Evaluating focused ion beam patterning for
position-controlled nanowire growth using computer vision

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Abstract. To efficiently evaluate the novel approach of focused ion beam (FIB) direct patterning of substrates for nanowire growth, a reference matrix of hole arrays has been used to study the effect of ion fluence and hole diameter on nanowire growth. Self-catalyzed GaAsSb nanowires were grown using molecular beam epitaxy and studied by scanning electron microscopy (SEM). To ensure an objective analysis, SEM images were analyzed with computer vision to automatically identify nanowires and characterize each array. It is shown that FIB milling parameters can be used to control the nanowire growth. Lower ion fluence and smaller diameter holes result in a higher yield (up to 83\%) of single vertical nanowires, while higher fluence and hole diameter exhibit a regime of multiple nanowires. The catalyst size distribution and placement uniformity of vertical nanowires is best for low-value parameter combinations, indicating how to improve the FIB parameters for position-controlled nanowire growth.

1. Introduction

III-V semiconductor nanowires are a promising material system for the creation of future optoelectronic devices [1]. Using lithography-based patterning of an oxide mask, well-defined nucleation sites are placed at will, often in homogeneous patterns to ensure identical growth conditions. This approach has been successful in achieving large arrays of similar nanowires, but wire-to-wire variations still need to be evaluated [2]. In order to improve nucleation and further reduce variation, direct oxide patterning is expected to be advantageous, allowing for more flexible hole geometry to optimize patterning and nanowire nucleation conditions.

In this work, focused ion beam (FIB) is used to pattern growth substrates for self-catalyzed GaAsSb nanowires grown using molecular beam epitaxy (MBE) [3]. The FIB patterning enables direct patterning of the oxide mask and is more flexible than the lithography based techniques conventionally used. The parameter space to optimize nanowire growth is efficiently explored on a single growth sample. To evaluate the effect of milling conditions on nanowire growth, a sufficient quantity of nanowires need to be characterized in an objective and efficient way. This is especially important for evaluating this rather novel approach to patterning for nanowire growth. By utilizing feature detection techniques from the field of computer vision [4] to automatically detect nanowires from top-down scanning electron microscope (SEM) images, a detailed and objective characterization of the parameter space is achieved.
2. Methods and materials
Self-catalyzed GaAsSb nanowires were grown in a Varian GEN II Modular MBE system [5]. To pattern the substrate for position-controlled nanowire growth, a FEI Helios NanoLab 600 DualBeam FIB was used at 30 kV to mill a growth matrix into a Si(111) wafer with a 40 nm thick SiO$_x$ film (figure 1(a)). The growth matrix consists of 8 × 8 hole arrays with linearly increasing combinations of ion fluence (0.418 – 3.329 $10^{17}$ ions/cm$^2$) and hole diameter (10 – 80 nm). Each hole array contains 270 holes in a hexagonal pattern with 1 µm pitch (figure 1(b)). The sample was cleaned using 1 % HF for 150 s before insertion in the MBE system.

Each array was imaged with 5 kV SEM in the DualBeam (figure 1(c)). Top-down images were then used as input for feature detection, implemented in open source Python libraries. By optimizing the SEM contrast for computer vision, Ga catalyst droplets were identified and used to count and characterize nanowires (figure 2(a)). A lattice based on the FIB-milled pattern, fitted to the detected nanowires, assigns each nanowire to a lattice point corresponding to a FIB-milled hole. The Python code for detection and analysis has been made freely available [6].

![Fluence vs Diameter diagram](image)

Figure 1. (a) Reference design created in FIB: ion fluence - diameter matrix with 64 arrays and supplemental reference fields. (b) Each array consists of 15 × 18 holes. (c) Tilted SEM of an array after growth, with a high yield of single vertical nanowires.

3. Results and discussion
Vertical nanowire growth is observed in all milled arrays, demonstrating the viability of FIB as an alternative patterning technique for nanowire growth substrates. There is a general trend that smaller, shallower (i.e., lower fluence) holes give a high yield of single vertical nanowires per hole (figure 2(a,b)). For wider and deeper holes multiple vertical nanowires are observed per hole (figure 2(d,e)). Between the single and multiple wire regimes, total nanowire yield is lower, dominated by more parasitic growth. This variety demonstrates the necessity of FIB patterning optimization to obtain full growth control among a broad range of possible structures.

The feature detection is consistently able to detect Ga droplets and distinguish them from other features in the SEM image, such as stray Ga droplets on the sample surface (figure 2(a,b)). This allows for automatic detection of nanowires giving a quantitative and objective analysis of how the FIB milling parameter space affects nanowire growth. The maximal yield of single nanowires, 83 %, was observed for two arrays at lower parameter combinations (figure 2(c)). At higher fluence and diameter, array have more variance with several holes containing one or multiple nanowires. The computer vision-based approach ensures correct characterization and classification of growth regimes. For example, a maximal yield of 35 % for two nanowires per hole is found (figure 2(d-f)) in the same array where one of the lowest yields of single nanowires (30 %) is observed. For larger arrays of nanowires, the convenience and reliability of automated over manual characterization becomes more important and ensures reproducibility.
Taking advantage of the breadth of information provided by computer vision on SEM images, mean droplet diameter and displacement from the fitted lattice positions can be evaluated (figure 3(a,b)). The catalyst diameter decreases with increasing ion fluence and hole diameter. This trend can be explained by the additional parasitic growth and multiple nanowires per hole for higher fluence-diameter combinations (figure 2(d)). With constant Ga flux across the sample during MBE growth, the Ga supply per wire decreases with increasing number of droplets.

At the same time, the mean deviation from fitted lattice positions increases with both fluence and diameter (figure 3(b)). Plotting the deviations in scatter plots (figure 3(c), shown for the growth matrix extremes), two distinct effects are identified: First, higher diameter holes consistently have nanowires nucleating further out from lattice centers, indicating that nanowires seem to preferentially nucleate along the hole side walls rather than in the hole center. For wider holes, the increased circumference allows for multiple nucleation sites. Second, deeper (i.e., higher fluence) holes lead to more off-center nucleating nanowires (figure 3(c(i)))) and less radially symmetric displacement. The off-center cluster of nanowire nucleation is believed to be linked to slow ion beam blanking, resulting in an asymmetric hole edge (visible in figure 2(b), blanking lines visible in figure 2(e)) leading to an uneven distribution of nanowire nucleation sites within a single hole. This can be remedied by the use of a faster beam blanker or alternative scan strategies when deeper holes are desired.

Figure 2. (a) Detected droplets overlaid on SEM image for low parameter combination array. (b) Tilted-view SEM image of single nanowire regime, from frame in (a). (c) Single nanowire yield plot across the growth matrix. (d) Detected droplets on SEM image for higher parameter combination. (e) Tilted-view SEM image of multiple-nanowire regime, from frame in (d). (f) Yield plot for two nanowires per hole across the growth matrix.
As droplet size and contact angle has been shown to influence both crystallinity and composition [7], the variation in yield, droplet size, and effective Ga/As ratio across the growth matrix is expected to influence the GaAsSb nanowire composition and optoelectronic properties [5]. To further investigate this, micro-photoluminescence spectroscopy, electrical probing of single nanowires, and transmission electron microscopy of the nanowire-substrate interface should be performed for the different arrays and correlated to the results from computer vision-based studies. In this way the trends in growth results can be linked to FIB patterning parameters to systematically study and achieve the optimal properties for nanowire-based devices.

4. Conclusion
FIB milling has been systematically studied as a promising and flexible direct patterning method for self-catalyzed nanowire growth substrates. Computer vision was successfully applied to detect Ga droplets on top of vertical nanowires for a substantial number of holes (17280 holes across 64 different arrays) on a single sample and thereby shed light on how the FIB milling parameters affect nanowire growth. The ion fluence and hole diameter were found to affect vertical nanowire yield, number of nanowires per hole, droplet size distribution, and nanowire displacement from patterned lattice position. These nanowire characteristics can thus be correlated and optimized in future growth trials.

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References
[1] Joyce H J et al. 2011 *Progress in Quantum Electronics* 35 23–75
[2] Nilsen J S, Reinertsen J F, Mosberg A, Fauske V T, Munshi A M, Dheeraj D L, Finland B O, Weman H and van Helvoort A T J 2015 *Journal of Physics: Conference Series* 644 012007
[3] Detz H, Križ M, Lancaster S, MacFarland D, Schinnerl M, Zederbauer T, Andrews A M, Schrenk W and Strasser G 2017 *Journal of Vacuum Science & Technology B* 35 011803
[4] Lindeberg T 1998 *International Journal of Computer Vision* 30 79–116
[5] Ren D et al. 2016 *Nano Letters* 16 1201–9
[6] NWstats GitHub repository URL https://www.github.com/NWstats/NWstats
[7] Munshi A M, Dheeraj D L, Todorovic J, van Helvoort A T J, Weman H and Finland B O 2013 *Journal of Crystal Growth* 372 163–9