yonenaga employed a  $2.4 \times 10^{-3}$  molar solution of  $\text{AgNO}_3$  in (3:2:5)  $\text{HNO}_3:\text{HF}:\text{H}_2\text{O}$ .

We have found no previously published data, however, on preferential etching of InAs/GaInSb superlattices, indicating the lack of a common etching solution with similar preferential etch rates for both compound materials.

Thus we submit this disclosure of a single etchant which reveals and distinguishes crystallographic defects on (100) T2SLs through etch pit structures defined by preferential wet chemical etching.

## 5 DISCLOSURE OF THE INVENTION

This disclosure involves a formula, mixing procedure, etching technique and application of an etchant for revealing defects in T2SL's grown lattice matched to (100) GaSb.

## BRIEF DESCRIPTION OF THE DRAWINGS

10 Figure 1 illustrates GaSb, InAs (F-43m space group), Zinc-Blende Lattice.

Figure 2 illustrates etch pits from inclusion and etch pits from dislocations in the film.

Figure 3 illustrates typical etch pits in type II TSL.

Figure 4 illustrates chemical etching results of raw wafers. Rectangular etch pits with well-defined crystallographic facets form in locations containing dislocations in the  
15 crystal structure.

Figure 5 illustrates correlation between electrically active defects, leakage current and etch pits. Shown are electrically active defects (dark spots) presented in devices with increased leakage current. Also shown is one to one correlation between electrically active defects mapped by EBIC and etch pits.

20

## BEST MODES FOR CARRYING OUT THE INVENTION

This disclosure involves a formula, mixing procedure, etching technique and application of an etchant for revealing defects in T2SL's grown lattice matched to (100) GaSb.

The etching agent comprises a (2.5:4.5:16.5:280) solution by volume or (1%:2%:9%:88%) by  
25 weight, of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O.

The etchant is made by mixing (49%) hydrofluoric aqueous solution with (30%) water-based peroxide, followed by sulfuric acid, and diluted with de-ionized H<sub>2</sub>O (DI-water).

Etching can be carried out at room temperature (around 20° C), without stirring.

Typical etching times for the (100) face of a T2SL are in the range of 60-90 seconds.

After etching, the samples are rinsed thoroughly in DI-water and then dried using N<sub>2</sub> gas.

Under these conditions, using a freshly prepared solution, rectangular etch pits with well-defined crystallographic facets form in locations containing dislocations in the crystal structure. The resulting etch pits are oriented with the longest axis aligned along the  $[0\bar{1}\bar{1}]$  crystallographic direction.

The morphology of the etch pits and surface may be varied by adjusting the ratios of the etching solution from the mixture given above. For example, increasing the HF or H<sub>2</sub>O<sub>2</sub> content leads to a more isotropic etch, with rounded etch pits. Increasing H<sub>2</sub>SO<sub>4</sub> or decreasing HF content introduces more anisotropic etching of the surface. In addition, increasing H<sub>2</sub>SO<sub>4</sub> or decreasing HF introduces nonuniform etching of the surface, making reliable identification of defects difficult.

Etching time is controlled by dilution with water, and for a typical long-wave infrared T2SL etched in the above solution with 88% DI-water by weight, etch pits ranging from 4-20 μm in diameter are formed after 60-90 seconds. Etch pits of this size are readily observed using optical differential interference contrast (Nomarski) microscopy.

The application of this novel etchant on T2SLs enables the detection and identification of various imperfections in the crystalline structure of the material, as etch pit formation is promoted and governed by the structure of the underlying defect.

For example, threading dislocations are indicated by large, deep rectangular pits, having bottom point symmetries reflecting dislocation direction. Misfit dislocations (half-loop arrays) are marked by twin shallow rectangular etch pits indicating the termination points of the half loops at the surface. More rounded, flat bottomed pits correspond to agglomerations of impurities.

In addition, the etch produces linear striations aligned along  $[01\bar{1}]$  and  $[\bar{1}10]$  directions in the presence of stacking faults in  $(01\bar{1})$  and  $(\bar{1}10)$  glide planes.

The correlation between etch-pit structure, the structure of the underlying crystallographic defect, and the activity level of the defect with respect to dark current, were studied using electron beam induced current (EBIC) mapping of defects and cross-sectional transmission electron microscopy (TEM) to reveal the defect structure.

5 TEM and EBIC confirmed strong correspondence between etch pit and crystallographic defect structure. We found no other published data showing successful preferential etching of InAs/GaInSb superlattices, though a mixture of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O has been reported for preferential etching of dislocations on GaAs<sub>x</sub>P<sub>1-x</sub> semiconductors where x is in the range of 0 to 1 and consisted of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O (6-18%:6-20%:20-55%:30-55%) by weight.

10 We tested several etchants suggested for preferential etching of GaSb and InAs, including a modified two-step etch process of Doershel et al. consisting of an initial polish in HF:HNO<sub>3</sub>:CH<sub>3</sub>COOH (2:18:40) followed by etch-pit delineation in HCl: H<sub>2</sub>O<sub>2</sub> (2:1).

Upon testing, it was found that this procedure generally produced a highly non-uniform surface against which etched features were difficult to discern.

15 The best performance was achieved by diluting the polishing etch as HF:HNO<sub>3</sub>:CH<sub>3</sub>COOH:H<sub>2</sub>O (2:18:40:30) by volume, and eliminating the second etch step HCl: H<sub>2</sub>O<sub>2</sub> (2:1) entirely.

While dilution lowered the etch rate improving control, etch pit definition was marginal however as the increased water content also heavily degraded the shape of the etch pits as the  
20 etch became more isotropic. As a result, dilution was limited to a level with etching times of only 10-20 seconds, making control of the etching process difficult. Constant vigorous agitation of the etch bath was also necessary to remove gaseous products nucleated on the sample surface.

As such, our discovery overcame such problems and produced the best results. Our formula,  
25 mixing procedure, etching technique and application of an etchant for revealing defects in T2SL's grown lattice matched to (100) GaSb are herein disclosed. The etching agent can comprise a (2.5:4.5:16.5:280) solution by volume or (1%:2%:9%:88%) by weight, of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O and, as discussed, variations are possible.

The above description is that of a preferred embodiment of the invention. Various modifications and variations are possible in light of the above teachings. It is therefore to be understood that, within the scope of the appended claims, the invention may be practiced otherwise than as specifically described. Any reference to claim elements in the singular, e.g., using the articles “a,” “an,” “the,” or “said” is not construed as limiting the element to the singular.

## Claims

What we claim is:

1. An etchant comprising:

a (2.5:4.5:16.5:280) solution by volume of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O.

5

2. A solution for etching type II InAs/GaInSb superlattice epitaxial materials comprising:

a (1%:2%:9%:88%) solution by weight HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O.

3. A method of making a solution for etching type II InAs/GaInSb superlattice epitaxial materials comprising:

10

mixing a hydrofluoric aqueous solution with a water-based peroxide to form a first solution;

mixing the first solution with sulfuric acid to form a second solution; and

diluting the second solution with de-ionized water.

15

4. The method of making of claim 3 wherein the hydrofluoric aqueous solution is a 49% by weight hydrofluoric aqueous solution and wherein the water-based peroxide is a 30% by weight water-based peroxide.

5. A method of etching type II InAs/GaInSb superlattice epitaxial materials comprising:

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forming an etchant comprising a (2.5:4.5:16.5:280) solution by volume of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O;

applying the etchant to a type II InAs/GaInSb superlattice epitaxial material;

conducting the step of applying the etchant to a type II InAs/GaInSb superlattice epitaxial material at a temperature of about 20 degrees C; and

controlling the etching time by diluting with water.

- 5     6. The method of etching type II InAs/GaInSb superlattice epitaxial materials of claim 5 wherein the etching is conducted for about 60 to about 90 seconds and results in etch pits of about 4 to about 20  $\mu\text{m}$  in diameter.
7. The method of etching type II InAs/GaInSb superlattice epitaxial materials of claim 6  
10 wherein the etching does not involve stirring.
8. The method of etching type II InAs/GaInSb superlattice epitaxial materials of claim 7 wherein there is no surface treatment before etching.
- 15   9. The method of etching type II InAs/GaInSb superlattice epitaxial materials of claim 6 further including
- rinsing in deionized water; and
- drying using nitrogen gas.
- 20   10. The method of etching type II InAs/GaInSb superlattice epitaxial materials of claim 6 further including forming rectangular etch pits with crystallographic facets in locations containing dislocations in the crystal structure.

11. The method of etching type II InAs/GaInSb superlattice epitaxial materials of claim 10 wherein the resulting etch pits are oriented with the longest axis aligned along the  $[0\bar{1}\bar{1}]$  crystallographic direction.

5 12. The method of etching type II InAs/GaInSb superlattice epitaxial materials of claim 6 further including forming increasing the HF or H<sub>2</sub>O<sub>2</sub> and producing an isotropic etch with rounded etch pits with crystallographic facets in locations containing dislocations in the crystal structure.

10 13. The method of etching type II InAs/GaInSb superlattice epitaxial materials of claim 6 further including

forming etch pits with crystallographic facets in locations containing dislocations in the crystal structure; and

correlating the shape of the etch pit to the type of dislocation or impurity.

15

14. The method of etching type II InAs/GaInSb superlattice epitaxial materials of claim 13 wherein a rectangular pit having bottom point symmetries reflecting dislocation direction correlates to threading dislocations and wherein twin rectangular etch pits are correlated to misfit dislocations with half loop termination points.

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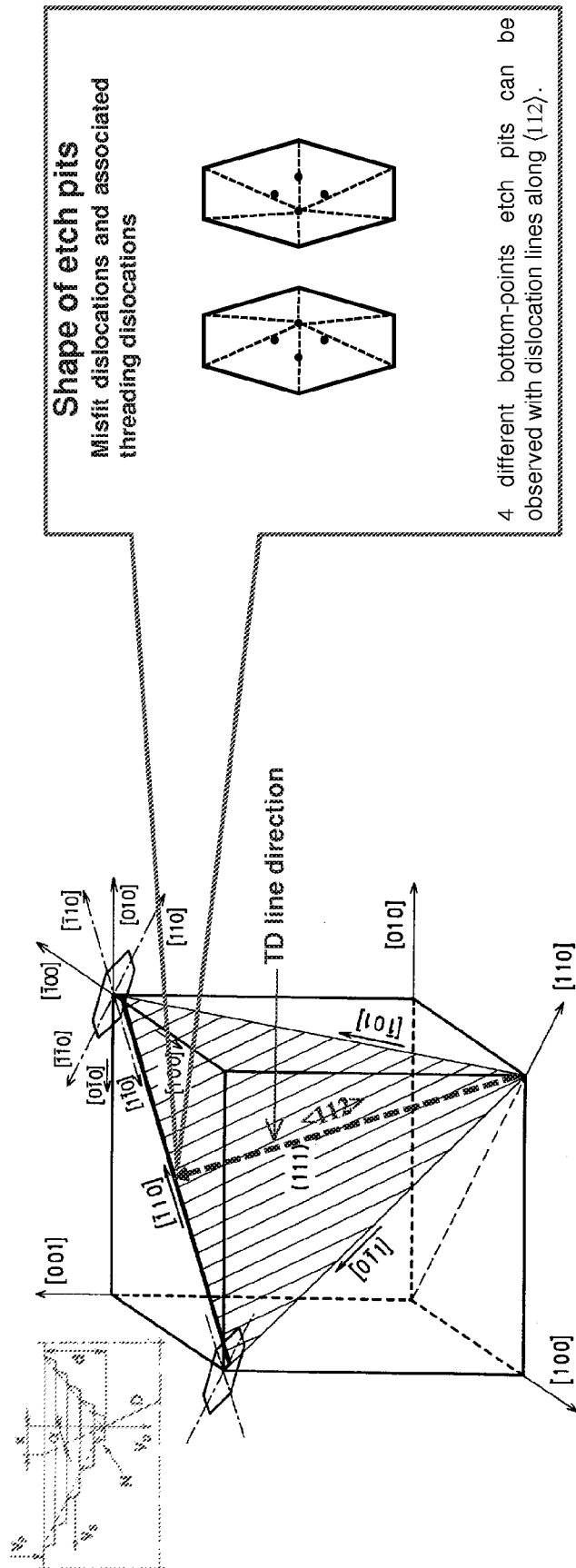
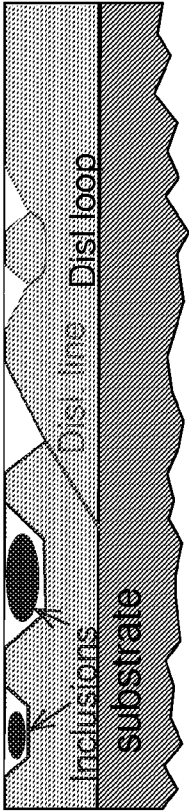


FIGURE 2



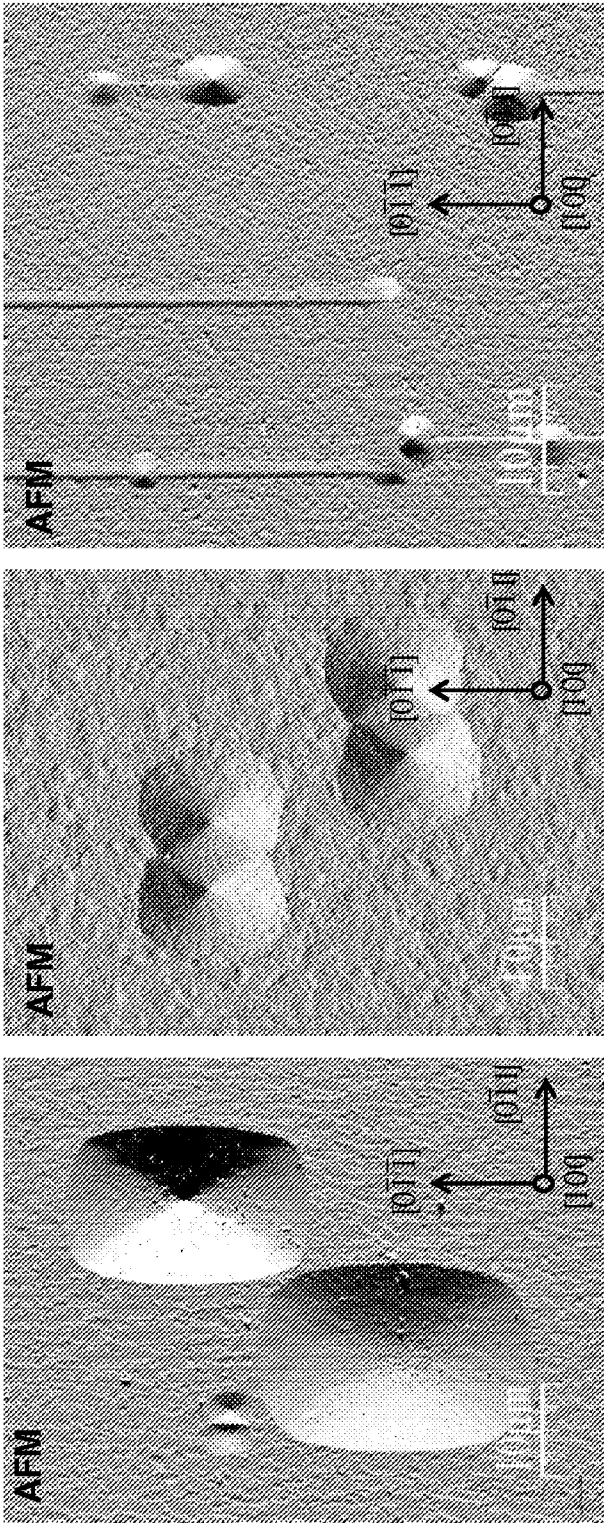


FIGURE 3

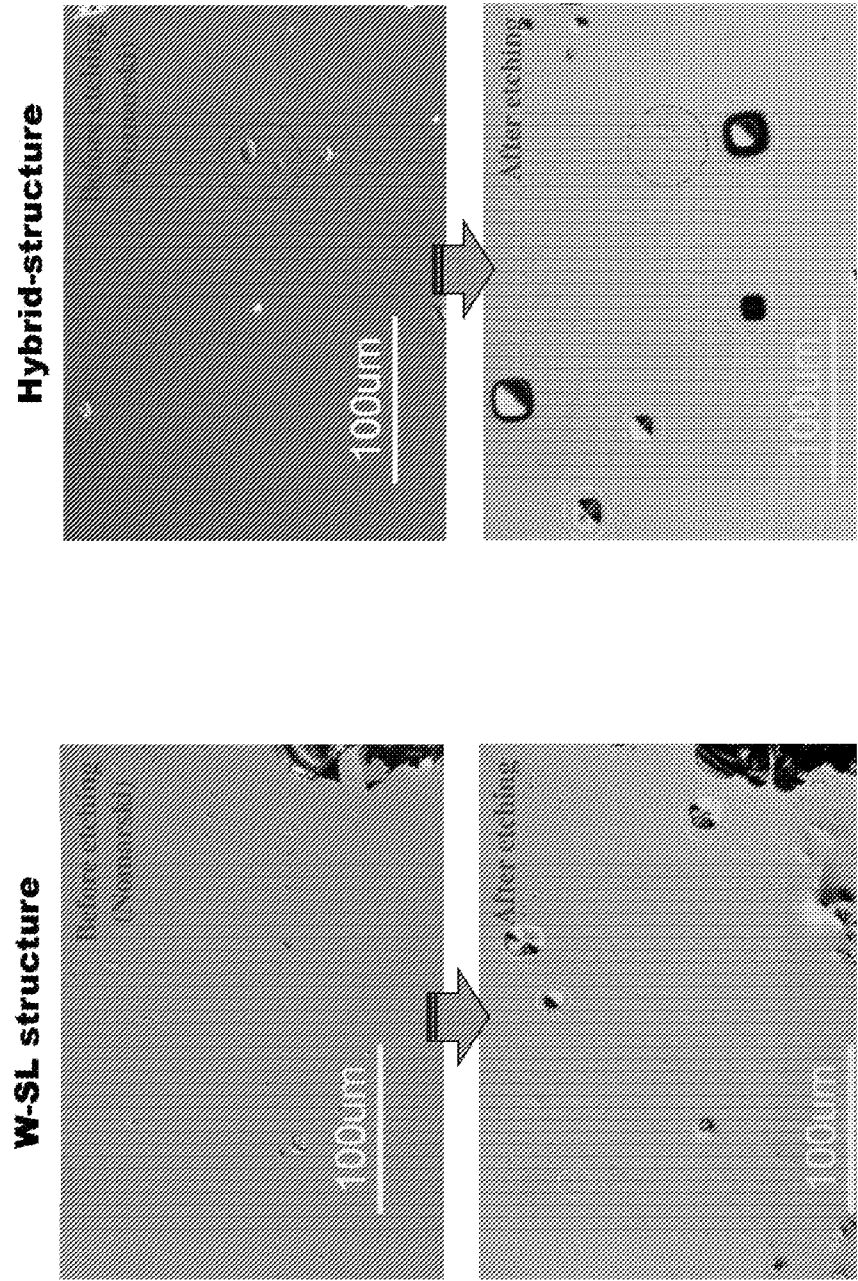
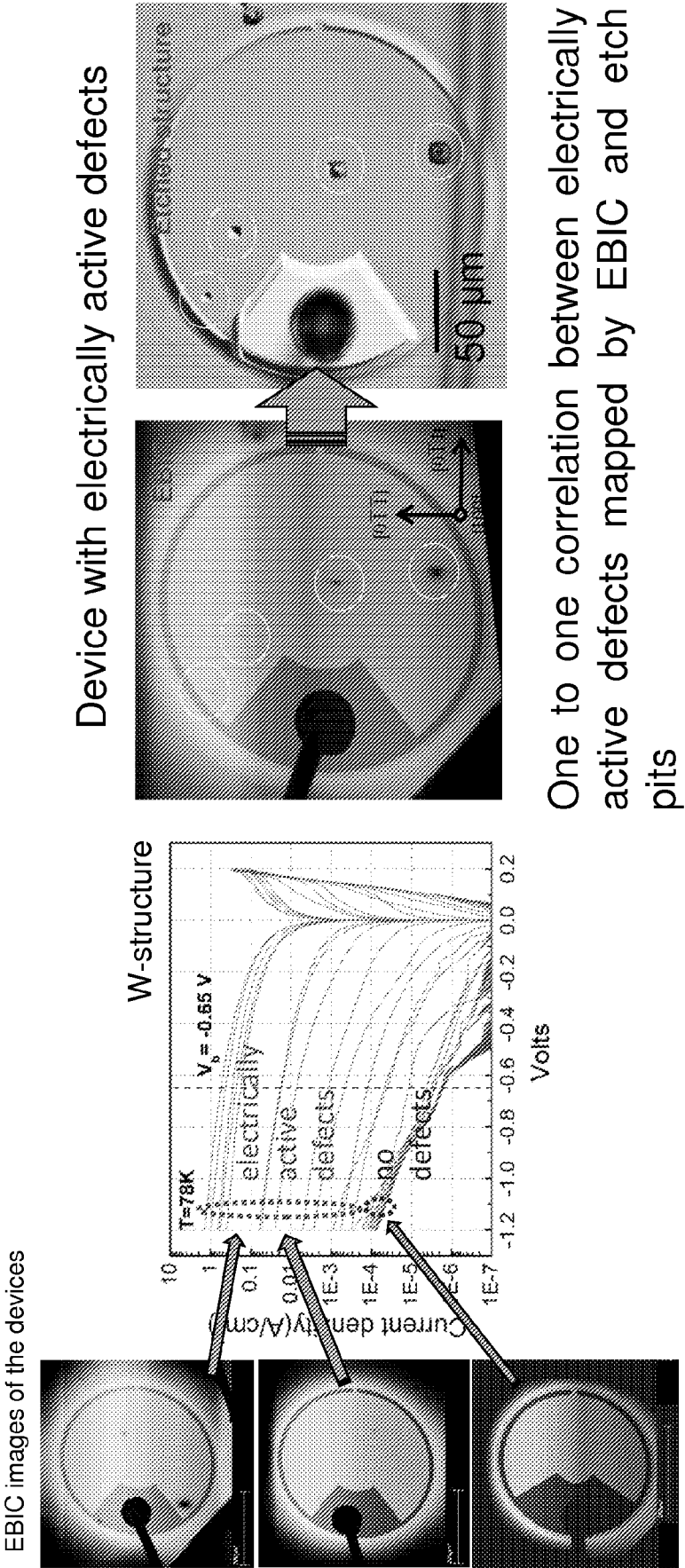


Figure 4

FIGURE 5



## INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 12/63700

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - C03C 25/68 (2012.01)

USPC - 216/108; 216/109

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

USPC - 216/108; 216/109

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched  
USPC - all classes; NPL (key word limited)

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Google Scholar/Patents; PatBase

Search terms - hydrofluoric w2 acid, HF, "hydrogen peroxide", "sulfuric acid", "InAs/GaInSb", peroxide, type II, T2SL, etching, hydrofluoric, Maximenko, Aifer

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                 | Relevant to claim No. |
|-----------|--------------------------------------------------------------------------------------------------------------------|-----------------------|
| X         | US 4,459,216 A (Nakazato et al.) 10 July 1984 (10.07.1984); entire document, especially cols 3 and 4               | 1, 2                  |
| Y         | US 2006/0086372 A1 (Mellies et al.) 27 April 2006 (27.04.2006); entire document, especially para [0019] and [0023] | 1, 2                  |

☐ Further documents are listed in the continuation of Box C.


\* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&amp;" document member of the same patent family

Date of the actual completion of the international search

25 February 2013 (25.02.2013)

Date of mailing of the international search report

25 MAR 2013

Name and mailing address of the ISA/US

Mail Stop PCT, Attn: ISA/US, Commissioner for Patents  
P.O. Box 1450, Alexandria, Virginia 22313-1450

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# INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 12/63700

## Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:  
because they relate to subject matter not required to be searched by this Authority, namely:
  
2. ☐ Claims Nos.:  
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
  
3. ☐ Claims Nos.:  
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

## Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

This application contains the following inventions or groups of inventions which are not so linked as to form a single general inventive concept under PCT 13.1. In order for all inventions to be examined, the appropriate additional examination fees must be paid.

Group I: Claims 1 and 2, directed to an etchant and a solution for etching type II InAs/GaInSb superlattice materials comprising a Claim 1: a (2.5:4.5: 16.5:280) solution by volume of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O.  
Claim 2: a (1%:2%:9%:88%) solution by weight HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O.

Group II: Claims 3 and 4, directed to a method of making a solution comprising the steps of  
-mixing a hydrofluoric aqueous solution with a water-based peroxide to form a first solution;  
-mixing the first solution with sulfuric acid to form a second solution; and  
-diluting the second solution with de-ionized water.

-----See Supplemental Sheet Below-----

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☒ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:  
1 and 2

### Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- ☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- ☐ No protest accompanied the payment of additional search fees.

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 12/63700

## Box No III Observations where unity of invention is lacking

Group III: Claims 5-14, directed to a method of etching type II InAs/GaInSb superlattice epitaxial materials comprising:

- forming an etchant comprising a (2.5:4.5:16.5:280) solution by volume of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O;
- applying the etchant to a type II InAs/GaInSb superlattice epitaxial material;
- conducting the step of applying the etchant to a type II InAs/GaInSb superlattice epitaxial material at a temperature of about 20 degrees C; and
- controlling the etching time by diluting with water.

The inventions listed as Groups I-III do not relate to a single general inventive concept under PCT Rule 13.1 because, under PCT Rule 13.2, they lack the same or corresponding special technical features for the following reasons:

Groups II and III do not require a solution and method of making a solution for etching having a (1%:2%:9%:88%) solution by weight HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O, as required by Group I.

Groups I and III do not require the steps of

- mixing a hydrofluoric aqueous solution with a water-based peroxide to form a first solution;
- mixing the first solution with sulfuric acid to form a second solution; and
- diluting the second solution with de-ionized water, as is required by Group II.

Groups I and II do not require a method of etching comprising the steps of forming an etchant, applying the etchant, conducting the application of etchant at 20 degrees C, and controlling the etching time by diluting with water, as required by Group III.

Groups I and III share the technical features of an etchant with a 2.5:4.5:16.5:280 solution by volume of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O.

However, etching solutions of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O having similar ratios were also known in the art, as evidenced by US 4,459,216 A to Nakazato et al. (col. 4, ln. 38-40). More specifically, correcting for the molar weights of each component, Nakazato teaches 10 g/liter HF; or (10 g/liter HF) x (1 mole HF/20g) = 0.5 mol HF/liter  
30 g/liter H<sub>2</sub>O<sub>2</sub>; or (30 g/liter H<sub>2</sub>O<sub>2</sub>) x (1 mole H<sub>2</sub>O<sub>2</sub>/34g) = 0.88 mol H<sub>2</sub>O<sub>2</sub>/liter  
150 g/liter H<sub>2</sub>SO<sub>4</sub>; or (150 g/liter H<sub>2</sub>SO<sub>4</sub>) x (1 mole H<sub>2</sub>SO<sub>4</sub>/100g) = 1.5 mol H<sub>2</sub>SO<sub>4</sub>/liter  
1 liter H<sub>2</sub>O; or (1000 g H<sub>2</sub>O/1 liter H<sub>2</sub>O) x (1 mole H<sub>2</sub>O/18g) = 55.55 mol/liter

Assuming a mole of each compound occupies the same volume and scaling for comparison to the Applicant's claims:

5 x 0.5 mol HF/liter = 2.5 mol HF/liter = 2.5 unit volumes HF/liter;  
5 x 0.88 mol H<sub>2</sub>O<sub>2</sub>/liter = 4.4 mol H<sub>2</sub>O<sub>2</sub>/liter = 4.4 unit volumes H<sub>2</sub>O<sub>2</sub>/liter;  
5 x 1.5 mol H<sub>2</sub>SO<sub>4</sub>/liter = 7.5 mol H<sub>2</sub>SO<sub>4</sub>/liter = 7.5 unit volumes H<sub>2</sub>SO<sub>4</sub>/liter;  
5 x 55.55 mol/liter H<sub>2</sub>O = 275 mol H<sub>2</sub>O/liter = 275 unit volumes H<sub>2</sub>O/liter.

Thus, Nakazato teaches a ratio of 2.5 : 4.4 : 7.5 : 275 solution by volume of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O, while the Applicant claims a ratio of 2.5 : 4.5 : 16.5 : 280 solution by volume of HF:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O. Further, since it would have been obvious to one of skill in the art that the increase of sulfuric acid would increase the etch rate, it would have required only routine experimentation for one of skill in the art to optimize the sulfuric acid ratio in order to achieve a desired etching rate.

Groups I - III share the technical features of a type II InAs/GaInSb superlattice epitaxial material. However, InAs/GaInSb superlattice epitaxial materials were known in the art, as evidenced by US 2010/0097690 A1 to Vurgaftman et al. (22 April 2010) (para [0007], discussing InAs/GaInSb superlattice epitaxial material), as well as WO 2004/059038 A1 to Bugge et al. (15 July 2004) (see p. 5, ln 25-30, where Al.sub.1-x-z.Ga.sub.x.In.sub.z.As.sub.1-y.Sb.sub.y, and 1-x-z equals zero; p. 6, ln. 5; 'InAs, GaInSb,' p. 8, ln 11). As these features were known at the time, as evidenced by the teaching of Vurgaftman, Bugge, and Mellies, they cannot be considered a special technical feature that would otherwise unify the groups.

Groups I - III therefore lack unity under PCT Rule 13 because they do not share a same or corresponding special technical feature.