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High Resolution Electrolyte for Thinning InP by Anodic Dissolution and its Applications to EC-V Profiling, Defect Revealing and Surface Passivation

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ABSTRACT

An extensive experimental study was conducted using various electrolytes based on HCl, o-H_3PO_4, H_2SO_4, HIO_3, HNO_3, HF, CH_3COOH, H_2O_2 and NH_4OH in an effort to find an appropriate electrolyte for anodic dissolution of InP. From the analysis of electrochemical C-V, $1/C^2$-V, G-V, I-V characteristics in the dark and under different illumination levels, X-ray Photoelectron Spectroscopy and SEM/Nomarski inspection of the surfaces, we have determined that the anodic dissolution of InP front surface layers by FAP electrolyte is a very good choice for rendering smooth surfaces, free of oxide and contaminants and with good electrical characteristics.

After experimentation with a fairly large number of p, n, p^+, n^+ Czochralski LEC/VGF grown InP substrates and n^+p and p^+n InP structures made either by thermal diffusion or epitaxy, the FAP electrolyte based on HF, CH_3COOH and H_2O_2 appears to be inherently superior to previously reported electrolytes (i.e., 0.5M HCl and the pear etch) for performing accurate EC-V profiling of InP at current densities of up to 0.3 mA/cm^2. It can also be used for accurate electrochemical revealing of either precipitates or dislocation density with applications to EPD mapping as a function of depth, and for defect revealing of multilayer InP structures at any depth and/or at the interfaces. Owing to its inherent qualities, the FAP electrolyte can also be used, with good results, for surface passivation of InP prior to deposition of an insulator or antireflective coating.

INTRODUCTION

One of the major problems yet to be solved in InP device technology is to remove, in a controlled manner, the contaminated and/or damaged surface layers due to processing steps such as masking, doping and annealing, in such a way that smooth surfaces with good electrical characteristics are obtained.

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For most semiconductors, including InP, simple chemical etching does not yield smooth surfaces with good electrical characteristics. Thinning the damaged surface layers of InP by plasma etching so as to obtain smooth surfaces is also not easily achieved due to preferential sputtering at the defect areas.

Anodic dissolution [1] can be successfully used for controlled thinning of surface layers, providing that a proper electrolyte and processing parameters are selected. For InP, none of the previously reported electrolytes such as 0.5M HCl [2] and the pear etch [3] seem to satisfy the criteria of an ideal electrolyte. They either dissolve InP preferentially at the defect areas, and/or form insoluble products on the surface. This makes the thinning process dependent on the surface chemistry and, therefore hard to control.

Thinning InP surfaces by anodic dissolution occurs via an anodic oxidation-dissolution process. The choice of the electrolyte is therefore crucial if smooth surfaces, free of contaminants and oxide, and with good electrical characteristics are desired. The electrolyte should contain both an oxidizing and a reducing component, chosen such that both elemental species of InP are nearly equal during dissolution. In other words, it should not promote preferential oxidation at the dislocation areas [4] or form stable oxide species on the surface [1].

From a fairly large number of new electrolytes we have tried in this study we have selected the so called "FAP" electrolyte [5], which proved to be inherently superior to previously reported electrolytes know to us, for thinning the heavily damaged n^+ and p^+ front layers of thermally diffused n^+p and p^+n InP structures [6]. In this study we investigated the use of this new electrolyte for other applications where smooth surfaces are required (e.g. electrochemical C-V (EC-V) profiling and surface passivation of InP), as well as for exposing the structural defects (precipitates and etch pit densities) at a given depth of front n^+- or p^+-InP layers.

Other studies using the FAP electrolyte, presently still under investigation include:

1. Photocapacitance spectroscopy.
2. Determination of surface state density of InP.
3. The use of this and/or related electrolytes for material characterization and/or processing of other III-V compounds.

EXPERIMENTAL

We have conducted an extensive experimental study using various electrolytic solutions based on HCl, o-H_3PO_4, H_2SO_4, HIO_3, HNO_3, HF, CH_3COOH, H_2O_2 and NH_4OH in an effort to find an appropriate electrolyte for anodic dissolution of InP.

The experimental study was performed using a large number of:

(a) n, p, n^+ and p^+ Czochralski LEC grown InP substrates from Crystacomm, Inc. and Nippon Mining Co.;

(b) p-InP substrates grown by the VGF method, from AT & T;

(c) thermallly diffused n^+p and p^+n InP structures fabricated by us at the Cleveland State University.

(d) high quality epitaxially grown n^+p InP structures from SPIRE Corporation.

The dissolution process and the quality of the surfaces after dissolution to different depths were characterized by:

(a) an analysis of C-V, $1/C^2$-V, G-V and I-V characteristics using a Polaron Model PN4200 profiler with an exposed area of 0.009 or 0.1025 cm^2;

(b) inspection of surface topography using Nomarski and SEM microscopy;

(c) Dektak inspection of the craters;

(d) and, on selected samples, X-ray Photoelectron Spectroscopy (XPS) study of the surface contaminants and oxidation stage after dissolution in different electrolytes.

RESULTS AND DISCUSSION

As a result of this study, a new electrolyte, which we call the FAP electrolyte was developed for controlled thinning of InP resulting in smooth surfaces with good electrical characteristics and free of oxide and contaminants. The FAP electrolyte has successfully been used for accurate EC-V profiling, defect revealing and surface passivation of InP prior to growing deposition of the insulator or anti-reflective (AR) coating layers.

ANODIC DISSOLUTION

Most of the processing steps used in fabrication of InP devices such as masking, diffusion and annealing, leave contaminated and/or damaged surface layers which are not desired if good quality devices are to be fabricated.

To limit our discussion we are going to refer here only to InP structures made by thermal diffusion used for fabrication of homojunction n^+p or p^+n InP solar cells. It is known that n^+p or p^+n InP structures made either by thermal diffusion or ion implantation have the surface stoichiometry destroyed. As an example, in the case of n^+p InP structures made by closed-ampoule diffusion of sulfur into p-InP substrates, we have previously found that the phosphorus depleted dead layer can extend up to several hundred angstroms below the surface [7] and it strongly affects the solar cell performance parameters [8]. Therefore, an

important step in the fabrication of high performance InP solar cells or other devices using thermal diffusion or ion implantation consists of removing, in a controlled manner, the destroyed surface layer.

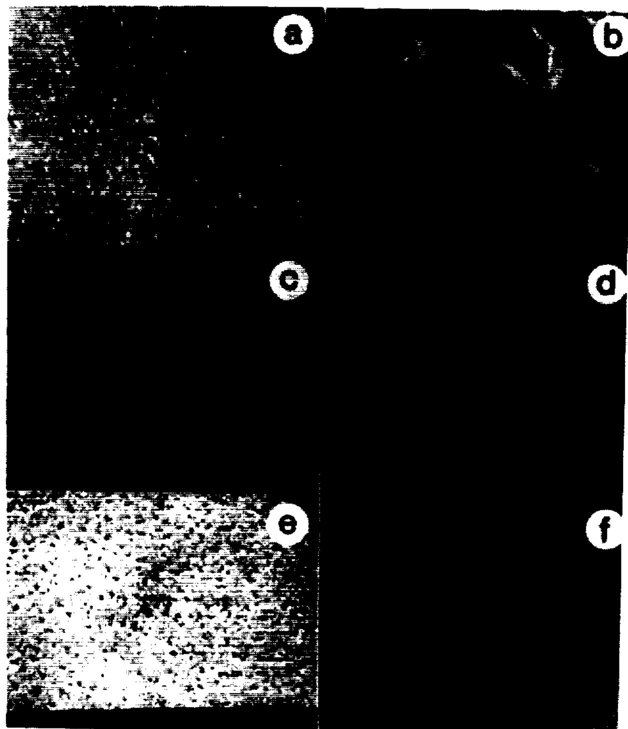

Figure 1. Nomarski and SEM micrographs of n^+p InP surfaces: a) initial (x1,100), and after photoanodic dissolution to a depth of 0.3 μm using: b) 0.5 M HCl; c) 0.5 M H_2SO_4 (x1,100); d) 1MHF (x1,100); e) 1 M HF:0.5M HCl (1:1); and FAP electrolyte (x1,100) [1].

By using different electrolytes it has been reported [9, 10] that during anodic dissolution of InP or GaAs, reactive intermediates capable of injecting electrons into the conduction band expose crystal planes other than those initially found at the surface. Usually, smooth surfaces can be obtained only if both elemental species of the InP are nearly equal. Also, surface defects generally inhibit anodic dissolution of the semiconductors by enhancing the recombination rate of photogenerated holes.

In an effort to find a well suited electrolyte for anodic dissolution of high defect density n^+p InP made by thermal diffusion [1], we have previously found that from a large number of possible combinations of oxidizing and reducing agents, the FAP electrolyte was the best answer [1]. As an example the micrographs presented in Figures 1 show the surface topography of the bottom of the craters after anodic dissolution to a constant depth of 0.3μm of high defect density n^+p InP structures using selected electrolytes. The polarization was maintained in the dissolution region (anodically relative to V_{fb}) so as to yield an anodic current density of $0.3 mA/cm^2$. As seen, the smoothest surface is obtained using the FAP electrolyte. From the C-V and I-V characteristics, and XPS inspection it was also found that the resulting surfaces are free of oxides and contaminants.

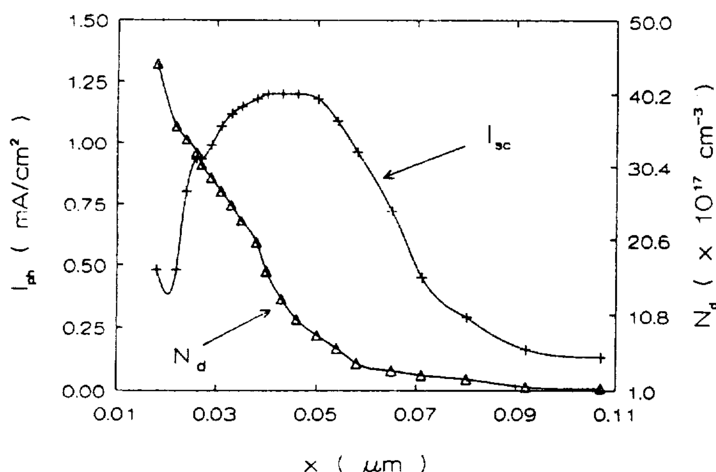

Figure 2. Variation of I_{ph} and majority carrier concentration (N_D) as a function of dissolution depth after photoanodic dissolution at 0.2V bias voltage of the n^+ layer of structure in Figure 1a, using the FAP electrolyte.

For similar n^+p InP structures as those used in connection with Figure 1, the effect of removing the highly damaged front n^+ layer using this electrolyte on

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the quality of the emitter is evident in Figure 2 by the variation of the photoelectrochemical short circuit current at the FAP electrolyte InP liquid junction under a constant low level illumination. The Isc reaches a maximum after removal of about 400Å from the surface corresponding to a surface carrier concentration of about $2 \times 10^{18} cm^{-3}$. Interestingly enough, AES profiling have shown in this case that the phosphorus depleted dead layer also extends to about 400Å below the surface [7].

EC-V PROFILING

Electrochemical C-V (EC-V) profiling of semiconductors is probably the most used and convenient method for accurate determination of thermal equilibrium majority carrier concentration depth profiles. The accuracy of EC-V profiles of III-V compounds in general and InP in particular, is strongly dependent on the surface chemistry, structural defect density, electrolyte type, current density and illumination level. For a given InP structure the electrolyte plays an essential role. In choosing the electrolyte, several criteria should be met, such as:

1. It should make good rectifying contact to both p- and n-InP surfaces;

2. The electron number for p-and n-InP should be constant;

3. The chemical etch rate should be much smaller than the anodic dissolution rate;

4. The crater must have a smooth bottom, free of oxide, with straight walls and no rounding at the crater rim.

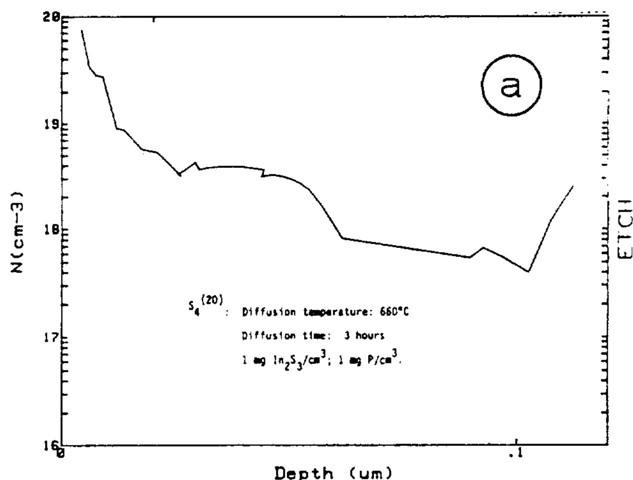

Figure 3. a) EC-V profile of a high defect density n^+p InP structure using 0.5 M HCl; b) close-up view of the bottom of the crater.

EC-V profiling utilizes an electrolyte-semiconductor liquid junction for the C-V measurements where the electrolyte is also an anodic etchant for the material being profiled [11]. The depth profiling is accomplished by alternately dissolving the surface of the semiconductor

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to some depth, thereby creating a crater-like etch profile, and then performing a low reverse bias C-V measurement.

None of the previously reported electrolytes, recommended for EC-V profiling of InP, such as 0.5M HCl [2] and the Pear etch [3] seem to satisfy the criteria of an ideal electrolyte because of one or more drawbacks such as: dissolving InP preferentially at the defect areas, forming insoluble products on the surfaces, producing rounding at the crater rim, introducing parasitic capacitance components at the electrolyte/semiconductor interface, etc., which result in inaccurate profiles.

This study was promoted by the necessity to record accurate EC-V depth profiles of high defect density n^+p InP structures. Using the 0.5M HCl electrolyte, a typical EC-V profile of these n^+p InP strucures is shown in Figure 3a. HCl based electroytes dissolve InP ppreferentially at the dislocation areas and since with this electrolyte the dissolution rate in dark for p-type InP is much higher than that of n-type InP, several micron deep pits are etched along the (111) planes before the thin (about 0.15 μm thick) n^+ layer is completely removed, as seen in Figure 3b. The large differences between the geometric and effective areas explain why the apparent carrier concentration values are over one order of magnitude higher than expected.

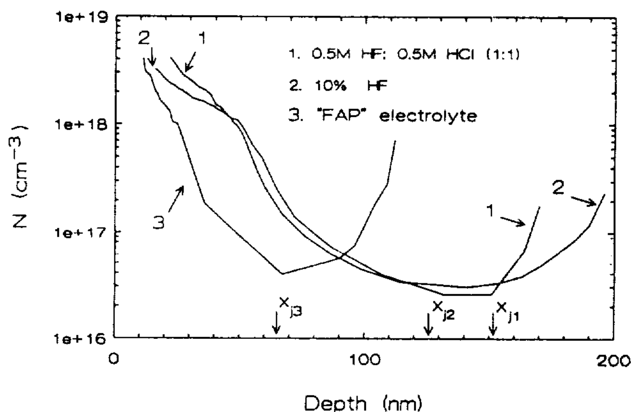

Figure 4. Comparison of EC-V profiles of an n^+p InP structure using three different electrolytes.

The accuracy of all carrier concentration profiles derived from measurements of Mott-Schottky capacitances are critically dependent on the area of the semiconductor contact and its edge definition. Due to the square low dependence of $|N_D-N_A|$ on the barrier area, small differences between the geometric and effective areas due to i) rough bottoms or ii) rounding at the crater rim, as is the case with 0.5M HCl aqueous solutions, will give rise to large errors in calculated carrier concentration depth profiles such as that shown in Figure 3a.

Using 0.5M HF:0.5M HCl (curve 1) or 10% HF (curve 2) electrolytes, as seen in Figure 4, we observe an improvement in the quality of the EC-V profiles of n^+p InP structures similar to the ones discussed in Figure 3a. The apparent junction depths (x_{j1} and x_{j2}) were still about twice than the expected value since these electrolytes still dissolve InP preferentially at the dislocation areas. Using the FAP electrolyte (curve 3), we claim the EC-V profile to be very close to the real one since all the requirements of good EC-V profiling mentioned before, are met.

Figure 5 shows the view of the craters after EC-V profiling of thermally diffused p^+n InP structures with low dislocation density [12]

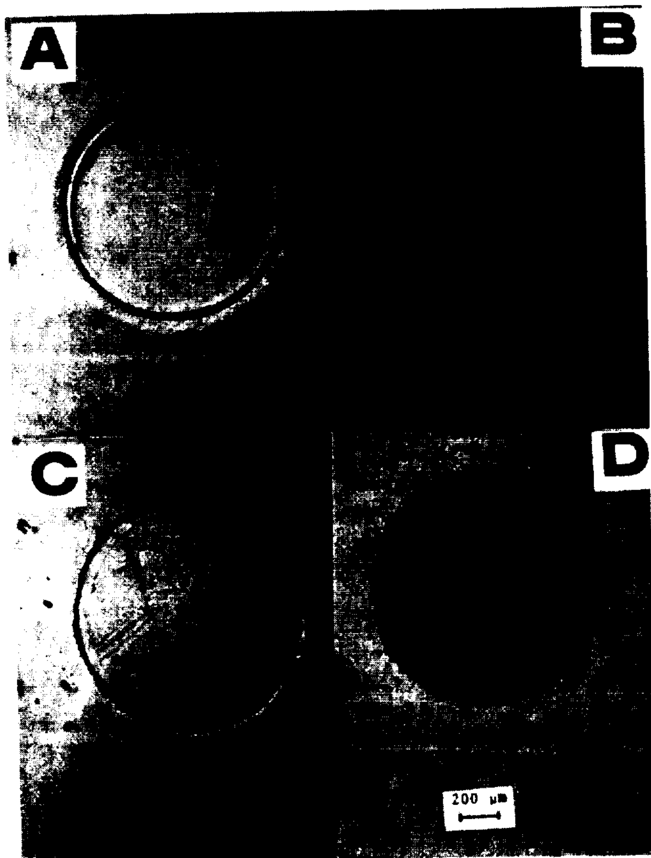

Figure 5. Nomarski view of the craters formed in good quality p^+n InP structures after EC-V profiling using: (A) the FAP electrolyte; (B) 0.5M HCl; (C) the Pear etch (in H_2O); (D) the Pear etch (in CH_3OH).

made by Cd diffusion into n-InP:S ($N_D = 3.5 \times 10^{16} cm^{-3}$) using four different electrolytes. We have also done a SEM inspection of the bottom of craters and verified that the smoothest surface is obtained by using the FAP electrolyte as seen in Figure 5A. Dektak profiles of the craters of Figure 5 shown in Figure 6 indicate that using the FAP electrolyte, the crater walls are straight with no rounding at the crater rim (as is seen to be the case for 0.5M HCl solution) and the crater bottom is flat. This, associated with very low parasitic capacitance components, explains the excellent agreement between the depth obtained by a dektak measurement and that calculated from the integrated current density of anodic dissolution using the FAP electrolyte.

In choosing a good electrolyte for the EC-V profiling, the C-V characteristics of the semiconductor-electrolyte interface must approximate a Schottky barrier over a reasonable voltage range such that the overall interface capacitance, C, is given by the well-known equation [11]:

$$1/C^2 = 2(V_{fb}-V-kT/q)/q\varepsilon\varepsilon_o|N_D-N_A| \quad (1)$$

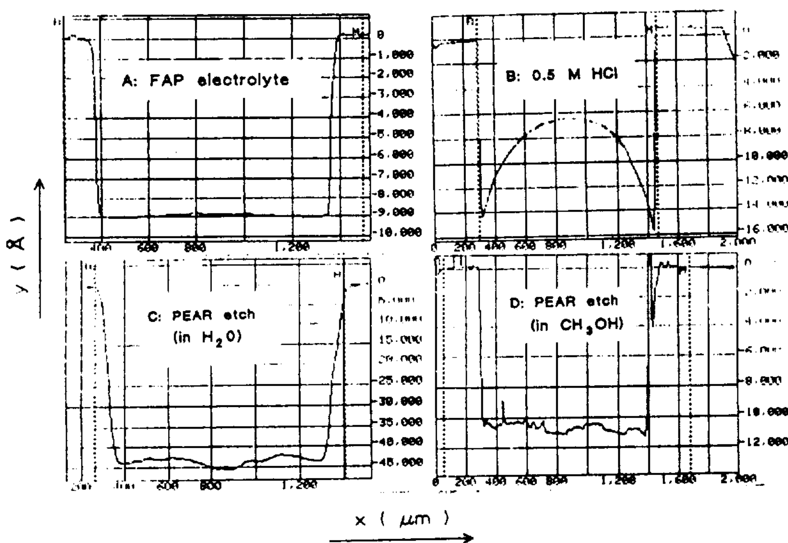

Figure 6. Dektak profiles of craters shown in Figure 5.

i.e. effects due to electrolyte, surface oxide layer, or surface states on the capacitance should be negligible. This is the case for the FAP electrolyte as shown in Figure 7 which shows C-V, $1/C^2$-V, G-V and I-V characteristics of a good quality p^+n InP structure [12]. The characteristics of Figure 7 were done after removal of the front oxide layer (≈ 30Å). One can see from Figure 7 that the FAP electrolyte is a very good choice for EC-V profiling of InP as compared to 0.5M

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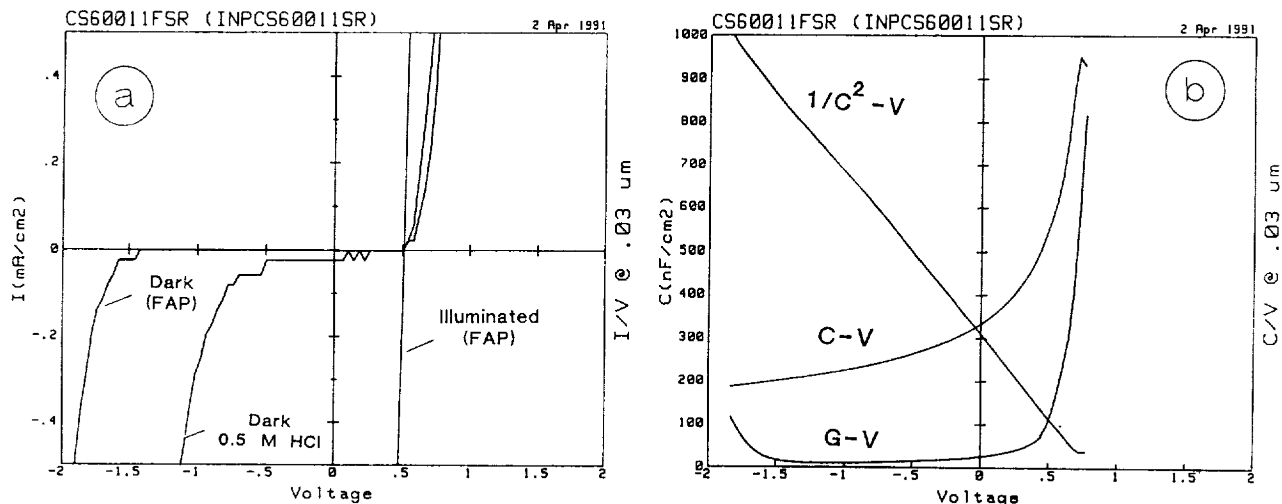

Figure 7. a) I-V characteristics of a good quality p⁺n InP structure using the FAP and 0.5 M HCl electrolytes; b) Electrochemical C-V, $1/C^2$-V and C-V characteristics at a frequency of 3 kHz using the FAP electrolyte after removal of 0.03 μm from the surface.

HCl solution. Although not shown here, in all cases studied, we found that the contribution of the parasitic capacitances to the overall interface capacitance was insignificant in the case of the FAP electrolyte as compared to previously recommended electrolytes.

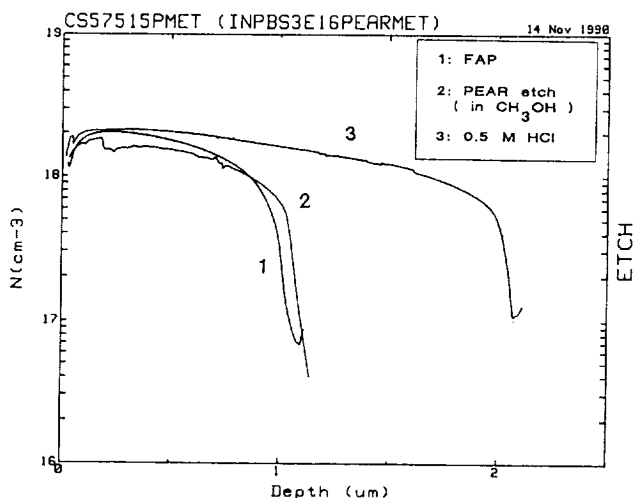

Figure 8. Comparison of EC-V depth profiles of good quality p⁺n InP structures using different electrolytes.

EC-V profiling of good quality p, n and p⁺n InP have shown that of all the previously reported electrolytes, the Pear etch (in methanol) is a reasonably good choice. However, even in this case the FAP electrolyte appears to be the best answer. In Figure 8 is presented a comparison of EC-V profiles in the case of a good quality p⁺n InP structure [12] using the FAP electrolyte, the 0.5M HCl and the Pear etch (in methanol). The large deviation of EC-V profile performed using the 0.5M HCl solution is mainly due to the rounding at the crater rim, while the fluctuations in the EC-V profile using the Pear etch is mainly due to fluctuations in the thickness of the residual surface oxide layer.

Using the FAP electrolyte after experimentation with fairly good quality p, n, p^+ and n^+ InP we found that the electron number for n- or p-InP is constant (n = 6). The relatively high anodic dissolution rate (i.e. of about 0.3 μm/h at 0.2 mA/cm²) is much higher than the chemical etch rate which for p-InP, in the dark, is below 5Å/h. The best results for p-type surfaces were obtained in the dark at a current density of 0.1 to 0.3 mA/cm² and for n-InP under low illumination at 0.05 to 0.25 mA/cm².

The low values refer to high defect density surfaces. The resulting surfaces in these cases were very smooth, free of oxide and contaminants, and with excellent electrical characteristics.

DEFECT REVEALING

Chemical etching (i.e. the Hubert etch, e.g. H_3PO_4:HBr(2:1)) is generally used for defect revealing of InP. The drawbacks are:

(a) High chemical etch rate (over 1μm/min. in most cases),

(b) The larger pits meld with the nearby smaller ones, thereby making the EPD appear smaller than it is in reality,

(c) It is not possible to perform defect density mapping as a function of depth, to reveal the defect density at the interfaces, etc.

In our experience the Hubert etch can be used for relativey accurate revealing of InP substrates or diffused structures of up to 10^5 EPD/cm^2. For higher defect density the larger pits meld with the nearby smaller ones, which makes the EPD appear to be much smaller than it really is [13].

Using higher resolution etching solutions such as HBr:HNO_3(3:1), which produce sharp pits on both p- or n-InP [14], accurate defect revealing of up to 5 x 10^6 EPD/cm^2 is possible, but due to a relatively large overall etch rate it is not possible to perform EPD revealing inside one given layer of a thin multilayer structure or at the interfaces.

Electrochemical revealing techniques can be made a lot more accurate than chemical revealing techniques, providing that the electrolyte and working conditions are carefully chosen.

The parameters that affect the width and the depth of the etch spots include the electrolyte type, dissolution time, current density and illumination level.

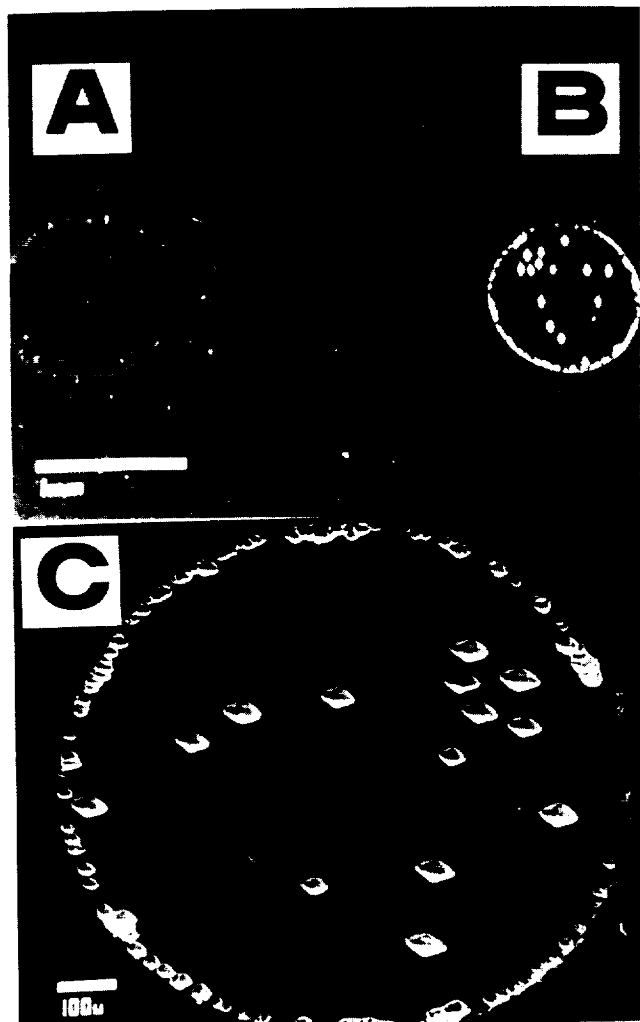

Figure 9. Defect revealing of a p^+n InP (Cd:S) structure (X_j = 2 μm) diffused at 575°C [12] after removing about 1 μm from the surface using the FAP electrolyte. SEM view of the bottom of the craters at a current density (J_c): (A) J_c = 0.3 mA/cm^2, in the dark; (B) J_c = 2mA/cm^2, under illumination; (C) close-up view of B.

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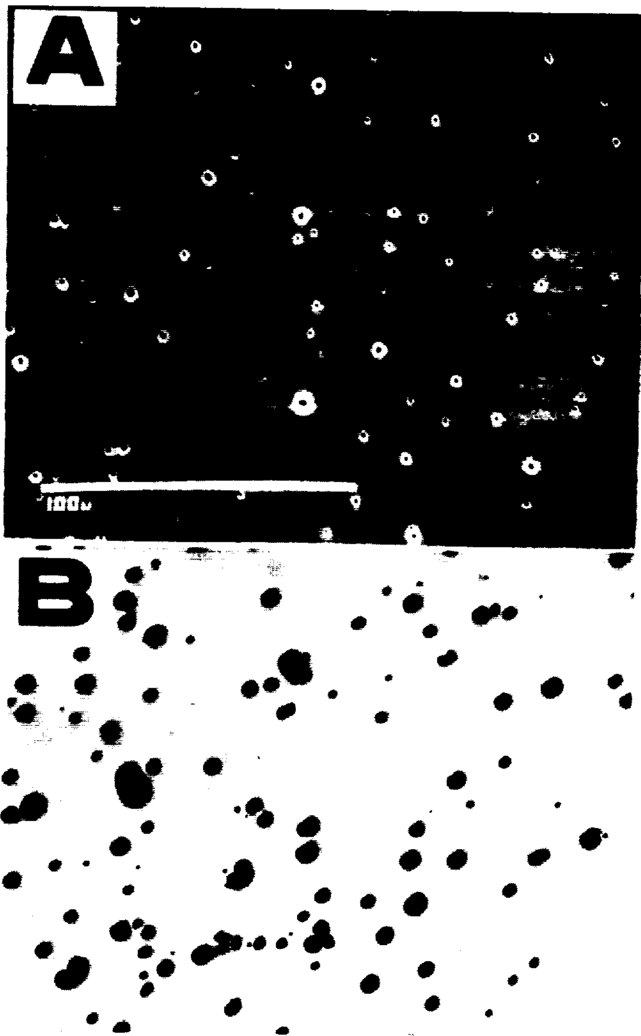

Figure 10. Deep precipitates and EPD revealed using the FAP electrolyte of (A) high defect density n^+p InP (S:Zn) structure (X_j = 150nm), after removing ~ 50 nm from the surface at J_e = 0.2 mA/cm^2; (B) EPD and deep precipitates revealed after first removing ~ 100nm from the surface (J_e = 0.2 mA/cm^2) of a good quality epitaxially grown n^+p InP (Si:Zn) structure (X_j = 0.2 μm) at J_e = 1.5 mA/cm^2. (X440)

As a by-product of this study we show that 0.5M HF:1M HCl(1:1) and 0.5M o-H_3PO_4:0.5M HF(1:1) electrolytes, due to a high aspect ratio (depth/width) of the etch pits, are good candidates for accurate defect revealing of high dislocation density n^+p InP surfaces [13]. However, as in the case of chemical revealing techniques, these electrolytes can be used for defect revealing at a given depth only for fairly homogeneous substrates.

The choice of the FAP electrolyte for defect revealing was done having in mind i) mapping of dislocation density as a function of depth and ii) defect revealing of thin multilayer InP structures. In this case the InP structure is dissolved first to the required depth as described in the first paragraph (i.e. current density of up to 0.3 mA/cm^2, see Figure 9a, in the dark for a good quality p^+-InP) and then by changing the working conditions (i.e. 1-3 mA/cm^2, under illumination for p^+-InP) preferential dissolution at the dislocation areas occur. (See Figures 9b and c). The high aspect ratio (of up to 3 recorded) after removal of only about 100Å from the surface, allows accurate mapping of dislocation density as a function of depth and/or at the interfaces. However, for each depth one needs a different spot to be used which imply that the structure should be very uniform over the area.

The FAP electrolyte was used for revealing deep precipitates in the case of n^+p InP stuctures made by closed-ampoule sulfur diffusion into Zn-doped InP substrates. The white spots seen in Figure 10a, revealed after removal of about 500Å from the surface were identified to be ZnS conglomerates.

Figure 10b shows the defect density EPDs and deep precipitates revealed after removal of about 1000Å of a good quality epitaxially grown n^+p InP structure (emitter thickness 0.2μm).

SURFACE PASSIVATION

In our experience [15] most of the chemical etching solutions used for surface passivation of InP leave a thin (at least 15Å thick) oxide layer on the surface, with a large content of contaminants, which are in part responsible for i) instabilities of devices such as MISFET's and ii) high SRV values in the case of InP solar cells.

In order to form clean, well-ordered InP surfaces, an alternative approach to chemical etching is anodic dissolution using a non-defect revealing electrolyte. This technique exploits the good inherent characteristics of the FAP electrolyte described in the previous paragraphs. After anodic dissolution using the FAP electrolyte of both p- and n-InP at current densities of up to 0.3 mA/cm^2, the resulting surfaces are very smooth, free of oxide and contaminants and with excellent electrical characteristics. This makes this technique desirable for surface passivation prior to growing or deposition of an insulator or a first layer AR coating. As an example, after removal of about 300Å from the surface using the FAP electrolyte, and further removal of about 1μm by chemical etching using a newly developed etchant [16] of a good quality p^+n InP structure [13] (initial thickness 2.5μm), we have recorded a surface state density minima $N_{ss,min}$ as low as $3 \times 10^{10} cm^{-2} eV^{-1}$ at the p^+InP/P-rich oxide interface. The newly developed so called "PNP" etchant, has the general composition $(o\text{-}H_3PO_4)_u:(HNO_3)_v:(H_2O_2)_t:(H_2O)_{1-(u+v+t)}$. The residual oxide layer left after using fuming HNO_3 is homogeneous with depth and rich in $In(PO_3)_3$. Due to its good passivating and optoelectronic properties [16] we do propose it to be used as a first layer AR coating in fabrication of high voltage p^+n InP solar cells made by thermal diffusion [17].

CONCLUSIONS

We have developed a new electrolyte which we have called the "FAP" electrolyte. We have tested it for anodic dissolution of InP surfaces, and have found the following:

1) Using the FAP electrolyte we have been able to remove heavily damaged InP surface layers and the resulting surfaces were very smooth, free of oxides and contaminants and with excellent electrical characteristics.

2) Based on our experience, we have found the FAP electrolyte to be the best choice for performing accurate electrochemical C-V (EC-V) profiling of InP, at current densities (J_c) of up to 0.3 mA/cm^2.

The experimental study of EC-V profiling of InP which was conducted using a large number of p, n, p^+, n^+ InP substrates and n^+p and p^+n InP structures seems to indicate:

(a) 0.5M HCl solution cannot be used for accurate profiling of InP even in the case of high quality substrates;

(b) The Pear etch (in the methanol system) could be used with

reasonably good results for high quality p-InP substrates. It gives relatively poor results on n-InP substrates and p^+n InP structures and the profiles are highly questionable for large front dislocation density n^+p InP structures;

(c) Due to its inherent qualities, the FAP electrolyte is, in our opinion, the best choice for performing accurate EC-V profiling of InP.

We have tested the new electrolyte with very good results for other applications, such as:

a) Revealing surface and deep dislocation defects in InP at higher current densities of 1-3 mA/cm^2, and

b) Removing the damaged and/or contaminated surface layer prior to extrinsic surface passivation, or deposition (growing) of the first layer AR coating.

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