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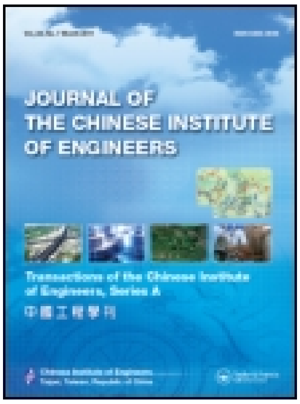

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Mesa etching characterization of InSb for high density image array applications

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MESA ETCHING CHARACTERIZATION OF InSb FOR HIGH DENSITY IMAGE ARRAY APPLICATIONS

Kow-Ming Chang, Jiunn-Jye Luo, Chen-Der Chiang, and Jacob Kou-Chen Liu*

ABSTRACT

The wet etching characteristics of InSb single crystal were investigated for high-density focal plane array applications. Two different chemical systems were used to prepare the mesa structures using standard lithography. The wet etching characteristics corresponding to these chemical systems were measured and analyzed. The results can be used to identify the dominant control mechanisms during the etching process. The etching conditions such as the chemical concentration will influence the etching characteristics, the effects of which lead to our understanding the dominant control mechanism after optimizing different ratios of wet etching chemicals. Citric acid/peroxide has been shown to produce a practical etching rate at room temperature. The dominant control mechanism for InSb mesa etching in citric acid/peroxide is surface reaction rate-limit oriented, in that it depicts promising potential in morphology and sidewall profile control for InSb mesa type device applications.

To verify the feasibility of these processes for device applications, a field emission scanning electron microscope was used to analyze the step coverage for dielectric deposition and metal layer coating. To meet the requirements of InSb high density array applications, a peroxide based chemical system with reaction rate-limit mechanism was concocted to bring to produce superior etching performance in comparison with a nitric acid based solution.

Key Words: mesa step height, mesa etching, etching mechanism, image array

I. INTRODUCTION

InSb pn junctions are widely used as infrared photovoltaic detectors and imaging sensors for the 3-5 μm spectral range (Gau *et al*., 2003; Parrish *et al*., 1991). Geometry scale-down in the junction area with low leakage current and high breakdown voltage in the reverse bias region is desired for advanced high density format area array applications (Ashley *et al*., 2002). High performance infrared image applications require detection pixels with uniform response to radiation in infrared spectral ranges of interest. This indicates the formation of uniform detection pixel structures in an array is one of the fundamental requirements for device processing.

For a (111) direction oriented InSb blanket wafer, solid state diffusion or ion implantation can be used to form the pn junctions. Mesa etching is used to create isolated pn junction islands on the InSb substrate for mesa type pn junctions. As shown in Fig. 1(a), the mesa is formed by removing the p-type conductivity material completely and part of the n-type conductivity substrate. The metallurgical junction will be formed ideally in the middle of the mesa height. This etching process defines the lateral dimension and step profile. The surface and sidewall of the mesa structure will make direct interface with the succeeding passivation or dielectric film. The control of surface morphology in mesa type p+n junctions is an important issue due to the electric field sensitivity characteristics (Lan *et al*., 1991) of the exposed peripheral.

Some dry etching processes (Greiner *et al*., 2003; Pearton *et al*., 1990; Vawter and Wendt, 1991)

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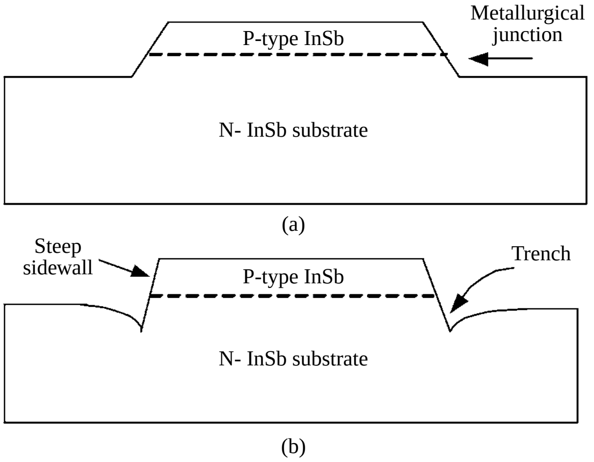


Fig. 1 (a) Schematic representation of ideal mesa structure for InSb p+n junction detection element in a two-dimensional infrared image array. (b) Schematic representation of typical mesa structure formed by lactic acid/nitric acid solution with trench and steep sidewall problems.

of InSb have been shown to produce more accurate geometric control over small device structures. However, the sidewall damage caused by physical sputtering of ions may result in electrical degradation (Fonash, 1985; Pang *et al.*, 1985; Mikkelsen and Wu, 1986; Pang *et al.*, 1988) of devices, especially for narrow bandgap compound semiconductor material. In addition to the damage issue, a low etching rate and large capital investment required for dry etching are two important factors to be considered in producing a cost effective process. Wet etching is still a widely accepted way to delineate the mesa structure on the InSb wafer surface.

Numerous wet etchants (Willardson and Goering, 1962) and etching processes have been investigated to provide the optimum surface requirements. Conventional mesa type (111) direction oriented InSb photovoltaic detectors are created by wet etching of a lactic acid/nitric acid solution (Chen *et al.*, 1992; Tu *et al.*, 1989). Unfortunately, the reproducibility and uniformity of the etching rates are hard to control (Chen *et al.*, 1992). Fig. 1(b) schematically indicates the steep sidewall and trench effect in this mesa structure. This suggests that the diffusion controlled rate-limit process is a factor in the etching process of (111) direction oriented InSb, using lactic/nitric acid solution.

Citric acid/hydrogen peroxide solutions have been studied intensively in processing GaAs based materials (Otsubo *et al.*, 1976; Juang *et al.*, 1990; DeSalvo *et al.*, 1992) and device structures due to superior performance in etching selectivity. The results indicate that GaAs etching can be controlled by a surface reaction rate-limit mechanism (Otsubo *et al.*, 1976). This etching mechanism is expected to yield better control on the device structure uniformity due to its linear proportional relation to etching time and independence from agitation and size of exposed etched area. This paper is the first report to address device structure uniformity issues in InSb high density detector array fabrication processing. We will study the feasibility of citric acid/hydrogen peroxide solution in (111) direction oriented InSb substrate wet etching and compare the wet etching characteristics with lactic acid/nitric acid solutions.

II. EXPERIMENTAL

Te-doped (111) direction oriented InSb substrates with an electron concentration of 1- 2 × 10^{15} atoms/cm^3 were used in this etching study. Standard H-line lithography technology and AZ-1350 photoresist was used to pattern the wafer surface with masked and exposed areas for mesa structure etching. The etching start condition was a surface etched by diluted 1/100 HF solution for 2 min followed by a 1 min immersion in diluted hydrogen chloride solution, insuring a native oxide thickness below 15Å. Otherwise, thicker native oxide will influence the access of oxidizing species to the InSb substrates and result in difficulty in controlling the step height of the mesas. After mesa etching, the samples were rinsed in deionized (DI) water for 5min and then dried with nitrogen.

For citric acid/hydrogen peroxide mixture, anhydrous citric acid solids were dissolved in deionized water with the concentration 50% by weight. To stabilize the reactions, this citric acid/water mixture was prepared at least one day before the etching process. The citric acid/peroxide mixture was prepared by mixing the citric acid/water mixture and 30% hydrogen peroxide at different volume ratios. For lactic acid/nitric acid mixture, 90% lactic acid was mixed with 70% nitric acid at two different ratios 10:1 and 20:1 by volume ratio.

The surface morphology and the etching rate of the InSb mesa structures corresponding to these two chemical systems were measured by optical microscope, AFM and Dektak surface profiler respectively. The step height is defined by the mesa plateau and the etched flat bottom regions as shown in the insert of Fig. 2(a). The device structure's array pixels for typical detector fabrication processes with citric acid/hydrogen peroxide solution were investigated by field emission scanning electron microscope and produced as figures.

III. RESULTS AND DISCUSSION

Figure 2 shows the etching time dependence of the step height for both 10:1 and 20:1 lactic acid/

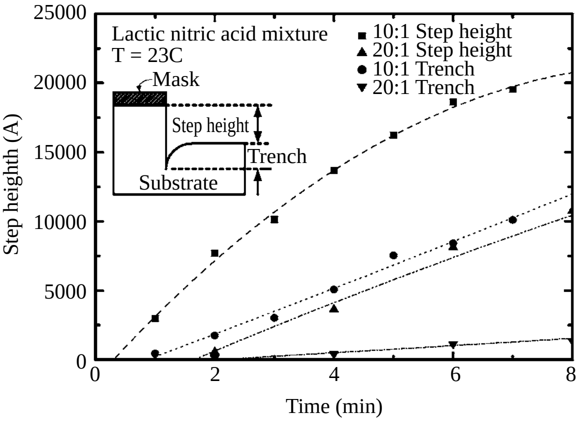


Fig. 2 Step height and trench depth of (111) B InSb mesa created by 10:1 and 20:1 volume ratio lactic acid/nitric acid solutions etching as a function of etching time.

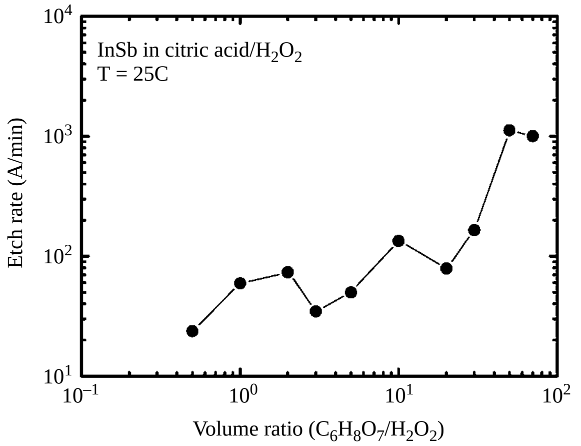


Fig. 3 Volume ratio dependence of etch rate of (111) direction oriented InSb in citric acid/H_2O_2 solutions at 25°C.

nitric acid solutions at 20°C. The legends and arrows in the figure indicate the corresponding solutions and the axis to be referenced. The step height tends to saturate after 6 minutes etching for 10:1 solution; 20:1 solution also has the same saturation trend but it occurs around 20 minutes. Further analysis of the etching data indicates the step height dependence on etching time roughly follows the square root relation.

The mesa trench curves for both 10:1 and 20:1 solutions, as shown in the same figure, indicate linear relation to etching time in the range of interest. In contrast to the square root dependence of step height on the etching time, the mesa trench will become more severe as the step height become higher. Typically, for 1.5 µm mesa height, the 10:1 mesa trench will drop to 35% of mesa height. The 20:1 solution can effectively suppress the trench effect to about 15% of mesa height.

The square root etching time dependence, stirring effect, and mesa trench effect indicate that the etching of (111) direction oriented InSb in lactic acid/ nitric acid solution 10:1 is dominantly controlled by diffusion rate-limit (DeSalvo *et al.*, 1992). According to the definition of this mechanism, the transport of fresh oxidizing species, nitric acid in this chemical system, was expected to control the reaction rate of etching. The trench and stirring effects were created by the non-uniform supply of oxidizing species during the etching process. As InSb substrate was protected by patterned mask materials such as photo resist on the surface. The etching solution above this mask material will not react to the mask and it will preserve the original freshness and composition locally. This solution on the photo resist protected surface will enhance the supply at the edges of mesa when compared to the InSb exposed surface. Higher volume ratio of lactic acid/nitric acid solution, for instance 20:1 in this study, may help to adjust the etching mechanism away from the diffusion rate-limit dominant mechanism to some degree. However, prominent mesa trench and sidewall problems, to meet the requirements of next generation infrared image applications, can not be removed completely.

To search for practical etching use conditions for citric acid/hydrogen peroxide solution, the citric acid/hydrogen peroxide solution volume ratio dependence of InSb etching rate was studied. In order to have a detectable mesa step for surface profiler measurement, the samples were etched for 10 minutes, the bath temperature was maintained at 25°C by hot plate and stirring. As shown in Fig. 3, the etch rate for volume ratio smaller than 30:1 is relatively small and insensitive to change of the volume ratio. However, it has a sharp increase for a volume ratio larger than 30:1 and will fall off after 70:1. For pure citric acid, there is no detectable etching after 10 minutes immersion. This tendency is similar to the etching of GaAs and AlGaAs (DeSalvo *et al.*, 1992) in citric acid/ hydrogen peroxide solution.

In contrast to Fig. 2, Fig. 4 is the InSb etching time dependence of the mesa height in citric acid/ hydrogen peroxide solution at 18°C corresponding to the volume ratios 50/1 and 10/1. The mesa height indicates sublinear proportional relation to the etching time. The etch rates tends to increase with respect to etching time. This phenomenon can be explained by the dissociation effect of hydrogen peroxide during the etching. The etching of InSb in citric acid/hydrogen peroxide solution didnít have an obvious stirring effect or mesa trench effect in the range of interest. In Fig. 5, the temperature dependence of the etch rate is shown. The etching rates increased exponentially with increasing etching temperature. By fitting with $\exp^{(-Ea/kT)}$ form, where

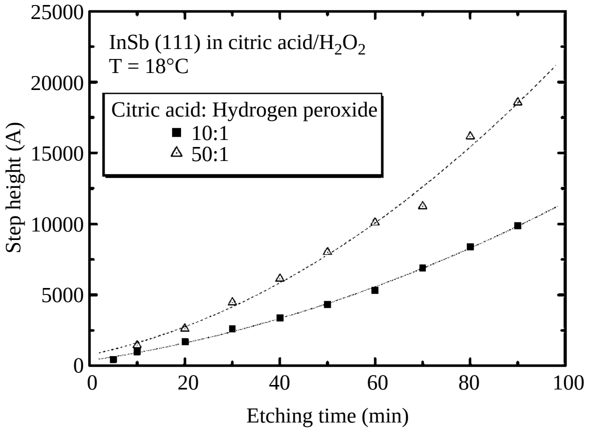


Fig. 4 Mesa step height as a function of etching time in 10:1 and 50:1 citric acid/hydrogen peroxide solution

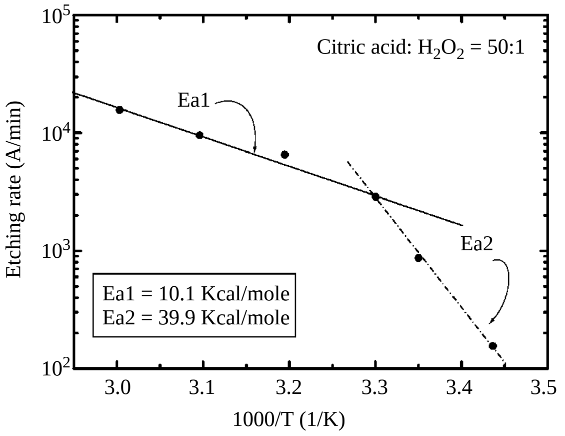
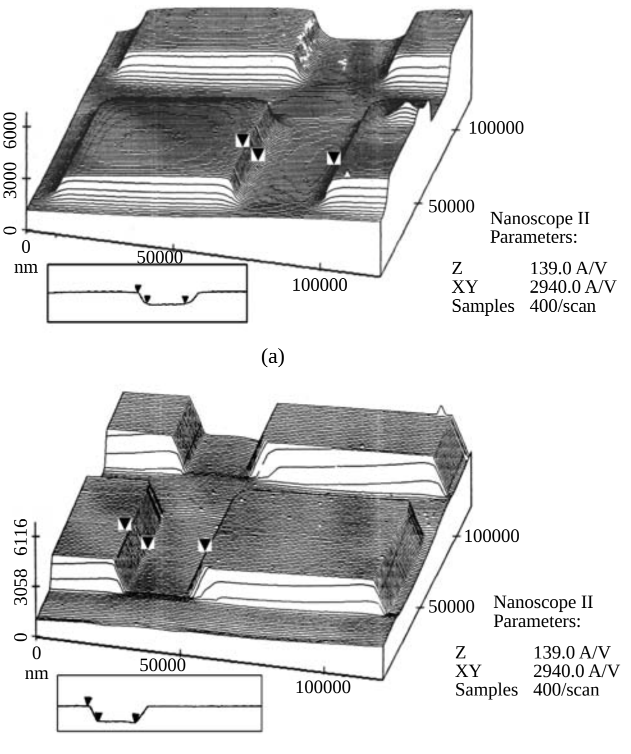


Fig. 5 Temperature dependence of the etching rates for InSb in the citric acid/H_2O_2 solution with volume ratio 50:1 in the temperature range from 18°C to 60°C. The activation energies Ea1 and Ea2 can be extracted from the data fitting of the etching rate vs 1/T in Logarithmic scale.



(a)

(b)

Fig. 6 (a) AFM Photograph and cross section view of (111) B InSb mesa array created by 10:1 lactic acid/nitric acid solution etching. Non-symmetric sidewall profiles can be seen in one mesa structure, the slope angle of the left-side step is 62 degree. (b) Mesa array created by 50/1 citric acid/hydrogen peroxide solution etching. The sidewalls in one mesa have relatively symmetric structure; the slope angle is 54 degree. The arrows indicate the corresponding locations in the mesa structure.

Ea and T represent the activation energy and temperature respectively, *k* is the Boltzmann constant. Two different activation energies were observed as the temperature changed from 18°C to 60°C. For temperature higher than 30°C, the activation energy is 10.1 Kcal/mole. This result is consistent with results of etching in 10:1 solution previous published (Otsubo *et al.*, 1976). But for the room temperature region, the activation energy is as high as 39.9 Kcal/mole. So, for practical mesa structure preparation, temperature higher than 30°C will be more appropriate for process throughput control.

The sublinear etching time dependence of etch depth, exponential temperature dependence of etching rate, and trench free behavior indicate (DeSalvo *et al.*, 1992) that the InSb mesa etching in citric acid/hydrogen peroxide solution is dominantly controlled by the surface reaction rate-limit mechanism. The dimensions and morphology of the InSb mesa array are expected to be more tightly controlled by citric acid/hydrogen peroxide solution than by the lactic acid/nitric acid solution.

As shown in Fig. 6(a), the AFM mapping and cross section view of InSb 2-dimensinal mesa array created by etching in lactic acid/nitric acid solution indicates two structure issues in the mesa structure. One is a non flat bottom surrounding the mesa plateau corresponding to the trench phenomenon measured by profiler. The other is edge dependent slope angles, which create two different slope sidewalls for two parallel mesa edges. The slope of the sidewall is 62° at the left side of the cross section view. This phenomenon may come from the crystal orientation effect. It has been shown that the steep sidewall may result in local bad coverage or void of film in the succeeding dielectric and metal deposition processes.

Figure 6(b) shows a similar mesa array structure created by citric acid/hydrogen peroxide solution etching at 50:1 volume ratio. The mesa height is

1.9 µm. The morphology for the sidewall and the isolation spacing between mesa plateaus are relatively more smooth and uniform than the lactic acid/nitric acid etched mesa structure. From the cross section view, the sidewall slopes for all the mesa edges shows relatively higher symmetry as well. The sidewall angle is equal to 54°. It is 13% smaller than the mesa sidewall created by lactic acid/nitric acid solution. These results imply that this etching process can exert more tight control over junction integrity, such as edges and corners for the mesa structure, which are considered to have a serious impact on the junction device performance (Sze, 1981) such as junction breakdown voltage and leakage current.

To verify the feasibility of the citric acid/hydrogen peroxide solution in device applications, the etching process was implemented into an InSb pn device fabrication process. As shown in Fig. 7, the scanning electron microscope photomicrography of 1.75 µm step height mesa structure prepared by citric acid/hydrogen peroxide solution measures the step coverage issues for the succeeding film deposition process. Our typical thin film processes are 2800Å dielectric SiO and 1200Å Au metal film prepared by evaporator respectively. The film thickness variation around the falling edge of this mesa structure indicates little shrinkage in thickness for either the dielectric or metal film. This performance guarantees good step coverage even for thin metal layer run over the high mesa height and provides high confidence for mesa arrays created by citric acid/hydrogen peroxide solution for high density format focal plane array applications.

IV. SUMMARY

The wet etching of InSb in citric acid/hydrogen peroxide solution provides mesa structures with advantageous characteristics of symmetric mesa edges, slope sidewalls, and flat bottom surfaces for the deposition of successive dielectric or passivation layer. It has been shown that ideal (111) direction oriented InSb mesa structures for high format array applications with more perfect surface integrity can be realized with citric acid/hydrogen peroxide chemical system compared to the lactic acid/nitric acid solution.

The basic characteristics of wet etching including etching time dependence of the etch depth, stirring and trench effect, and temperature dependence of etching rate have been investigated and compared. It has been shown that the etching of (111) direction oriented InSb in lactic acid/nitric acid solution is dominantly controlled by the diffusion rate-limit mechanism. In contrast, etching in citric acid/hydrogen peroxide solution is dominantly controlled by the surface reaction rate-limit mechanism.

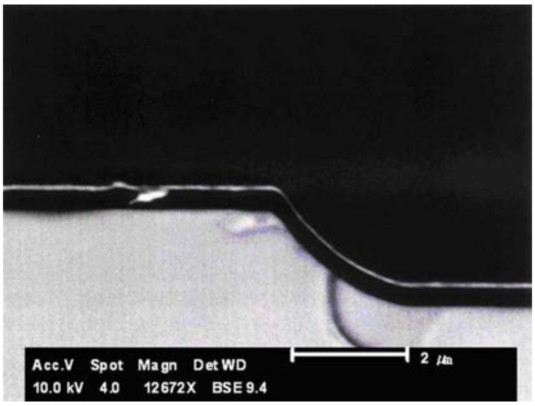


Fig. 7 SEM photomicrograph of the mesa prepared by 50/1 citric acid/hydrogen peroxide solution on (111) B InSb substrate, the mesa height is equal to 1.75 um. The surface and sidewall were coated by 2800 Å SiO and 1200 Å Au metal film respectively.

It should be noted that the surface reaction rate-limit mechanism of wet etching is more suitable for mesa array structure uniformity control, compared to a diffusion rate-limit chemical system.

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