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THE USE OF ONE-COMPONENT PLASMA IN THE ICP-RIE ETCHING PROCESS OF PERIODIC STRUCTURES FOR APPLICATIONS IN PHOTODETECTOR ARRAYS

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Abstract

The paper presents the effect of ICP-RIE etching time using one-component plasma on various parameters of an InAs/GaSb type II superlattice matrix. In the studies, two samples used at different BCl3 gas flow rates were compared and it was found that using a lower flow rate of 7 sccm results in obtaining a smoother sidewall morphology. Next, five periodic mesa-shaped structures were etched under identical conditions, but using a different time. The results indicated that the ICP-RIE method using a BCl3 flow rate of 7 sccm, ICP:RIE power ratio of 300W:270W allowed the ICP:RIE formation of a periodic mesa-shaped structure with smooth and perpendicular sidewalls.

Keywords: ICP-RIE, dry etching, type II InAs/GaSb superlattice, BCl_3.

1. Introduction

The development of infrared (IR) detection technology depends primarily on improvements in the InAs/GaSb SL epitaxy [1] and post-growth processing [2]. To achieve optimal performance, the device architecture [3] as well as the mesa structure must be optimized to have vertical and smooth sidewalls to prevent crosstalk in the *focal plane arrays* (FPAs) with small pixel pitches, where the aspect ratio of the perimeter to the surface area is high [2, 4]. The roughness of the surface mesa, presence of the reaction products, and surface density of electrically active defects, including broken chemical bonds, can all affect the magnitude of the surface leakage current [5]. Mesa-type structures can be created through wet or dry etching. Previous studies indicated that both inorganic and organic acidic etchants work well for wet etching of InAs/GaSb superlattices (SLs) [5, 6]. Wet etching has a number of advantages, such as a small number of broken chemical bonds, reduced density of free carriers, and consequently, low leakage currents [6, 7]. However, undesirable reaction products are also produced and remain on the sidewall surfaces, leading to a significant contribution of leakage current. Wet etching is also anisotropic, resulting in undesired mesa sidewall geometries [8]. On the other hand, the dry etching of InAs and GaSb materials very frequently utilizes gaseous chlorine in combination with an inert gas, such as argon [9, 10]. Gaseous chlorine is preferred due to its high volatility and high etch rate, while argon ions simplify the desorption of the reaction products by bombarding the etched surface. BCl_3 etching has a lower etch rate, but its use results in smoother mesa sidewalls [11]. The utilization of BCl_3/Ar plasma has proven to be efficacious in discrete detectors. Nonetheless, it exhibits suboptimal performance when employed for mesa

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formation within matrices where the inter-pixel spacing is approximately 3 micrometers. The process involving a two-component plasma has been meticulously optimized for epitaxial structures containing one binary alloy. Upon the application of the same plasma methodology to matrices founded on SL II-type InAs/GaSb, it was ascertained that the outcomes deviated from the initial results due to the presence of indium. In dealing with plasma etching, various parameters can be controlled, such as generator power, substrate temperature, chamber pressure, and the plasma ratio and composition. In [12] using a one-component plasma of BCl_3 is reported. However, achieving a uniform etch rate for heterostructures containing two different materials is difficult as the chlorine-based plasma technology suffers from both the presence of indium chlorides on the etched surfaces and the different etching rates of the InAs and GaSb materials [13, 14, 15]. This means that the digestion process should be aimed at achieving a specific goal, rather than general optimization. This article focuses on examining the possibility of using BCl_3 single-component plasma in the dry etching process in order to obtain smooth and vertical sidewalls of SL mesa structures in a repeatable way. The BCl_3 flow rate used for etching should be relatively low to prevent excessive etching of the sample and relatively high to avoid excessive elongation of the etching process [12]. The measurable goal was to obtain a periodic mesa-shaped pixel structure with perpendicular, smooth sidewalls and rectangular corners. According to the definition given in [16], the surface roughness was assessed in our work, not the profile roughness. The paper describes in detail the inductively coupled plasma - reactive ion etching (ICP-RIE) processes of InAs/GaSb type II SL heterostructures and examines the effect of ICP/RIE etching time on etch rate, mesa sidewall slope, and the surface morphology between the pixels. Due to the lack of inert gas and increased difficulty with desorption of the gas reaction products, the verification of etch repeatability for mesas with varied depths is the key issue for one-component plasma etching. The investigated periodic structures were evaluated using *scanning electron microscopy* (SEM) as well as *optical profilometry* (OP), which is commonly used for the roughness evaluation [17].

2. Experiment

The investigated periodic structures of mesa-shape pixels were formed in a type II InAs/GaSb SL heterostructure (total thickness of 6.4 μm) using inductively coupled plasma - reactive ion etching (ICP-RIE). The nominal pixel pitch (*pp)* was 30 μm while the fill factor (*ff)* was 81%. The sample preparation process involved defining the shape of the mesa in the photolithography process. The first step after cleaning the SL samples was heating to remove any leftover water, thereby creating a highly hydrophobic surface using *hexamethyldisilazane* (HMDS). The next steps in the process were the depositing of AZ nLof 2070 photoresist film applying spin-coating to align the samples using the vacuum contact method with a photolithographic mask, exposing the samples to UV and heating them to complete the photoreaction. The final step was to remove the unexposed negative photoresist by immersing the samples in developer solution (AZ 726 MFI). The mask formation process for ICP-RIE dry etching was then finalized. Next, a set of periodic structures was produced with ICP-RIE etching. To select an appropriate BCl_3 flow rate, r_{BCl3}, two samples were prepared: 1A and 1B. Sample 1A was etched for 7:30 min at a BCl_3 flow rate of 14 sccm, while Sample 1B was etched by 8:18 min at flow rate equal to 7 sccm. Then, to verify the effect of etch time on the quality of the mesa-shaped pixels, Samples 2A–2E were subjected to a similarly procedure, *i.e*. they were etched at BCl_3 of 7 sccm for 6:30 min, 7:10 min, 7:31 min, 7:40 min and 8:18 min, respectively. The material used in the experiment was an InAs/GaSb type II superlattice. Moving to the next epitaxial layer would disrupt the test picture. A longer etching process would result in going beyond the contact layer. All the investigated SL samples were etched at room temperature by applying ICP:RIE powers of 300W:270W [10]. The powers were selected based

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on our own experience (with alternative power settings *e.g.* 250W:225W or 330W:300W), however, this configuration gave the best results. The surface morphology around the mesas and mesa size were examined using SEM. Based on the measured pixel dimension, the fill factor (*ff)* was determined. The optical profilometer was used to determine the roughness of the mesa top (*Ra_t)* and the mesa surroundings (*Ra_s)*, the etch depth/etch rate (r_{etch}) and the sidewall slope. The slope angle of the sidewalls (α) was calculated from the trigonometric function tangent of the acute angle in a right triangle (tgα). All measured and estimated parameters are collected in Table 1.

3. Results

In the first part of experiment, both periodic structures were etched to a similar depth, at over 6 µm. Fig. 1 presents SEM images of the mesas for Samples 1A and 1B.

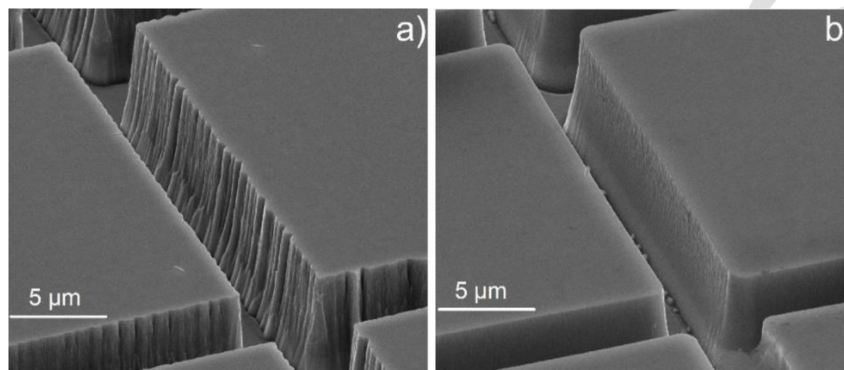

Fig. 1. SEM images of the sidewalls for: (a) Sample 1A, r_{BCl3} = 14 sccm, (b) Sample 1B, r_{BCl3} = 7 sccm.

The surface morphology of the area surrounding the pixels appears to be comparable (Fig. 1), with no significant differences despite the different gas flow rates of 14 sccm (Fig. 1a) and 7 sccm (Fig. 1b). Nevertheless, the roughness between the mesas after etching is three times worse than the roughness on the mesa top surface protected against etching. The fill factor, understood as the ratio of the photosensitive area to the total area of the pixel (30 x 30 µm), is on average about 10% lower than the nominal value of 81%. The etch rate, regardless of the BCl_3 flow rate used, was similar at about 0.8-0.9 µm/min. The sidewalls were much more developed and heavily corrugated at the higher BCl_3 flow rate, despite obtaining similar sidewall slopes of about 80º. It is worth noting that the pixel corners formed an almost 90º angle in both cases. Based on the sidewall development, a BCl_3 flow rate of 7 sccm was more favorable from the point of view of achieving the set goals.

Table 1. Parameters describing the mesa structures formed in the type II InAs/GaSb SL heterostructure under the power conditions P_{ICP} = 300 W, P_{RIE} = 270 W used in the experiment.

Sample	r_{BCl3} [sccm]	*Ra_t* [nm]	*Ra_s* [nm]	*ff* [%]	r_{etch} [µm/min]	α [°]
1A	14	6.4	14.4	72.0	0.9	81
1B	7	5.1	15.2	75.3	0.8	80

The second part of the experiment was devoted to the effect of etch time on the parameters of the mesa structures. Fig. 2 shows the SEM images of the mesas formed by etching during different time periods. One can see the cleanliness of the space between the mesas and the smoothness of the morphology of the top of pixels for all investigated samples. Most of the

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resulting pixels were not significantly different from each other (Fig. 3a, c, d, e). The morphology of the sidewall of these pixels was similar to each other and differed only in minor details. The sidewalls, which can be seen in Figs. 3a and 3c, are the smoothest and without ripples, although slight ripples are visible near the top edge.

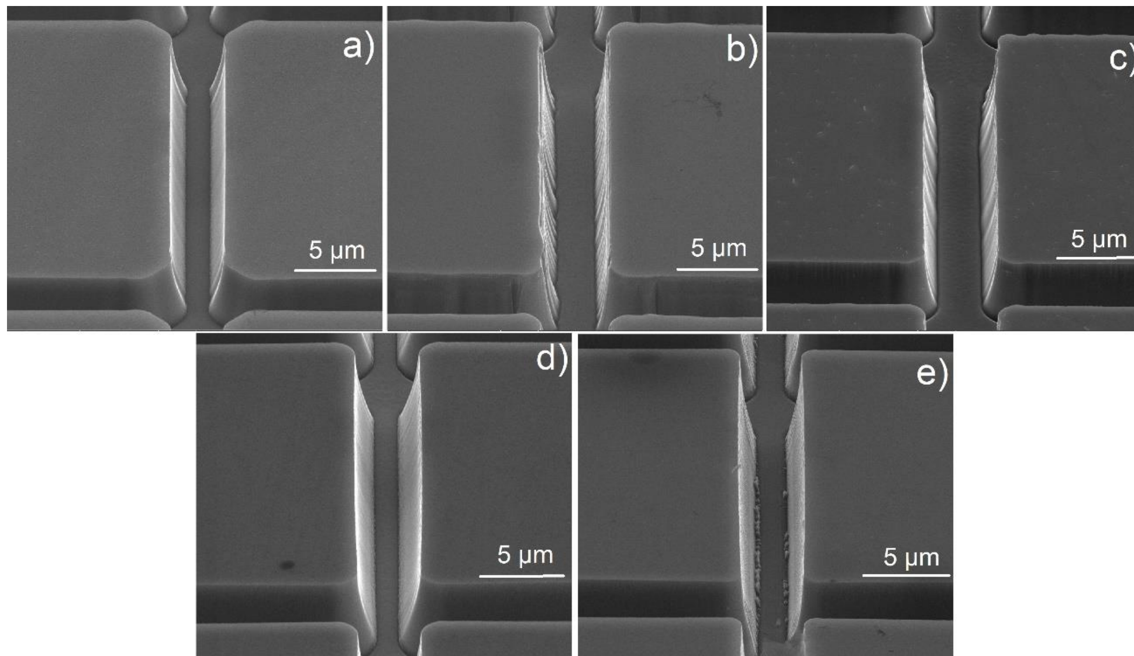

Fig. 2. SEM images of the mesa gap for: Sample 2A, t_{etch} = 6:30 min (a), Sample 2B, t_{etch} = 7:10 min (b), Sample 2C, t_{etch} = 7:31 min (c), Sample 2D, t_{etch} = 7:40 min (d), Sample 2E, t_{etch} = 8:18 min (e).

The sidewalls shown in Figs. 3d and 3e are of a lesser quality but similar to each other. Over-etching all over the surface was observed. In contrast, the most undulating and developed sidewall is seen in Fig. 3b. Note a sharp top edge present in each sample.

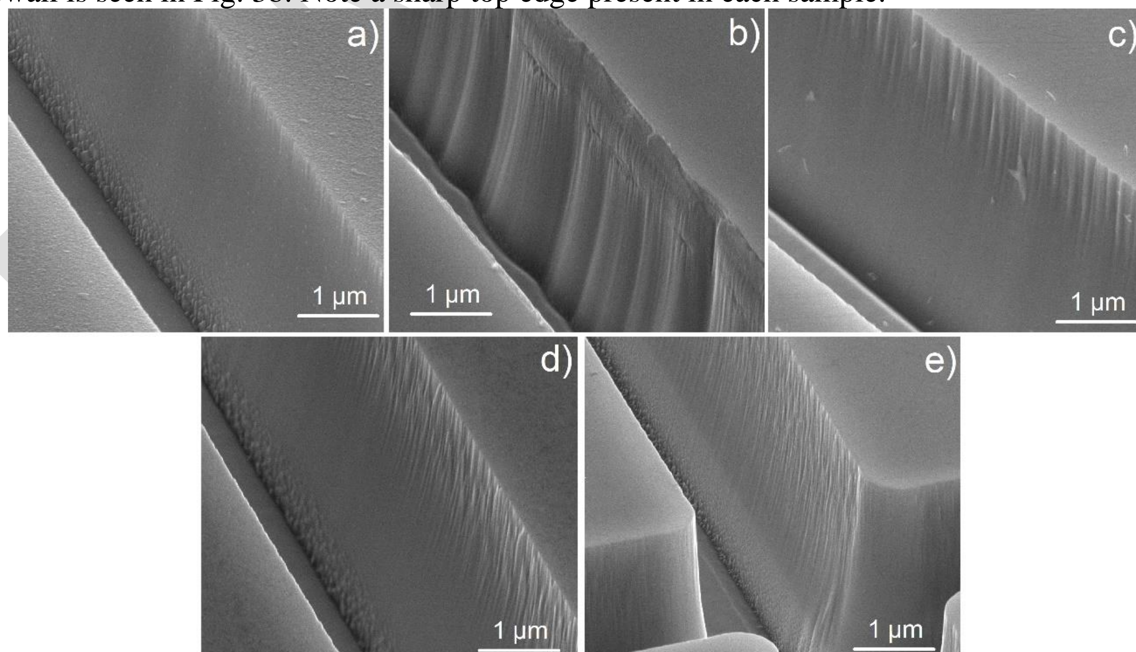

Fig. 3. SEM images of the sidewall for: Sample 2A, t_{etch} = 6:30 min (a), Sample 2B, t_{etch} = 7:10 min (b), Sample 2C, t_{etch} = 7:31 min (c), Sample 2D, t_{etch} = 7:40 min (d), Sample 2E, t_{etch} = 8:18 min (e).

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The roughness of the SL epi-structure before ICP-RIE etching was 4.5 nm. As was expected, the roughness of the mesa tops was similar to the surface roughness of the as-grown SL. The roughness of the mesa surroundings was three times higher compared to that of the mesa top as this was exposed to the plasma ion bombardment during etching. The surface morphology of the as-grown SL and surroundings of mesas are presented in Fig. 4.

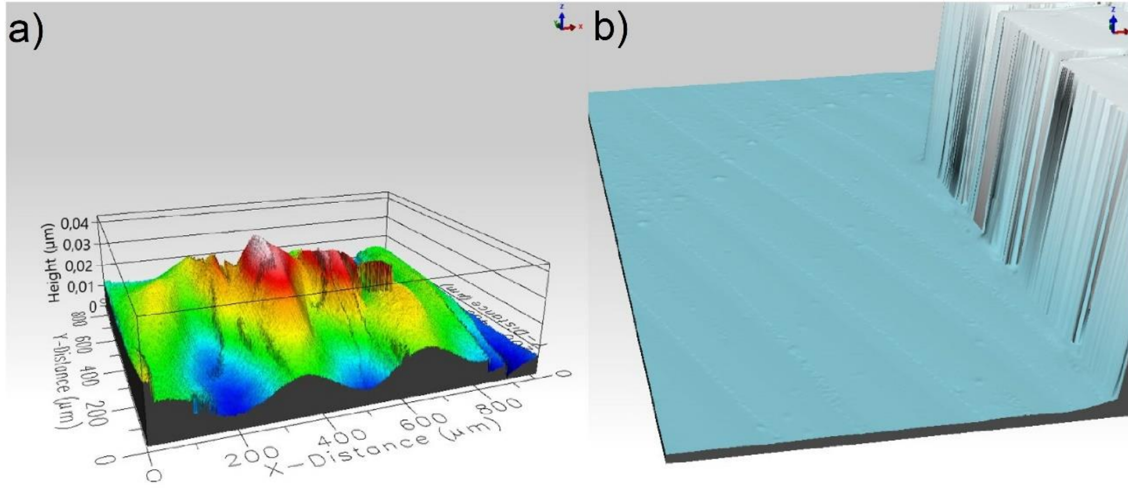

Fig. 4. View of the structure before the etching process (a) and the pixel surroundings of Sample 2A (b) from the optical profilometer. The roughness of Ra_t and Ra_s was measured on the top surface and around the pixels, respectively.

The pixel sizes in the horizontal direction (perpendicular to the growth direction) were marked in the SEM images (Fig. 5). The top surface sizes were utilized to calculate *ff*, while the bottom pixel sizes were used to approximate the sidewall angle. The fill factor for the tested samples was calculated, with the largest about 80% and the smallest about 75%.

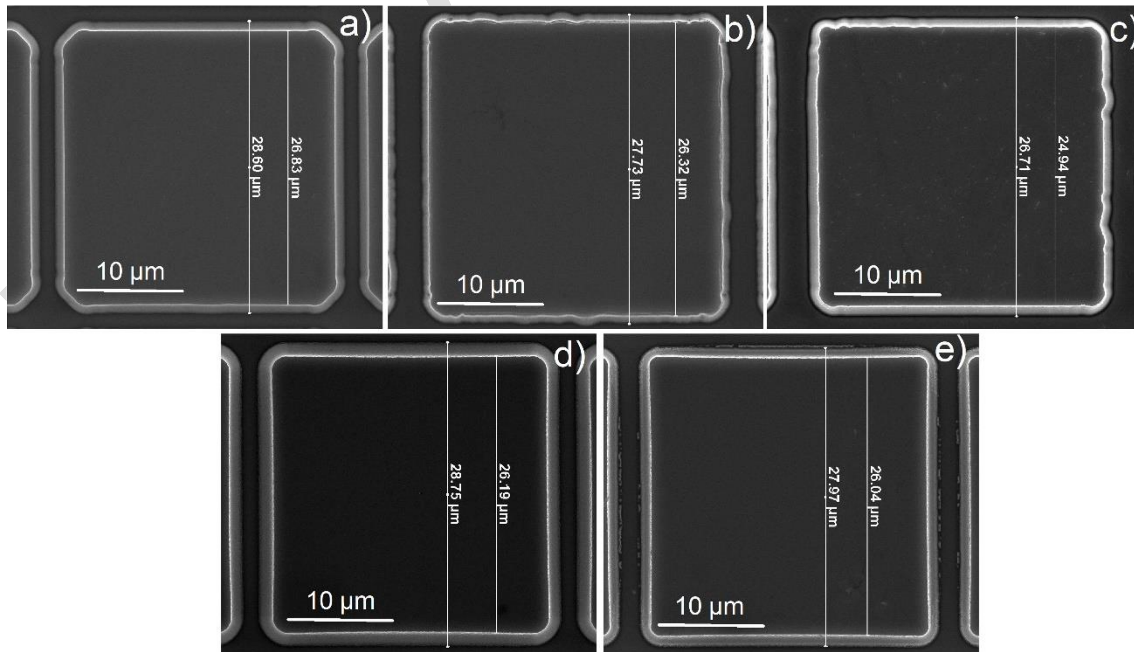

Fig. 5. SEM images of the top view of pixels for: Sample 2A, t_{etch} = 6:30 min (a), Sample 2B, t_{etch} = 7:10 min (b), Sample 2C, t_{etch} = 7:31 min (c), Sample 2D, t_{etch} = 7:40 min (d), Sample 2E, t_{etch} = 8:18 min (e).

The depth of the etch, which was indicated on the example of the mesa profile in Fig. 6, was used along with the pixel size to estimate both the etch rate and the slope angle.

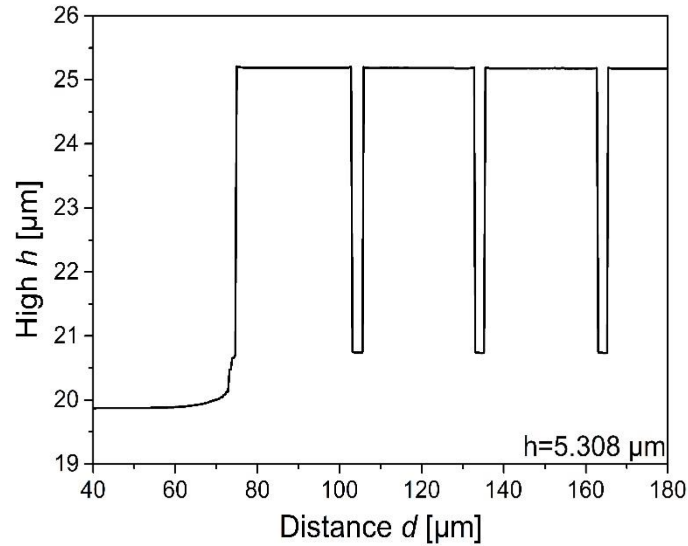

Fig. 6. Profiles of the mesas (Sample 2B, t_{etch} = 7:10 min) measured using an optical profilometer.

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All parameters estimated in the experiment are collected in Table 2.

Table 2. Parameters describing mesa structures formed in the type II InAs/GaSb SL heterostructure under power conditions P_{ICP} = 300 W, P_{RIE} = 270 W, and BCl_3 flow rate equal to 7 sccm used in the experiment.

Sample	*Ra_t* [nm]	*Ra_s* [nm]	*ff* [%]	r_{etch} [µm/min]	α [°]
2A	3.4	13.9	79.6	0.65	79
2B	4.3	14.0	77.0	0.72	81
2C	4.7	14.9	75,9	0.71	81
2D	5.0	15.0	75.7	0.73	80
2E	5.2	15.2	75.3	0.76	80

4. Analysis of results

The dependences shown in subsequent Figures from 7 to 10 present the effect of etch time on the characteristic parameters describing the pixel formation: roughness, fill factor, etch rate and slope angle of the sidewall. Etch time was the only variable parameter during the etching process. The other parameters were constant: ICP:RIE powers of 300W:270W, maintained one-component plasma with a BCl_3 flow rate of 7 sccm, the sample temperature of about 23°C, and a type II InAs/GaSb superlattice heterostructure as the object of the experimental investigation.

The first dependence presented in Fig. 7 concerns the roughness. One can see that the surface roughness after the etching process (around the pixels, labelled as Ra_s) is three times higher than the one that was not etched (on the pixel top, labelled Ra_t), but it is slightly dependent on the etch time in the range from 13.9 nm to 15.2 nm. Maintaining the roughness at an almost unchangeable level can be evidenced in the stable etching conditions.

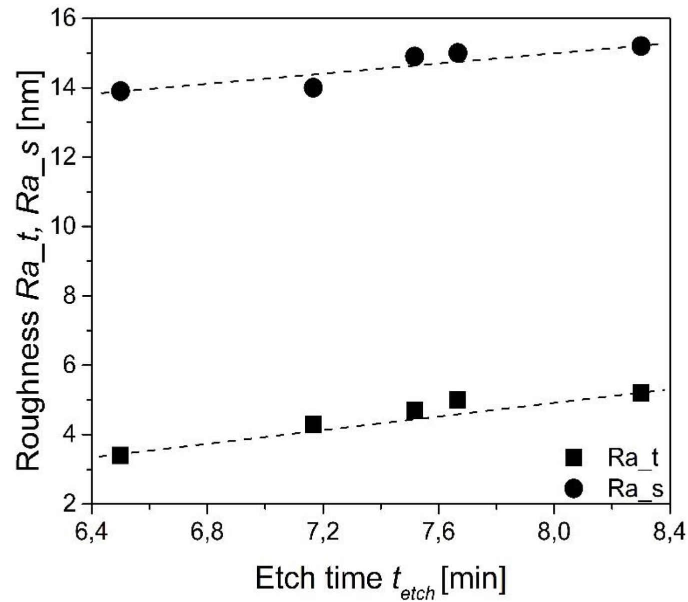

Fig. 7. Roughness of Ra_t (squares) and Ra_s (circles) vs. the etch time for the investigated structures.

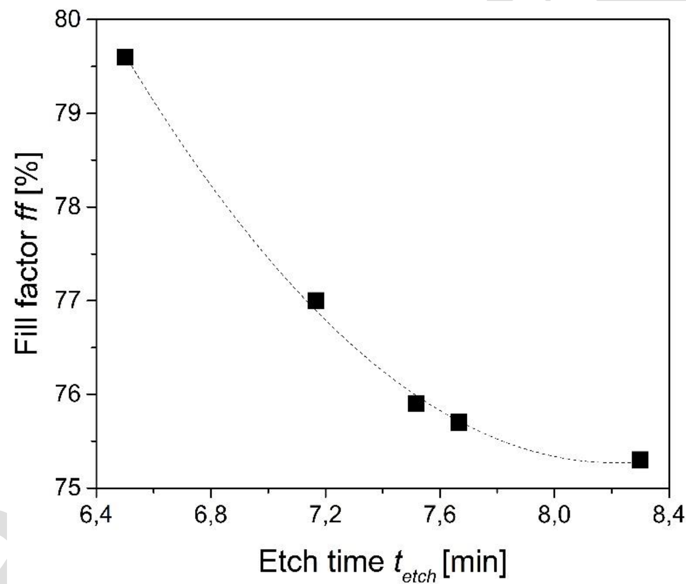

Fig. 8. Fill factor vs the etch time for the investigated structures.

The dependence of the fill factor on the etch time is shown in Fig. 8. One can see that the longer the etching, the smaller the fill factor. Although ICP RIE etching is anisotropic, the *ff* value decreased with the depth of etching. It is likely that with longer etching, pixel dilation is more apparent, which affects the fill factor. However, a fill factor above 75% is a satisfactory result [18].

The depth of etching was in the range of 4.2 µm – 6.4 µm and, as expected, it was linearly dependent on the etch time. An increase in the etch rate from 0.65 µm/min to 0.76 µm/min was observed in the investigated period of time, see Fig. 9. The time-dependent characteristic in our case may be due to insufficient heat removal from the sample during increasingly longer etching processes. In an ideal case, the etch rate is independent of the etch time.

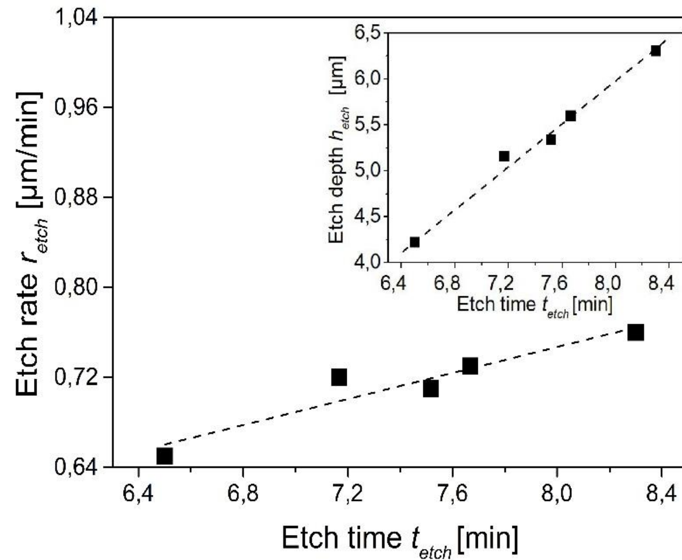

Fig. 9. Etch rate vs etch time for the investigated structures.

Fig. 10 presents the slope angle of the sidewalls estimated for all analyzed samples. The length of the etching has a minor effect on the sidewall angle. A slight difference of 3° can be observed between the values of slope angle determined for the etched samples. Obtaining a mesa with an angle of about 80° results in an increase in the active area of the detector and a decrease in the sidewall surface of the mesa. It can also reduce the leakage currents of the photodetectors.

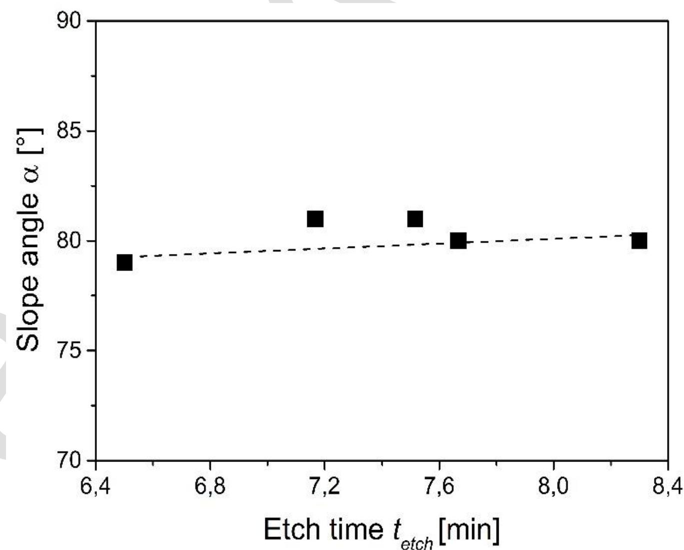

Fig. 10. Slope angle of the sidewalls vs the etch time for the investigated structures

5. Conclusions

The effect of etch time (6:30 min, 7:10 min, 7:31 min, 7:40 min and 8:18 min) on the roughness of the mesa walls, fill factor, etch rate and slope angle of the sidewalls was investigated for the InAs/GaSb type II superlattice used in the ICP-RIE process.

In the first part of the experiment, two samples were compared for respective BCl_3 gas flow rates (7 sccm and 14 sccm). A BCl_3 gas flow rate of 7 sccm allowed the achievement of a smoother sidewall morphology, compared to that of 14 sccm. This indicated that the lower BCl_3 gas flow rate is more preferable for further investigations than the higher one.

In the next step, five periodic mesa-shaped structures under identical conditions (ICP:RIE at 300W:270W powers and a BCl_3 gas flow rate of 7sccm) were etched, but for different times. After evaluating the morphologies of the mesa sidewalls (Fig. 3), it can be concluded that they are smooth, and that they do not differ significantly from each other, except with the sample etched for 7:10 minutes. The roughness of the pixels' surroundings in comparison to the roughness of the mesa top wall was three times higher, but this was kept constant with the length of the process time at one level. This factor proves the stable conditions of ICP-RIE etching using one-component plasma. The fill factor decreases with increasing etch time, which may be caused by the length of the process. The longer the process takes, the greater the possibility of noticeable sideways etching. Analyzing the graph in Fig. 9 for the etch rate, one can see that it changes in a strictly controllable way. The etching time has also no effect on the angle of the sidewall (Fig. 10), and the conditions chosen in this way make it possible to obtain a sidewall with an angle of about 80 degrees, resulting in an increase in the active area of the detector and a decrease in the volumetric sidewall of the mesa.

The conclusion for the presented results is that stable and reproducible etching conditions have been developed for the one-component BCl_3 plasma ICP-RIE method. Particularly, dry etching under a BCl_3 flow rate of 7 sccm, ICP:RIE power ratio of 300W:270 W at RT allowed the forming of the periodic mesa-shaped structure with smooth and perpendicular sidewalls (slope angle of about 80°) in a type II MWIR InAs/GaSb superlattice.

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