Progress in orientation-patterned GaP for next-generation nonlinear optical devices

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ABSTRACT

Orientation-patterned GaP is a promising nonlinear material for frequency conversion in the mid and longwave IR (2-5 μm and 8-12 μm) by quasi-phase matching. As an alternative to OPGaAs, OPGaP has the advantage of having lower two-photon absorption in the convenient pumping range 1 – 1.7 μm. We report recent results on development of thick QPM GaP for high power tunable laser sources radiating in the mid IR. Two are the major challenges to producing OPGaP: development of simple techniques for preparation of patterned templates and a technology for fast epitaxial growth of thick, high quality GaP on these templates. The focus was to adapt/simplify the wafer fusion process for OPGaP template preparation. Then increase the growth rate and layer thickness of regrown material, while maintaining vertical domain propagation. The growth experiments were conducted in a horizontal quartz reactor, using a standard hydride vapor phase epitaxial process. The growth was performed on: (i) plain (100) GaP; (ii) half-patterned (HP) and (iii) orientation-patterned (OP) templates, fabricated on (100) GaP. Up to 370 μm thick layers with high crystal and optical quality were reproducibly grown on plain material. Growth on HP templates resulted in up to 470 μm thick layers with rectangular mesa's shape. These results were used to determine the optimal substrate and pattern orientations. HVPE growth on OP templates achieved stable growth rates of 50-70 μm/h with domain walls propagating vertically, following the periodicity of the initial pattern, and resulted in the first 350 μm thick device quality OPGaP.

Keywords: hydride vapor phase epitaxy, nonlinear optical materials, quasi-phase matching, orientation patterned templates, frequency conversion devices, laser sources in the mid IR and THz region

1. INTRODUCTION

Compact, high-brightness, tunable, and room temperature operating laser sources in the mid IR and THz are in great demand for IR counter measures and other military and commercial applications, such as scanners, gas sensing, leak detection, pollution monitoring, process control, and medical imaging. Starting with the evaluation of a great variety of binary and ternary semiconductors [1] as direct sources, the field expanded to include a number of other alternative approaches. One of the most promising of these is to convert the frequency of available mature pump lasers into the wavelength of interest via a nonlinear material. Initially employing bulk birefringent phase-matching (BPM) crystals [2], the idea was eventually extended to compensating phase velocity dispersion exploiting the quasi-phase matched (QPM) approach. Compared to BPM, where Poyting-vector walk-off, inconvenient phase-matching directions and temperatures are a problem, QPM provides the advantage of engineered phase matching that can be designed to match any frequency within the transparency range and in a direction to coincide with the largest element of the $\chi^{(2)}$ tensor.
The first practical realization of such a structure was periodically-poled ferroelectric oxide crystals, like $LiNbO_3$ (PPLN). PPLN is one of the only QPM materials that has reached the production line; however, the usage of PPLN is limited to wavelengths shorter than 4 μm [3] due to strong intrinsic absorption. In non-ferroelectric materials QPM is achieved by

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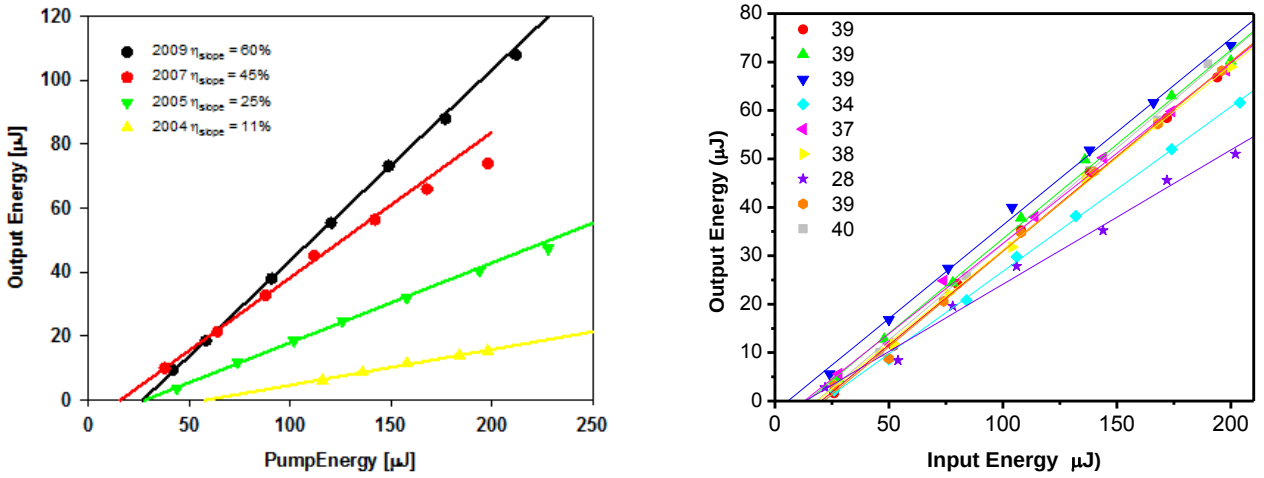
spatially inverting the nonlinear susceptibility. Initially this was done through alternating the orientation of wafers in a stack [4]. However, high optical losses observed at the wafer interfaces and the small layer thickness that is needed to satisfy the QPM conditions made these approaches unsuitable for practical devices. The use of planar processing methods, adopted from the microelectronic industry, made possible the practical realization of microstructured materials for QPM interaction. Thus, QPM is now more favorably realized in semiconductors by growing thick layers of material on a template, designed with the desired pattern periodicity. GaAs with its broad IR transparency, high nonlinear optical susceptibility and mature growth technique [5] is an example of a successful QPM story. However, OPGaAs is not a perfect material for due a high two-photon absorption (2PA) in the convenient pumping range 1–1.7 μm [7]. Compared to GaAs, the GaP has a negligible 2PA in the same region, with comparable nonlinear susceptibility, higher thermal conductivity, lower photorefractive index and broad transparence, [8]. These factors make GaP one of the most promising QPM materials at the moment. Thus, the rapidly increasing interest has led to the design of the first GaP frequency conversion device (FCD) based on stacked GaP wafers [9] and to the first demonstration of QPM parametric fluorescence in periodically inverted GaP [10]. Epitaxial growth of periodic GaP by MBE [10, 11] and MOCVD [12] is ongoing. The only growth approach with proven capability of producing apertures large enough for high power applications is halide vapor phase epitaxy (HVPE) [13]. This article summaries the efforts to resolve, some specific issues related to the HVPE growth of GaP on plain GaP wafers, such as low growth rates [14], poor crystalline quality [15] and absorption between 2-4 μm, observed in n-type GaP [16, 17]. A description of how the optimal substrate and pattern orientation were determined by growth on HP templates to improve the techniques for preparation of OPGaP templates. Additional details how the wafer bonding technique was adapted to of GaP are also given. Finally, special attention is paid to the growth on OPGaP templates, which resulted in the first device quality 350 μm thick OPGaP.

2. OPGaAs

The interest of AFRL in QPM materials dates back to the mid 70's with the works of Szilagyi, Hordvik and Schlosberg, who describe a series of experiments with a stack of GaAs plates with alternating polarity, ordered at a Brewster angle for frequency doubling of laser radiation at 10.6 μm [18]. The interest to the QPM GaAs, grown by HVPE on OPGaAs templates, was resumed near 30 years later with the advance of the technology for fabrication of microstructured materials as the following years are marked with a rapid and constant improvement of the output power (Fig. 1-a) and the homogeneity of the material (Fig. 1-b)



(a) (b)

Figure 1. Output energy as a function of the pump energy in progress (a) at different position of the laser beam (b).

After achieving mm-thick OPGaAs with good domain fidelity, the study now is focused on investigation of the impurity incorporation and other defects with impact on the optical properties of the structure, as well how these properties change in the transition area between the domains. Development of compact fiber coupled devices is the other current direction of the OPGaAs research.

3. EXPERIMENTAL PROCEDURES

HVPE growth experiments were performed in a 3-inch in diameter horizontal hot-wall quartz reactor, designed at AFRL and customized to work at low pressure, as shown in Fig. 2. The hot wall reactor is heated with a 3-zone resistive furnace. PH_3, Ga, and HCl were used as precursors, and H_2 was used as a carrier gas to mix, dilute and deliver the reactant species. Growth conditions such as total gas flow less than 250 sccm, substrate temperature in the range of 700–740°C, and reactor pressure < 10 Torr, resulted in fast, high quality, thick growth with high reproducibility of the measured material parameters. Three different types of substrates have been used: (i) unpatterned (bare) GaP wafers; (ii) HP templates, on which every other stripe is masked by 1-µm thick Si_3N_4 layer; and (iii) OP templates. OP templates were fabricated by either a sub-lattice inversion MBE assisted process; or by a wafer fusion bonding technique. (100) GaP wafers without and with 2° or 4° miscuts towards (011) or (111)B were used to fabricate the patterns. The stripes of the patterns were oriented along either [011] or [01$\bar{1}$] direction. Details about these patterning techniques are given elsewhere [19, 20].

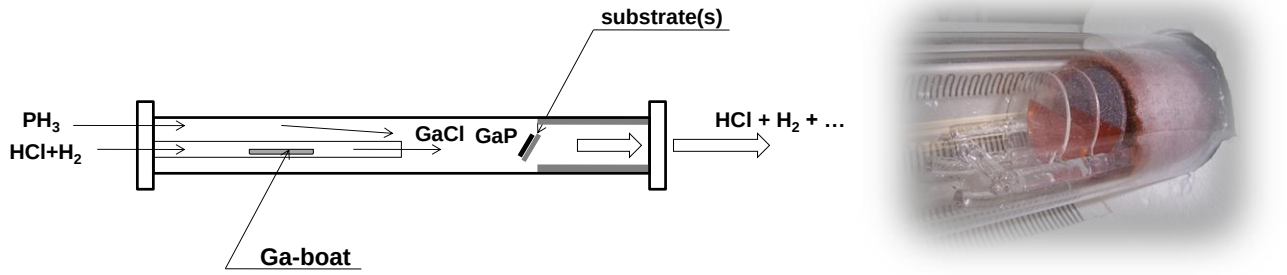


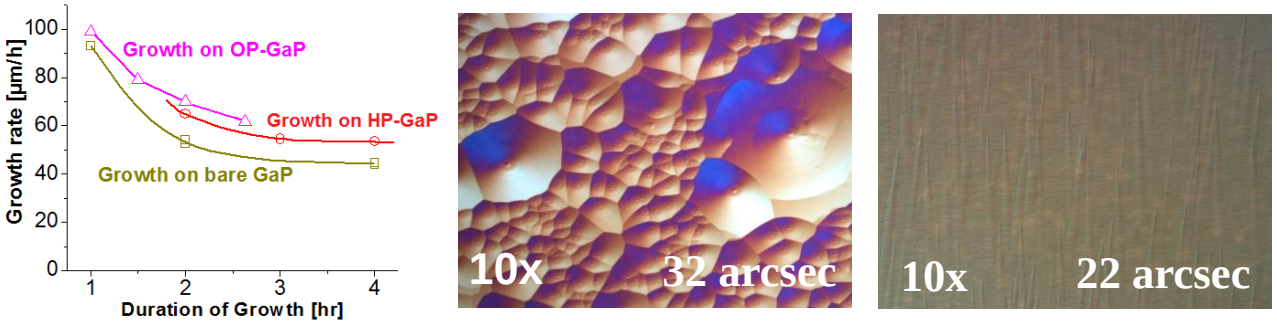
Figure 2. HVPE growth of GaP (a). Two halves of 2-inch GaP wafer, grown in an 8-long, stable process, which was free of any visible (in front and around the substrates) parasitic reactions (b)

Surface morphology and cross section were characterized using Nomarski and Atomic Force Microscopy (AFM). The structural quality of the HVPE GaP/GaP layer was determined by high resolution x-ray diffraction (HRXRD) using omega-two theta rocking curves of the (004) plane. Electrical (mobility and carrier concentration) and optical (IR transmission and absorption) properties were evaluated using room temperature Hall measurements (Van der Pauw method), and a Cary 5000 spectrophotometer and Thermo-Nicolet FT-IR, respectively.

4. RESULTS

4.1. Growth on plain GaP wafers

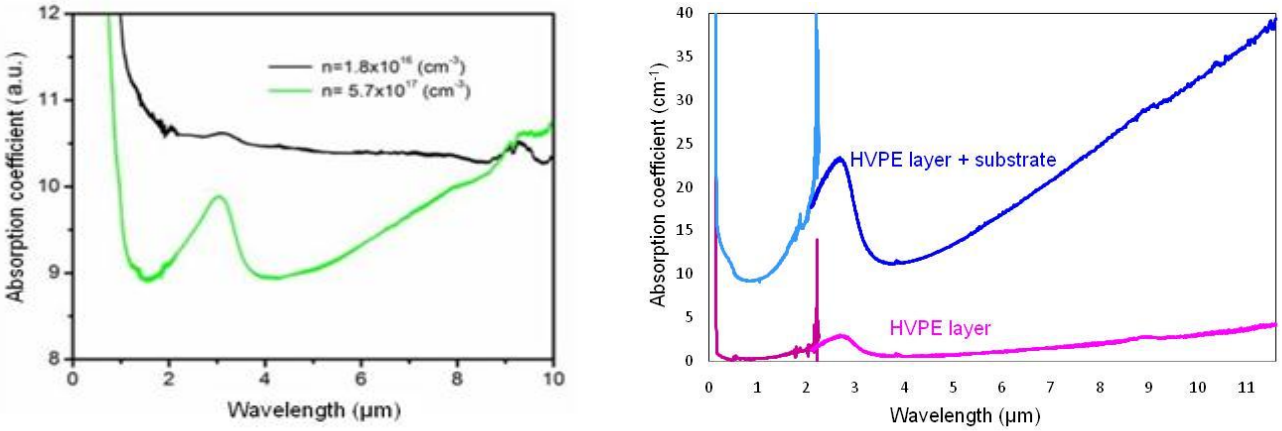
By properly chosen growth parameters, based on understanding the growth mechanism (especially the importance of the de-chlorination rate on the substrate surface), the growth rate was significantly increased not only during the growth on plain, but also during the growth on HP and OP templates (Fig. 3-a). The growth rate in 1-hour long experiments on plain substrates achieved ~ 100 µm/h and leveled off at 45 µm/h at longer growth durations (4-8 h). The growth rate on (100) GaP wafer with a 4 degree misorientation towards (111)B was faster than on wafer with no missorientation., which is due to the high density of atomic terraces providing more bonding sites. Different surface morphology, including intensive hillock growth around screw dislocations, orange peels growth, or specific mosaic structures, crossed by stacking faults were observed (Fig. 3-b). On wafers with no missorientation proper corrections in the supersaturation helped to switch the 3D growth to look-like 2D growth [21] with roughness of the grown layer < 1 nm (Fig. 3-c).



(a) (b) (c)

Figure 3. Growth rate on different substrates/templates as a function of the growth duration (a). Different types growth on the layer surface (b and c)

IR absorption losses, due to n-type charge carriers in the 2 to 4 μm range [22, 23], were also studied as a function of the carrier concentration. It was found a direct correlation between the IR absorption and the carrier concentration. Fig. 4-ashows the IR absorption of two samples grown at two different temperatures of the furnace mixing zone. A reduction in the mixing zone temperature by 10°C reduced the incorporation of Si coming from the quartz parts led to a reduction in the electron concentration from $5.7x10^{17}$ cm^{-3} to $1.8x10^{16}$ cm^{-3}. Growths at such conditions (Fig. 4-b) clearly show that the IR absorption in a 370 μm thick HVPE layer is significantly reduced (especially in the 2-4 μm region) in comparison to the IR absorption when the two, the layer plus the substrate, are still together (before polishing off the substrate).



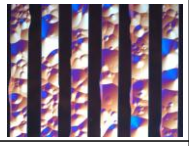
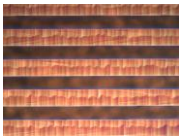
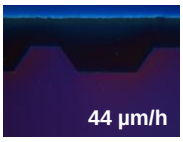
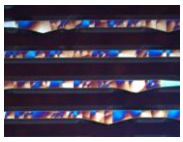
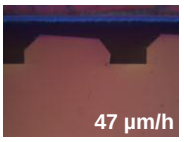
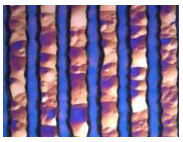
(a) (b)

Figure 4. IR absorption of GaP at different carrier concentration (a). IR absorption of "the HVPE layer + the substrate" and only of the HVPE layer, after polishing off the substrate (b)

4.2. Growth on half-patterned GaP templates

We call a template "half-patterned" (HP) when every other strip is masked off with a layer (in this case a thin Si_3N_4 layer), which prohibits the growth on the masked area (upper left corner of Table 1). This approach allows one to observe the behavior of the oppositely oriented domains separately, without their mutual interference.

Table 1. The mesas shape, growth rate and surface morphology during the growth on HP GaP at different substrate and pattern orientation

Half-patterned templates Si_3N_4 / GaP	100 GaP		100 GaP 4 iscut to ards 111	
	Cross section	op surface	Cross section	op surface
Stripes alon 01 $\bar{1}$	 47 µm/h		 78 µm/h	
Stripes alon 011	 44 µm/h		 47 µm/h	

The experiments conducted on HP templates with stripes oriented along [01$\bar{1}$] indicated that at certain growth conditions (moderate temperature and supersaturation) the mesas had rectangular shapes bounded by {110} and {100} planes. The growth on 4° misoriented samples was faster, 78 µm/h, than the growth on (100) GaP, 47 µm/h. The mesa shapes when the stripes were oriented along [011] exhibited different faceting. The (100) oriented wafers formed a trapezoid bounded by {111} and {100} planes. The mesas on 4° misoriented wafers had a more complex shape involving facets that form a 45° angle with the (100) surface, which is more likely related to a stepped structure consisting of (111) steps and (100) terraces. The growth rate for the 4° misoriented sample was again higher, 47 µm/h versus 44 µm/h for the (100) GaP. This reveals the important detail that the growth rates for the domains with the opposite crystallographic orientations are almost equal, when the pattern is directed along [011], while the rates for the pair oriented along [01$\bar{1}$] are significantly different. However, again, the fastest growth was achieved in patterns with stripes oriented along [01$\bar{1}$]. Moreover, this is the only miscut and pattern direction, in which the growth could be even hillock-free (upper right corner of Fig. 4). In this case, when the growth continued as long as 8 hour and resulted in a 470 µm thick HP structure, the mesa's still had rectangular shape and smooth surface morphology.

Although these results were in a good agreement with previously reported data for other related materials [24, 25], they did not answered unambiguously the question which is the best direction for patterning of the OP templates. The results were used as a feedback and significantly contributed to the improvement of OP template preparation process. The thick growth on HP templates which maintained rectangular shape of the domains and smooth top surface morphology clearly showed that growth on patterned GaP template can be fast and successful.

4.3. Preparation of OP templates

There are two major OP template preparation techniques, well described in the case of OPGaAs [26]: (1) by an entirely MBE process, where the polarity of the material is changed by the deposition of a thin Ge intermediate layer, and (2) the wafer bonding technique where two wafers with oppositely oriented polarity are fused. The difference in producing OPGaP templates is that, Si is used for the non-polar inversion layer in MBE growth templates and there is no etch stop (such as AlGaAs in the case of OPGaAs), which makes domain fabrication more difficult [20]. Fig. 5 shows schematically the major steps in the wafer boding process.

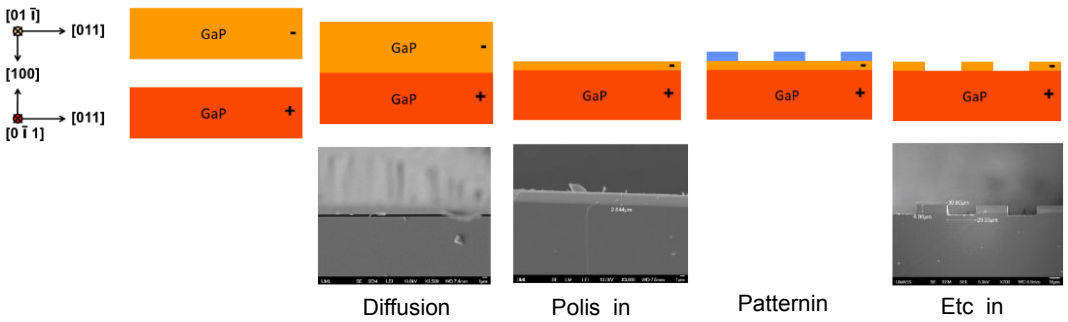
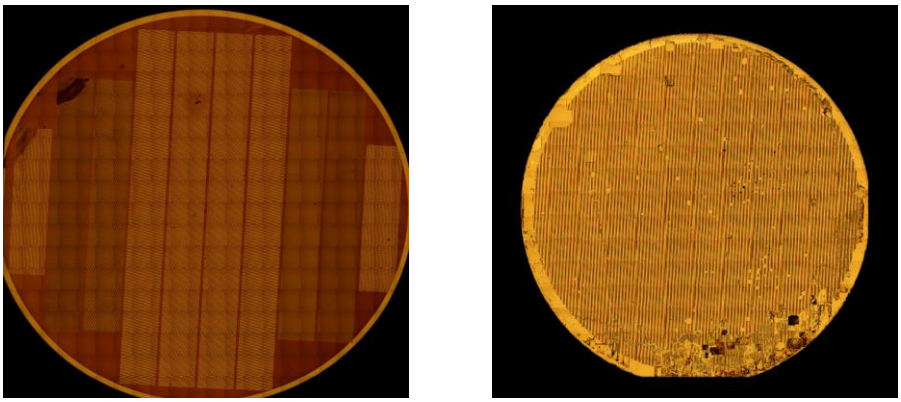


Figure 5. Major steps in preparing OPGaP by the wafer-bonding technique

The development of the techniques for preparation of OP templates (including the implementation of the fly-cut technique) resulted in the production of the first 3-inch MBE assisted and 2-inch wafer bonded OPGaP templates, shown on Fig. 6:

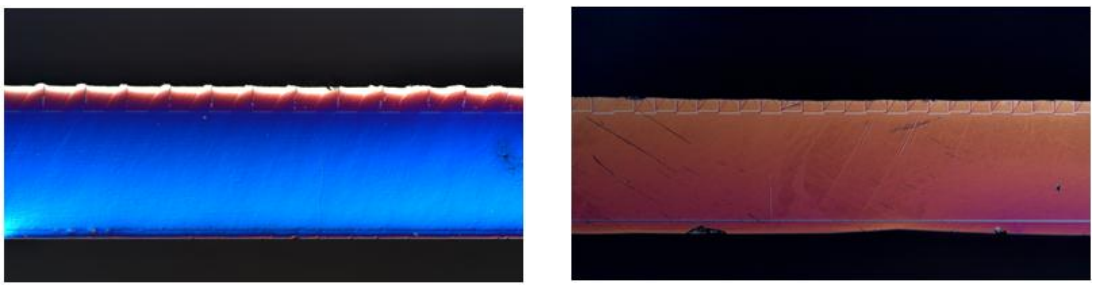


(a) (b)

Figure 6. First 3-inch MBE assisted (a) and 2-inch wafer bonded (b) OPGaP templates, produced at BAE Systems and UML

4.4. Growth on oriented-patterned GaP templates

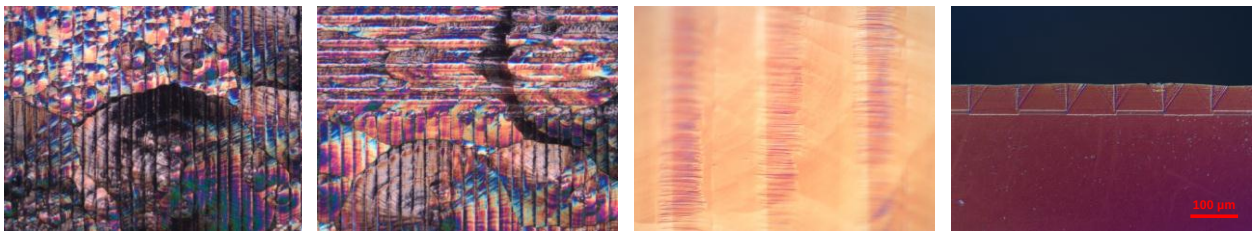
A number of experiments were conducted on smaller OPGaP templates from both types. The growth behavior on the different type templates slightly differs, although the growth on both types is possible.



(a) (b)

Figure 7. Growth on MBE assisted (a) and wafer bonded (b) OPGaP templates

It was found that, to some extent, properly chosen growth conditions can control the hillock growth, flattening the hillocks in the horizontal direction and widening them in vertical direction. Thus sometimes a hillock can span several domains with alternating opposite crystallographic orientations without having any undesirable effect on the pattern (Fig.8-a). A change in the pattern orientation (Fig. 8-b) does not interrupt the growth on the pattern too, although that the domains grow differently. Exactly like in the case of HP templates, the hillock growth is more pronounceable when the domains are oriented along [011] and less when the domains are in the direction [01$\bar{1}$] (Fig. 9-a, b; see also Table 1), when growth on the pattern could be even hillock-free (Fig. 8-c). Thus, the first 350 µm thick device-quality OPGaP was recently grown (Fig. 9).



(a) (b) (c) (d)

Figure 8. Hillock (a, b) and hillock-free (c) growth on OP templates and improved domain fidelity (d)

(a) (b)

Figure 9. Growth on OPGaP templates with stripes oriented along [011] (a) and [01$\bar{1}$] (b)

5. CONCLUSSION AND FUTURE DIRECTIONS

It was demonstrated that HVPE is a successful approach for growth of thick OPGaP structures. Stable growth rates as fast as 50-70 µm/h were achieved on unpatterned and patterned templates in up to 8-hour long growth experiments. IR absorption was found to be strongly dependent on the electron concentration. The 3 µm absorption characteristic of n-type GaP was reduced by minimizing background impurities in the material. Growth on HP templates identified that 4° misoriented towards (111)B (100) GaP wafers with stripes oriented toward [01$\bar{1}$] provide the best pattern transfer into the grown material. The growth on such templates was hillock-free and provided a near perfect rectangular shape of the mesas with the fastest deposition rate of 78 µm/h and the smoothest surface morphology on the top surface of the domains. OPGaP has been grown on both sub-lattice MBE and on wafer fusion bonded templates. The results from growth on OP templates, in general, confirmed the results from the growth on HP templates. OPGaP layers, up to 350 µm thick, were achieved in 6-hour long experiments. By properly choosing the growth conditions the hillocks were widened in horizontal direction and shortened in vertical, which allowed often a single hillock to span several domains with alternating opposite crystallographic orientations without interrupting the pattern.

In order to follow the current trends in the market and requirements this research must gradually include more and more promising materials, growth approaches and template preparation techniques. The coefficients of the nonlinear susceptibility, the width of the IR transparency, the two-photon absorption coefficients, and the thermal conductivity will

be some of the criteria to decide how suitable for nonlinear applications is a given material? The maturity and the diversity of the growth and template preparation techniques will be also carefully considered. For example, the III-Nitrides, more specifically GaN, has already attracted the scientific attention [27] as a promising QPM material, although its nonlinear susceptibility, still comparable to the one of $LiNb0_3$, is fairly small compared to the GaAs and GaP nonlinear susceptibilities. The advantages of GaN are: (i) the better thermal conductivity; (ii) the wider bandgap (iii) established HVPE technology for growth thick layers and (iv) there is a significant progress in the development of selected polarity growth on GaN using MOCVD and MBE for template fabrication. Another material of interest for application in frequency conversion devices is ZnSe [28]. Small ZnSe crystals, spontaneously nucleated from hydrothermal solution of sodium hydroxide, have been recently grown at AFRL.

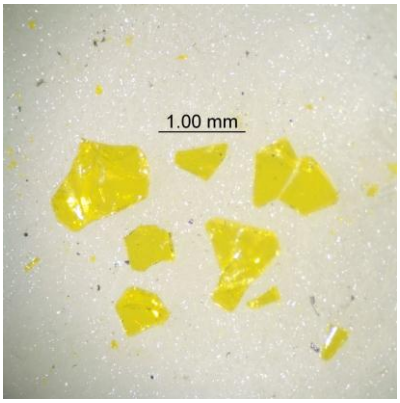


Figure 10. ZnSe crystals spontaneously nucleated from hydrothermal solution of sodium hydroxide

ZnTe is another promising II-VI (similar to ZnSe) candidate, which in point of view of nonlinear coefficients and two-photon absorption coefficient is even better than ZnSe [29].

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