Cleaning method of InSb $[\bar{1}\,\bar{1}\,\bar{1}]$ B of n-InSb [111] A/B for the growth of epitaxial layers by liquid phase epitaxy

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Abstract: The crystal structure of InSb [111] A/B surfaces shows that this structure is polarized. This means that the surfaces of InSb [111] A and InSb $[\bar{1}\,\bar{1}\,\bar{1}]$ B contain two different crystallized directions and they have different physical and chemical properties. Experiments were carried out on the InSb [111] A/B surfaces, showing that tartaric acid etchant could create a very smooth surface on the InSb $[\bar{1}\,\bar{1}\,\bar{1}]$ B without any traces of oxides and etch pit but simultaneously create etch pit on InSb [111] A surfaces. After lapping and polishing, some particles remained on the InSb $[\bar{1}\,\bar{1}\,\bar{1}]$ B surface, they could not be removed easily by standard cleaning process and if these particles remain on the surface of the substrate, the growth layer was not uniform and some island-like regions were observed. The purpose of this work is to remove these particles on the InSb $[\bar{1}\,\bar{1}\,\bar{1}]$ B surface. Some morphology images of both surfaces, InSb [111] A/B, will be presented.

Key words: cleaning InSb; lapping; polishing; InSb $[\bar{1}\,\bar{1}\,\bar{1}]$ B
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1. Introduction

The crystal structure orientation [111] A/B of the group III–V semiconductors is polarized and this polarity has many problems, notably that damage depth and chemical-attack rates on $[\bar{1}\,\bar{1}\,\bar{1}]$ B planes are about twice what they are on [111] A[1]. Therefore the cleaning and etching processes of these structures are difficult in respect to nonpolar [110] surfaces[2, 3]. The crystal structure of the InSb [111] A/B is that of zincblende form and consists of two InSb [111] B surface or B-face (Sb) and InSb [111] A or A-face (In) that are located in two different crystallized orientations. Each In-atom is connected to four Sb-atoms and they have different electronic structures. The Sb-atom has five free electrons, and three of them have a covalent connection with other Sb-atoms. The other two electrons are free and can generate a pair of electrons in the bulk of the semiconductor. It was found that the A-face and B-face have sp^2 and sp^3 hybrid electronic arrangement, respectively. The work function of the B-face is less than the A-face; as a result, the B-face is more active than the A-face. The B-face surface could be etched more uniformly, because it has two free electrons and can interact easily with electron acceptor locations. Generally, all of the surfaces that have (h11) crystal direction (h is an integer number) are polarized and by increasing the number of Miller-specification, the crystal defects will be decreased[4]. CP4 etchant was used for the A-face[5, 6], which is composed of {$5HNO_3$:3HF :$3CH_3COOH$}, which has a high etch rate (25 μm/s) and after etching a substrate surface will be oxidized and non-uniform etched, but etchant B-face is based on tartaric acid and has a low etch rate (0.36 μm/s), showing that this etchant could create a very smooth surface without any etch pit on the B-face but simultaneously create an etch pit on the A-face[7]. Therefore we used the B-face for the growth of an epitaxial layer by a liquid phase epitaxial method.

2. Experiments

Before lapping and polishing processes, the B-face could be distinguished by tartaric acid. To prepare this solution, we used 27 gr tartaric acid and mixed with 73 mL water, and then it was put inside an oven for 10 min at 60 ℃ in ultrasonic equipment to solute acid completely. The substrate was put in the tartaric acid etchant {1HF:$14H_2O_2$:20 tartaric} solution for 2 min. After etching, the A-face surface of the substrate was observed by optical microscope model Leica, and it was found that some etch pit is created on the A-face while the B-face was etched uniformly without any etch pit, as shown in Fig. 1. After identifying the B-face, it was lapped and polished to obtain a very smooth surface without any particles or defects. The lapping solution was prepared from150 galuminum oxide mixed with 200 mL glycol and the surface was lapped for 40 min at a speed of 20 rpm. Some particles remained on the substrate surface after lapping, as shown in Fig. 2. To remove them, the polishing solution was prepared from 200 g magnesium oxide mixed with 200 mL glycol. The polishing was done at a speed of 20 rpm for 60 min. After lapping and polishing, the surface was cleaned with acetone, alcohol and DI water, and dried with pure nitrogen gas, and it was put in petroleum ether for 12 h. After that, it was cleaned with carbon tetrachloride and trichloroethylene twice at 60 ℃. At the end of cleaning, the substrate was cleaned with acetone and rinsed with DI water. Some particles were still observed on the B-face, as shown in Fig. 2.

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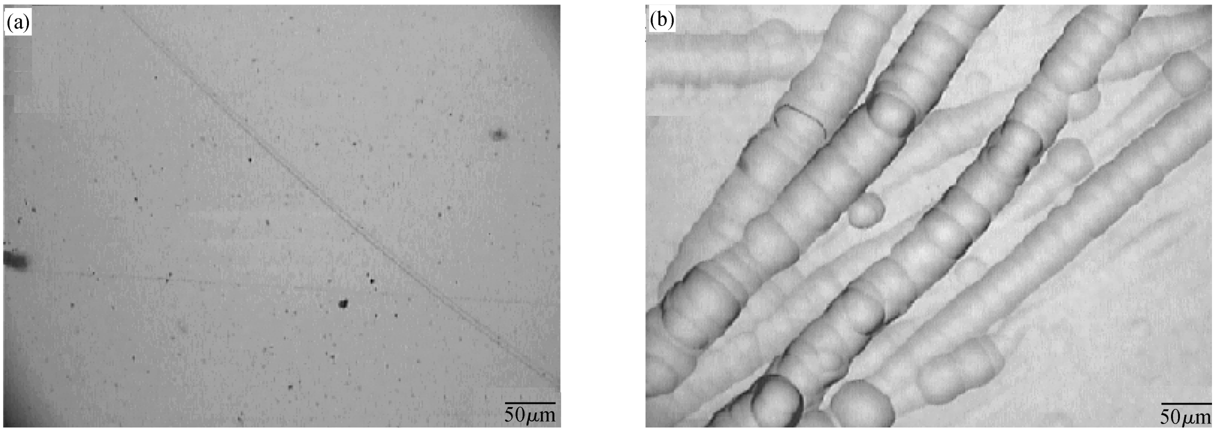

Fig. 1. (a) Surface morphology of InSb $[\bar{1}\,\bar{1}\,\bar{1}]$ B after being etched for 2 min by tartaric acid etchant without any etch pit. (b) Surface morphology of InSb [111] A after being etched for 2 min by tartaric acid etchant with etch pit on A-face.

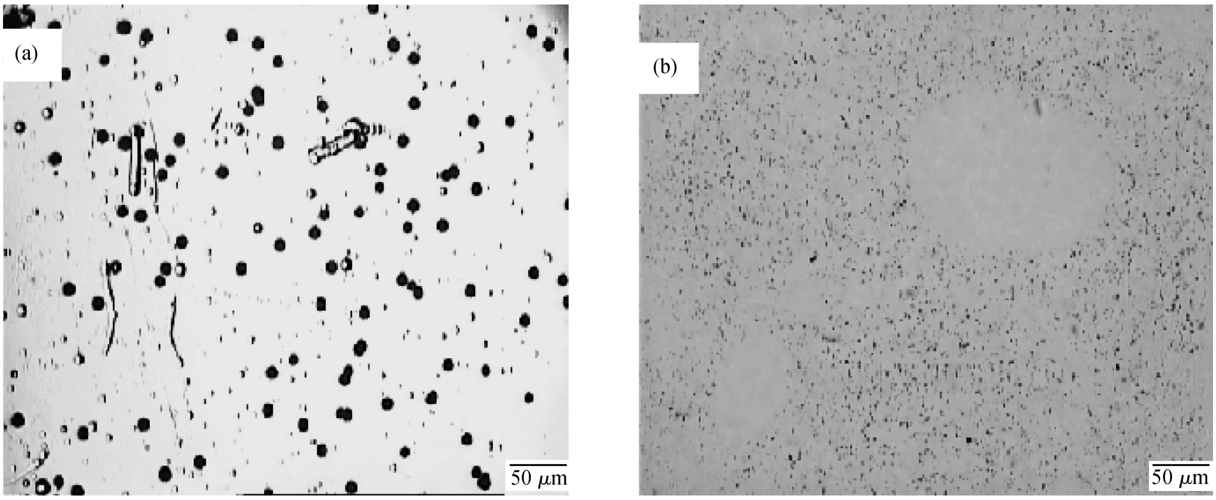

Fig. 2. (a) Surface morphology of InSb $[\bar{1}\,\bar{1}\,\bar{1}]$ B after lapping and polishing process. (b) Surface morphology of InSb $[\bar{1}\,\bar{1}\,\bar{1}]$ B after cleaning by standard method.

3. Results and discussion

As indicated in Fig. 2(b), with a standard cleaning method, the B-face InSb could not be cleaned and thereforeit was necessary to change the cleaning method. After lapping and polishing, in order to remove the particles, instead of putting the substrate in alcohol, it was put in methanol and ultrasonic equipment for 10 min. After this step, the substrate was heated at 70 ℃ with ammoniac solution 25% with hydrogen peroxide (1 : 1) to clean it completely. This cleaning step was very important to remove the whole particles from the substrate surface, as shown in Fig. 3. In order to obtain a cleaned surface, the substrate was etched with tartaric acid for 2 min and then with DI water. The substrate surface after cleaning and etching is shown in Fig. 3(b). As result, the substrate surface was cleaned completely and it was suitable for growing epitaxial films. In order to perform the LPE process, a special pure graphite slider boat was used to grow the epitaxial layer. The cleaned substrate and materials were placed inside the slider and the whole unit was transferred into the quartz tube furnace. The surface quality of the epitaxial layer depended on several preconditioning factors, such as substrate preparation cleaning, polishing, etching, melt homogenization temperature, uniformity of epitaxial layer, growth temperature and cooling rate[8]. We grew two epitaxial layers on two B-face InSb (111) and the thickness of the epitaxial layer was measured by an α-step model 500. The first substrate was poorly cleaned, so that after growing we observed some islands on the surface, as shown in Fig. 4(a). The second substrate was cleaned according to our introduced cleaning method. The experimental result in Fig. 4(b) shows a uniform epitaxial layer without any islands.

4. Conclusion

The aim of this article was to clean a B-face InSb substrate for the growth of epitaxial layers and deposition of dielectric layers. We worked on the B-face because by using tartaric acid the B-face would be etched very smoothly without any etch pit. Whereas the etchant for the A-face is CP4, which oxidizes the surface, trace and non-uniform surface will be etched. Moreover, at the end of lapping and polishing, some particles remained on the substrate surface and could not be removed by standard cleaning methods. By using a new cleaning method, all of particles were removed and the substrate was ready for

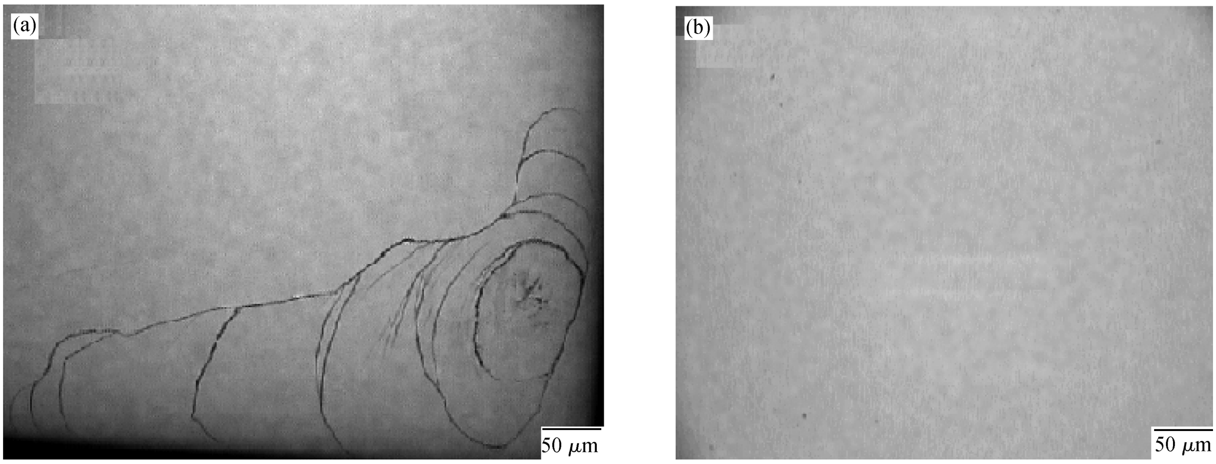

Fig. 3. (a) Surface morphology of InSb $[\bar{1}\,\bar{1}\,\bar{1}]$ B after cleaning by new method. (b) Surface morphology of InSb $[\bar{1}\,\bar{1}\,\bar{1}]$ B after etching by tartaric acid with an etch rate of 0.36 μm/s.

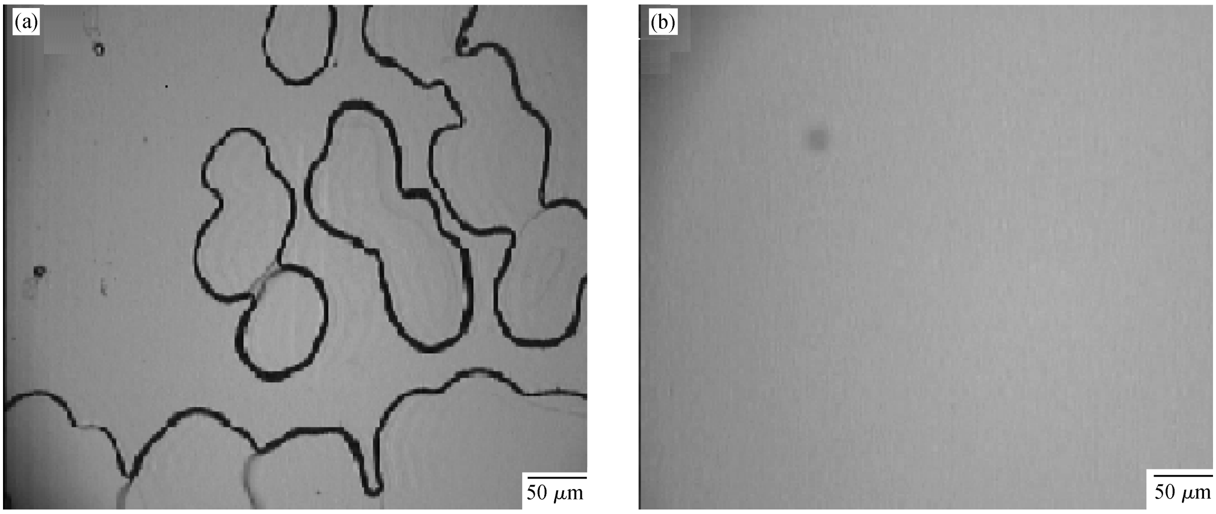

Fig. 4. (a) Surface morphology of epitaxial layer growth on InSb $[\bar{1}\,\bar{1}\,\bar{1}]$ B when some particles remain on the surface of the substrate and island like growth was observed. (b) The surface of the substrate is very clean and without particles, therefore epitaxial layer growth is uniform and without the island region.

growing uniform epitaxial films.

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