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Nanoporous Films for Epitaxial Growth of Single Crystal Semiconductor Materials:
Final LDRD Report

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Abstract

This senior council Tier 1 LDRD was focused on exploring the use of porous growth masks as a method for defect reduction during heteroepitaxial crystal growth. Initially our goal was to investigate porous silica as a growth mask, however, we expanded the scope of the research to include several other porous growth masks on various size scales, including mesoporous carbon, photolithographically patterned SU-8 and carbonized SU-8 structures. Use of photolithographically defined growth templates represents a new direction, unique in the extensive literature of patterned epitaxial growth, and presents the possibility of providing a single step growth mask. Additional research included investigation of pore viability via electrochemical deposition into high aspect ratio photoresist. This project was a small footprint research effort which, nonetheless, produced significant progress towards both the stated goal as well as unanticipated research directions.

Acknowledgements

Table of Contents

List of Figures

Nanoporous Films for Epitaxial Growth of Single Crystal Semiconductor Materials: Final LDRD Report

Executive Summary

The purpose of this LDRD is to develop new nanoporous templating materials for heteroepitaxial growth of single crystal GaN on patterned substrates to reduce defect density, yielding higher quality material. Defect reduction strategies for Group-III Nitride semiconductors are of immense technological importance, evidenced by the huge capital investment devoted to solving this crucial problem. The performance of III-N based devices is limited by high defect density material. Current research has shown that growth on patterned substrates can significantly reduce defect density. The well understood kinetics of bulk crystal growth are modified when the crystal growth occurs in a confined space. Lateral epitaxial overgrowth (LEO) techniques achieve partial success at addressing this difficult problem under the demanding conditions of III-N growth, but the added expense, time and complexity of the process are recognized deficiencies. Our approach is to use non-conventional patterning techniques to combine self-assembly and top-down photolithography (interferometric lithography) to devise hierarchical carbon growth templates with pattern features in the nanometer, sub-micron and micrometer range to allow multiple length scales of defect filtering and termination of dislocations. It is worth noting that this size scale stretches below that easily obtainable using standard semiconductor industry patterning techniques. At the same time, our approach requires fewer process steps and avoids high energy reactive ion etching which are typical of LEO defect reduction approaches.

Significant results obtained during the course of this project include:

- **Optimized high aspect ratio interferometric lithography**. Used interferometric lithography to generate high aspect ratio 2-D and 3-D structures in photoresist. We have demonstrated deep sub-micron structures to 20 micron thick films with 20:1 aspect ratio holes and 3-D fcc photoresist structures. These structures are the starting material for several technical areas of interest to Sandia such as photonics, metamaterials, catalysis, intracellular probes, energy storage, and chem/bio sensors.
- **Successfully demonstrated the use of galvanic plating to fill these templates, yielding high aspect ratio metallic rods.** Resist patterns were filled using galvanic plating. The result was periodic metal nanorod arrays, offering the possibility to replace time consuming LIGA generated photonic patterns.
- **Demonstrated conversion of high aspect ratio resist structures to carbon**. These high aspect ratio resist structures can be converted to carbon via pyrolysis. Although there is a significant amount of shrinkage, the patterns maintain their geometry and adhesion to the substrate. The result is the capability to produce carbon structures with sub-micron periodicity and high aspect ratio with application in energy storage, electrochemical applications, and to study the optical properties of these wavelength scale structures.

- **First attempt to use photolithographically defined carbon structures as a growth mask for patterned heteroepitaxy of GaN.** The carbon growth mask successfully survived the GaN nucleation temperature (~ 750 °C) in the MOCVD reactor.
- **Developed a soft self-assembly technique to synthesize nanoporous carbon template materials that are stable at high temperature.** Electrochemical characterization and nitrogen sorption isotherm indicated the substrates are accessible to both liquid and vapor phase for further crystal growth. These materials exhibit ideal high surface area for water purification and energy storage.

We continue to make progress towards fabrication of a hierarchical carbon growth mask for heteroepitaxial growth of GaN on sapphire. We will pursue higher pyrolysis temperatures and possible chemical modification of the carbon mask to produce a template capable of surviving the GaN growth conditions and yielding a highly selective growth template. We have followed several out-of-the-box approaches under this project, and the inevitable corridor effect continues to present further attractive research directions. We feel the results attained toward the stated purpose and the glimpse at further research exemplifies the role of small footprint LDRD projects such as this one.

1. Introduction

High quality heteroepitaxy, or crystal growth of one material system on a substrate of another material system, is a long sought goal which will continue to increase in importance. GaN and AlN, important wide bandgap semiconductors as UV light sources and RF electronics materials, are typically grown on sapphire or SiC due to the lack of suitable bulk substrates. In addition, there is considerable interest in growing opto-electronic material systems on Silicon substrates to incorporate and leverage mature silicon electronics processing with optical materials. The inevitable threading defects which result from the lattice mismatch in growing heteroepitaxially degrade both the optical and electrical performance of semiconductors.

Current research has shown that growth on patterned substrates can significantly reduce defect density. Cantilever epitaxy and nano-heteroepitaxy (NHE), techniques with features on the micrometer and 100s of nanometer scales have demonstrated >100X reduction in defect density. The NHE theory predicts that features in the ~10-20nm range could offer defect free heteroepitaxy in many important growth systems, however, patterning a substrate over large areas at these dimensions is not possible with current lithography techniques.

This work resulted as a follow-on project to LDRD 99405 where the initial proposal focused on using nanoporous surfactant templated silica as a growth mask to achieve the benefits of nano-heteroepitaxy. While we have done some preliminary work on this material system, we expanded the scope of the research to a broader range of growth masks including meso-porous carbon and the UV curable photo-epoxy SU-8. Although we discovered SU-8 is incapable of surviving the growth conditions typical in high quality GaN growth, we decided to pursue carbonization of the photoresist patterns to yield the template for growth. In contrast to conventional approaches to nano-heteroepitaxy where an amorphous growth mask is deposited as a planar film, patterned using one of the available lithography approaches, and transferred to the substrate by

plasma etching of the growth mask, all of the approaches explored in this work are single step patterning solutions. As such, these "soft patterning" approaches require no energetic plasma etching of the amorphous layer, and thus avoid potential damage to the underlying single crystal substrate.

This work involved several cross-disciplinary steps, including collaboration on both the crystal growth and electrochemical deposition. In both instances, achieving consensus between investigators on acceptable film durability and more importantly potential contamination of the growth reactor dictated a methical approach to the research. Patterned with interferometric lithography (IL), dense arrays of 2-D holes with diameter ~100nm are readily achievable. SU-8 patterns were chosen as pilot systems to explore the aspects of crystal growth and electrodeposition, and establish the necessary protocols for collaborating across disciplines before migrating to the more challenging nano-porous films. Furthermore, if successful, development of a one-step SU-8 growth mask would have tremendous impact in heteroepitaxy research.

2. Results and Discussion

2.1 High Aspect Ratio Patterning

One significant accomplishment under this LDRD was the addition of high aspect ratio patterning to our capabilities using interferometric lithography (IL). In IL, coherent plane waves are combined to generate an interference pattern which is then used to expose photoresist. The resulting interference pattern exists over the entire volume throughout which the beams overlap, which can be ~ cms. Because no mask is involved there are no diffraction limits on the pattern generated hence IL is capable of producing submicrometer scale features throughout the volume. Furthermore, the geometry of the pattern can be controlled by changing the angle of incidence and number of plane waves used to generate the pattern, making 1-D, 2-D and complex 3-D patterns possible. Figure 1 shows some example SEM images of high aspect ratio 2-D structures. In this case, a prism was used to generate the interfering plane waves. The sample was created with two separate exposures with a 90 rotation in between exposures so that the resulting pattern has a rectangular symmetry when view from above. The tilted 2-D holes shown in Figure 1c. were created by placing the substrate at a angle with respect to the plane of incidence of the interfering plane waves.
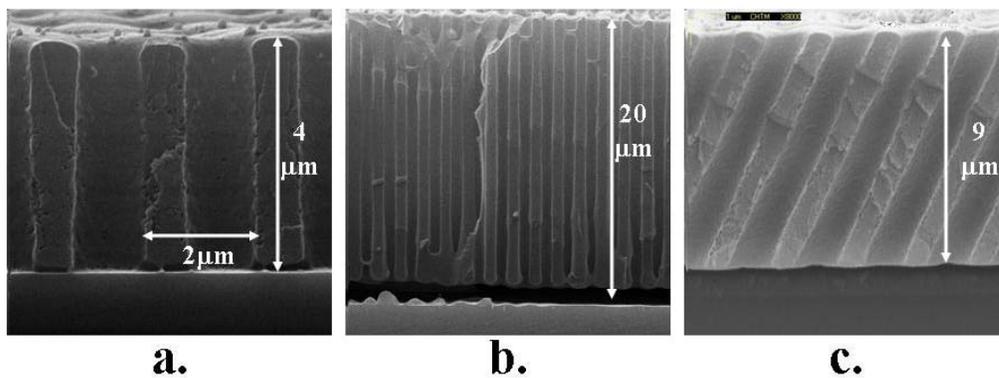
Figure 1. High aspect ratio photoresist structures created with interferometric lithography. a.) Cross section SEM image of a 2-D rectangular array of holes with aspect ratio of 4 in 4 micrometer thick resist. b.) Cross section SEM image of a 2-D rectangular array of holes with aspect ratio of ~ 20 in 20 micrometer thick resist. c.) Cross section SEM image of a tilted 2-D array of holes with aspect ratio ~ 9 in 9 micrometer thick resist.

Figure 2 shows some examples of 3-D patterns generated with three exposures. The fill factor, or ratio between the polymer and air in the pattern is adjusted by changing the exposure dose, post exposure bake and development parameters. In these images the pattern is nominally that of a face centered cubic geometry.
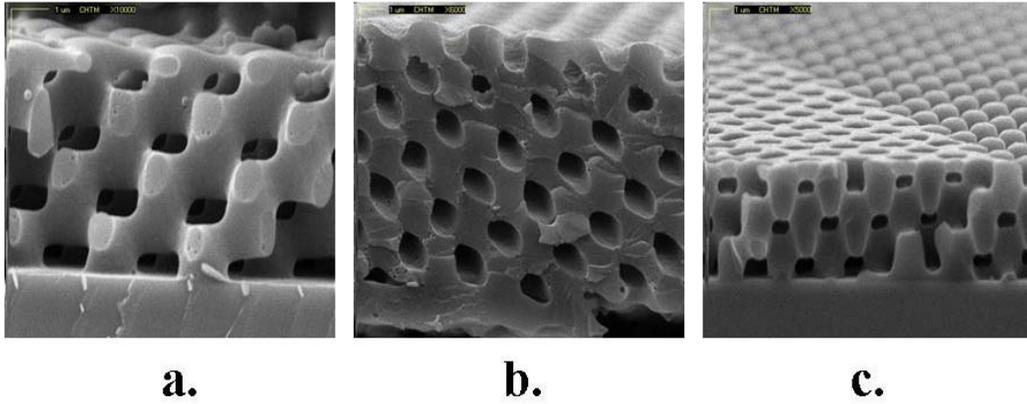

Figure 2 Cross sectional SEM images of 3-D patterns in photoresist created with interferometric lithography.

2.2 Galvanic Plating

Electrochemical deposition into patterned films was originally envisioned as a metric of pore viability, and was a milestone in the original proposal. We established a collaboration with 1725 photonics microsystems and electroforming to perform electrochemical deposition into our films. 1725 routinely electroplates into patterned resist structures with dimensions from micrometers to 100's of micrometers, and hence plating into structures with dimensions ~10's nanometers presented a challenge. Furthermore, the plating bath operates at a pH of ~9.5, a potential problem for silica based films. In order to establish the appropriate sample preparation, coating and plating parameters, we decided to use a piece-wise approach to plating, pursuing plating in "larger," photo-lithographically generated patterns, with the ultimate goal of moving to mesoporous silica structures.

A galvanic gold plating bath was chosen due to its familiarity and ease of use. In a typical plating operation, a non-conducting substrate (in this case a cleaved microscope slide) is coated with a Ti/Au/Ti metal stack to act as a seed layer for plating. The first Ti layer (~100 Å) serves as an adhesion layer for the subsequent Au layer (~2000 Å). The final Ti layer (~100 Å) serves as a protection layer for the top of the gold surface. Although gold is inert, prior experience on photonic bandgap structures showed that covering the gold with another Ti layer which can be etched out after patterning, but prior to the electroplating step improves the interface between the between the seed layer gold and electroplated gold.

In a testament to its versatility, interferometric lithography (IL) was chosen as the lithography approach. Whereas the patterns generated in the crystal growth section of this research were in thin resist (<300 nm) IL was used here to generate high aspect ratio holes in thick negative resist (~5 micrometers). One choice impacting the research was selection of the proper photoresist. In many applications, it is desirable to remove the resist template after electroplating. With typical commercial resists, this can be performed in a simple acetone soak. In contrast, sometimes resist removal can be difficult with fully cross-linked SU-8 (the same photo-epoxy used in the crystal growth section). However, SU-8 is has excellent mechanical properties, and in particular is resistant to basic solutions, while typical i-Line photoresist is developed in a basic solution, possibly leading to template instability in the high pH plating bath. In order to determine the best approach, samples with SU-8 as well as a commercial i-Line negative resist, NR1-3000PY (Futurrex) were prepared and plated into.

Microscope slides were cleaved into 1 inch squares, cleaned in piranha (2:1 H_2SO_4:H_2O_2), and coated with a Ti/Au/Ti metal stack in an e-beam evaporator. The samples were then coated with photoresist (either SU-8 or NR1) and soft baked. The coated samples were then exposed using the same IL setup as described in section I, post exposure baked and developed. At this point, the samples were then flood exposed and hard baked in order to increase the resiliency of the templates. Prior to plating, the samples were immersed in dilute HF (1:100, HF:H_2O) to etch out the Ti protecting layer. An electrode was then connected to a corner of the sample and the sample was placed in the plating bath.

The initial plating run used a current density of 5mA/cm2. For this initial run, the NR1 resist sample suffered almost immediate adhesion issues, with delamination and peeling. Figure 3 shows a cross section SEM photograph clearly indicating some filled
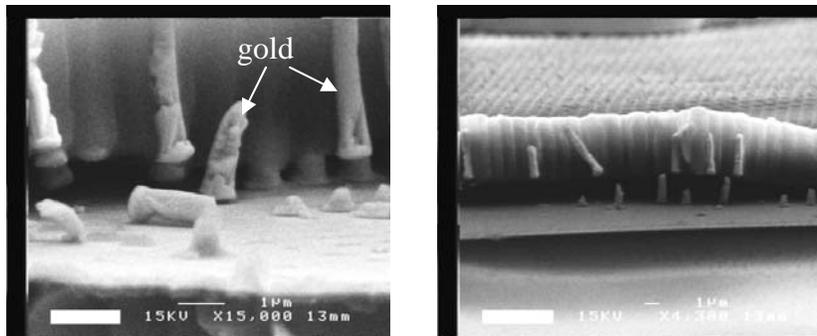
Figure 3. SEM images of filled NR-1 high aspect ratio holes before acetone removal of resist. After resist removal, no posts remained on substrate

pores, however after cleaning, none of the deposited posts survived. Because the NR1 system obviously requires more attention in order to optimize the exposure/post-treatment regime, we decided not to pursue further plating into these samples at this time. The SU-8 sample did not suffer from this delamination problem, however the sample surface appeared to undergo severe non-uniform plating, resulting in large globules of gold being deposited on the surface of the resist. Real time monitoring of the plating

process revealed the problem and plating was terminated. Figure 4 shows some high magnification optical images of some of the features which appeared during plating. SEM photographs (Figure 5) were taken before and after soaking the SU-8 samples in
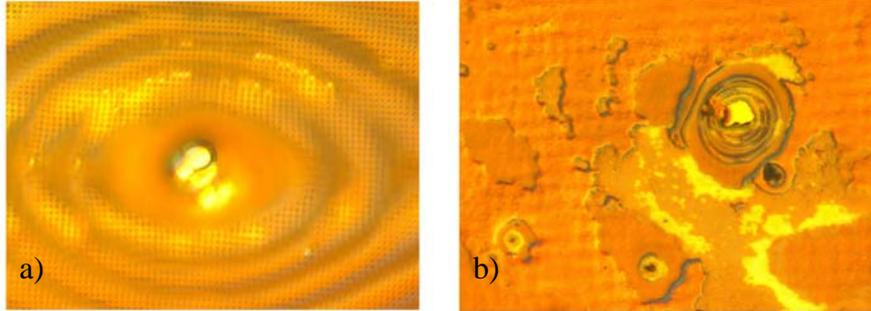
Figure 4. Optical images taken after plating at a current density of 5 mA/cm^2. a.) shows a large (>50μm) gold globule; b.) shows obvious areas of film non-uniformity.

piranha. These photographs demonstrate some of the same features which indicated that the plating failed, however they also show that a fair amount of holes were successfully filled with electrodeposited gold.

The obvious scalloping at the edge of the posts is a direct replication of the standing wave pattern in the resist. Because laser light is used to pattern the resist, and no
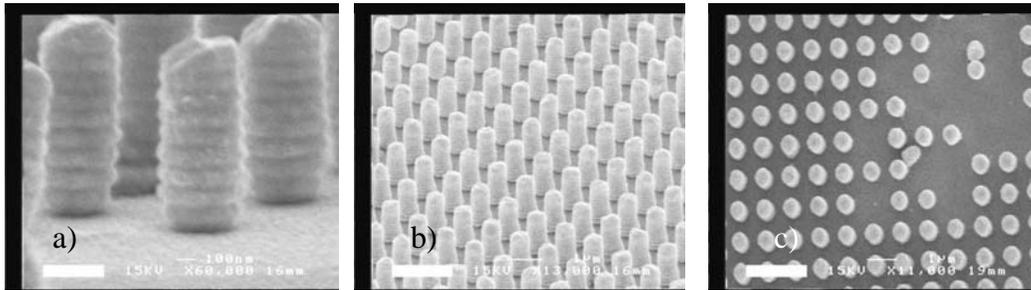
Figure 5. a.) Cross section SEM image of gold posts plated into SU-8 template at 5mA/cm2; b.) Lower magnification 45 degree angle SEM image of posts; c.) Top down SEM image of gold posts.

ARC layer is used, a standing wave forms due to reflection off the metallic substrate. This standing wave exposes the resist with a periodic modulation about the edges of the holes with period $\lambda/2n$. Appearance of a standing wave is most prominent when operating right near the exposure threshold of the resist, and can be controlled by applying a DC exposure.

Although 5mA/cm^2 is the lower limit for controlled current of the plating bath instrumentation, a second run using an SU-8 coated sample was performed in a sub-optimally controlled run with the current set at ~1mA/cm^2. This sample appeared to have no evidence of the uncontrolled globule deposition on the surface when viewed through the optical microscope. Upon cleaning the sample in piranha, large areas, perhaps as much as one quarter of the sample, were covered with high aspect ratio posts such as those in Figure 6. The surface features occurring periodically between the posts are the

unetched remains of the Ti protection layer. That the Ti layer was so completely etched is an indication that the Ti pre-etch step was carried out for too long. This would have
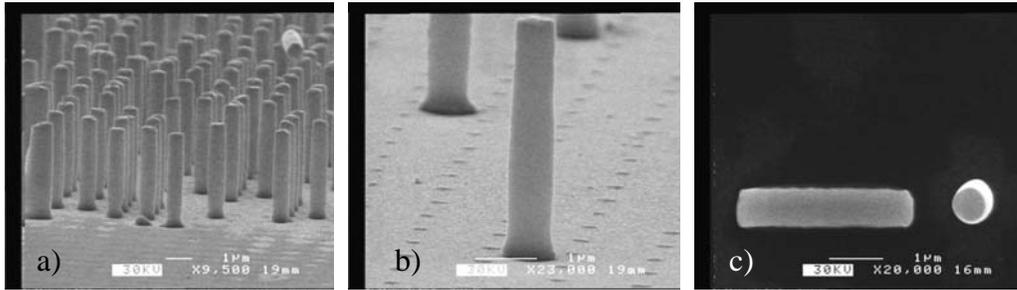
**Figure 6**. a. Low magnification SEM image of array of gold posts. Aspect ratio ~5; Higher magnification SEM image showing single post; c.) Top down SEM image showing one standing post and one fallen post.

undercut the resist template, lessening adhesion, and will be optimized in future attempts to shorter etch times.

The posts are approximately 3.5 micrometers tall, and 0.5 micrometers in diameter. The period is ~1 micrometer. Some of the posts appear to have a "foot" at the bottom, which is to be expected as the null of the standing wave pattern exists at the metal/resist surface. However the extent of the standing wave effect is largely removed by a DC exposure to assist in the hardening of the resist. Many of the posts appear to have been successfully formed, but subsequently fell over, indicating that there is some sort of adhesion issue. Indeed some of the standing posts seem to have a clear undercut interface between the post and seed layer. Further work will explore whether this is due to a material mismatch between the seed layer and electroformed gold.

It is important to note that all the results obtained here were achieved in a single iteration between 1815 and 1725. No attempt was made to push the limits of the lithography from an aspect ratio or pattern density standpoint, and the entire plating process including pre-etch, current density choice, and plating duration were made as game time decisions. As such, we can expect improvement in each of these areas as we optimize the process. In any event, plating into these photolithographically defined holes was a valuable intermediate step in the learning process toward the ultimate goal of plating into nanoscale pores.

The ability to rapidly form large area arrays of periodic, high aspect ratio patterns is of extreme interest to the photonics and Microsystems community. Currently 1725 uses LIGA to pattern thick resist in order to form photonic bandgap structures in the near to mid IR spectral range. While able to generate arbitrary patterns with the requisite aspect ratio, LIGA is a slow and expensive process. The high aspect ratio patterning results from above indicate, with optimization and process work, interferometric lithography may offer a streamlined, in-house alternative to LIGA.

13

2.3 Carbonized 2-D and 3-D Structures

Previously we determined that SU-8 structures are incapable of surviving under typical GaN nucleation conditions with temperature ~ 530 °C. In order to maintain the
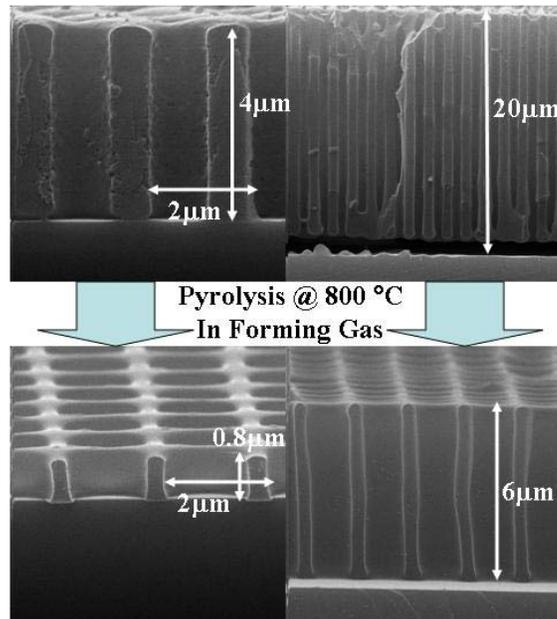

Figure 7. SEM images of 2-D resist structures before (top row) and after (bottom row) pyrolysis at 800 °C.
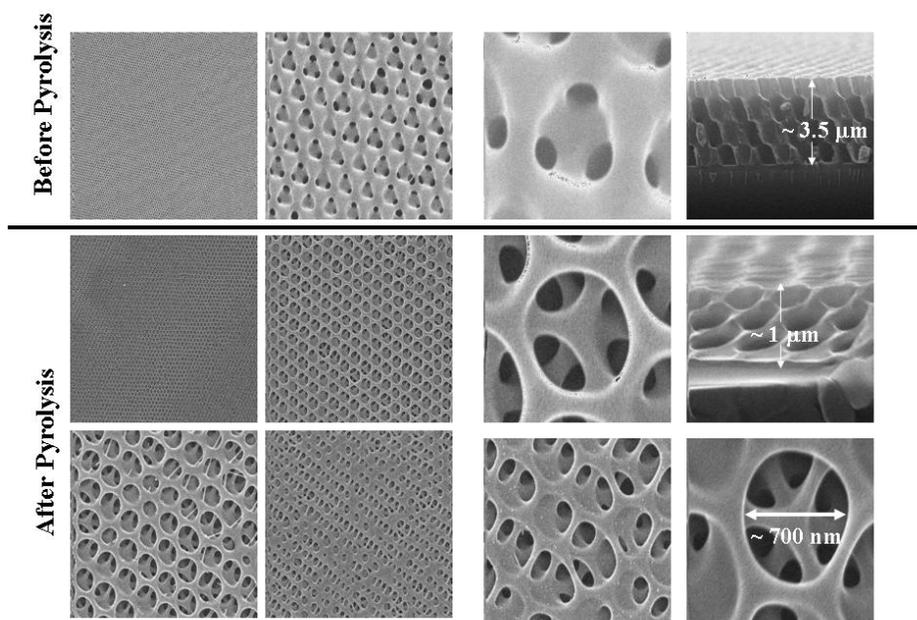

Figure 8. SEM images of 3-D resist structures before (top row) and after (bottom 2 rows) pyrolysis at 800 °C in forming gas.

advantages of a lithographically defined growth mask, but increase survivability, we pursued a strategy of converting the photoresist structures to carbon prior to growth. Following published reports [1] of pyrolyzed photoresist, we placed photoresist structures in a tube furnace with flowing forming gas (N_2:H_2 95%:5%) and elevated the temperature to 800 °C at 2 °C/min, held for an hour and then ramped down to room temperature at a similar rate. Figure 7 contains SEM images of several representative 2-D patterns both before and after pyrolysis. Significant shrinkage is apparent from these samples. Shrinkage in the vertical direction is more dramatic and obvious than the horizontal direction, however, it is apparent that the "walls" of photoresist are thinner after pyrolysis in both the 2-D. In the images on the left of Figure 7, the magnification is constant (both show approximately 3 periods of the structure.) While the resist shrinks in both the vertical and horizontal directions, the patterns maintain their geometry and adhesion to the substrate, exhibiting no obvious cracking or flaking. Figure 8 contains SEM images of a 3-D pattern both before and after pyrolysis. Once again, the film shrinks significantly in all dimensions with the photoresist necks in the pictures are drastically thinner in the comparable carbon structure.

2.4 Hydrogen Bonding Assisted Self Assembly of Oriented Mesoporous Carbon

Fabrication of templates with lateral dimensions in the 10-20 nm range is beyond the limit of conventional and interferometric lithography. Our approach is to attempt to create templates using self-assembly in order to reach these dimensions. Nanostructured carbon materials, including carbon nanotubes, membranes, and particles, are an important nanomaterial for many applications in nanoelectronics, sorption and separation, sensors, catalysis, and energy conversion and storage (e.g., double layer capacitors, and hydrogen storage). Varied methods have been developed to synthesize nanostructured carbon materials, such as DC arc-discharge, laser ablation, chemical vapor deposition, templating, etc. However, direct fabrication of complex carbon nanostructures with controlled form (e.g., film, particle, tube, etc.), dimension, and surface architecture remains a significant challenge.

Using a self-assembling solution, we developed a simple and direct wet chemistry method to simultaneously synthesize nanoporous carbon films and particles by combining hydrogen bonding assisted self-assembly of PS-P4VP and carbohydrate precursors by spin-coating and aerosol processing [2,3]. These nanostructured carbon materials exhibit large nanopore (> 20 nm), controlled 1–3 dimensional structures, and surface chemistry. It is our intention to use these carbon templates to realize the defect reduction this size pattern is supposed to provide.

In a typical preparation carbohydrate precursors such as turanose, raffinose, glucose, etc. were added to dimethyl formamide (DMF) for sonication. After the addition of polystyrene-copoly-(4-vinylpyridine) (PS-P4VP), the carbohydrate precursors quickly dissolved in DMF. We believe that this increased dissolution is caused by the formation of hydrogen bonds between the carbohydrates and pyridine blocks, which promotes the dissolution of carbohydrate precursors in DMF and helps explain why the carbohydrates remain exclusively in PVP domains, enhancing the self-assembly process. During spin-

coating, evaporation of DMF progressively enriches the concentrations of the non-volatile constituents including carbohydrates and PS-P4VP on the substrates. Drying induces microphase separation and formation of ordered PS-PVP/carbohydrate composite films. After spin coating and subsequent thermal treatment under Ar, the polystyrene fragments are removed and the carbohydrate/PVP converts to carbon, resulting in nanostructured carbon films. Figure 9 contains SEM and TEM images of these porous structures.
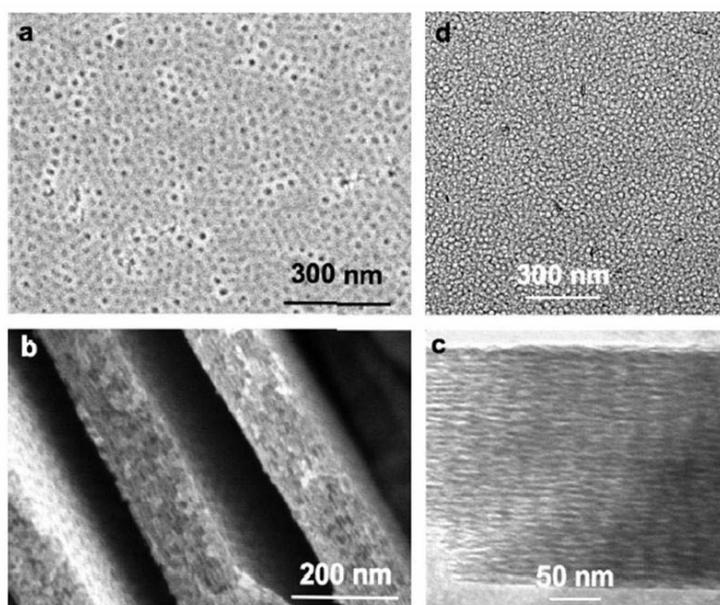

Figure 9. Representative plan-view and cross-sectional view scanning electron microscopy (SEM, a and b) and transmission electron microscopy (TEM, c and d) images of 3-dimensional mesoporous carbon films. Image b shows cross-sectional view of three individual films. These films were prepared by using a homogenous DMF solution containing 8.7 wt% turanose and 4.3 wt% PS-P4VP (polymer source, MPVP

2.5 GaN Crystal Growth Results

The majority of the work performed under this project involved fabrication of templates capable of surviving the harsh growth conditions necessary for GaN growth. However we did perform a few growth runs on various growth templates. In the first attempt GaN was grown on a sapphire substrate with a mesoporous carbon film as the growth mask. In this case, a sapphire wafer was spin coated with a block copolymer/carbohydrate solution. After pyrolysis at 800 °C, the copolymer is removed, and the carbohydrate is converted

16

into amorphous carbon, yielding a mesoporous carbon film. This sapphire wafer was then placed in the growth chamber of the GaN MOCVD system for a typical two step GaN growth process. After heating to 530 °C, a 20 nm thick GaN nucleation layer was grown followed by heating in NH_3, H_2, and N_2 to the growth temperature at 1050 °C. After heating the Ga source was reintroduced and GaN was grown for 45 min. Poor surface morphology was immediately evident upon removal of the substrate from the growth chamber. The deposited film was soft, smearing on contact, and polycrystalline in nature. Due to the obvious shortcomings of this growth, no further analysis was performed on this sample. This result demonstrated the some of the issues of growth in such highly confined structures, encouraging us to pursue larger structures in order to understand the subtle growth issues, and then fine tune our process to culminate in growth in mesoporous substrates.

We also made one attempt at growth in lithographically defined carbonized structures. In this case a 2-D array of holes was exposed in 4 μm thick SU-8 with a rectangular pattern and period 2 μm. After pyrolysis at 800 °C in forming gas, the pattern shrunk to ~0.8 μm thick, but maintained the 2 μm period. Two growth runs were attempted on these patterns. In the first growth run, the temperature was elevated to the nucleation temperature of 530 °C, where 20 nm of GaN were deposited and then the growth run
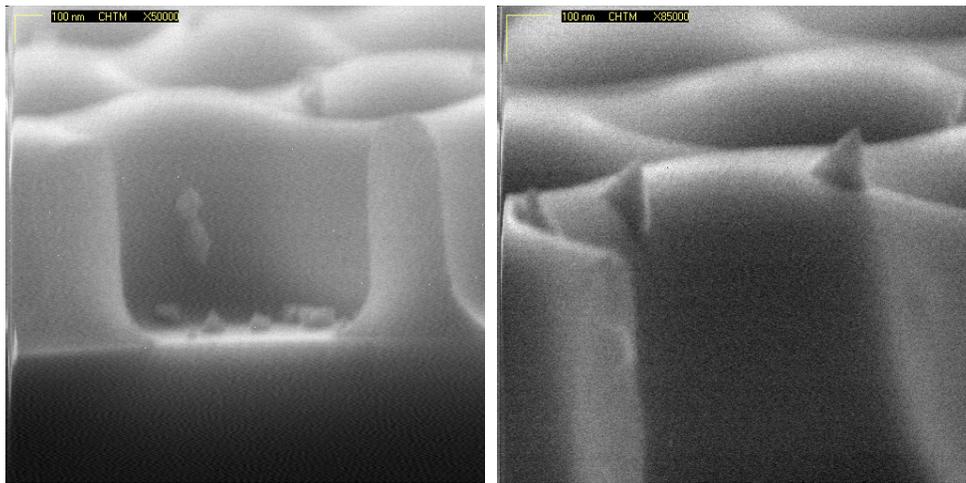

Figure 10. SEM images of carbonized growth template after 20nm thick nucleation layer was grown. Growth evident both on the sapphire substrate as well as the carbon growth mask.

terminated. Figure 10 shows two SEM images from this growth run. Clearly the growth template has survived, and some nucleation is evident on the sapphire substrate. There is also growth on the template, indicating that mask selectivity will be an issue.
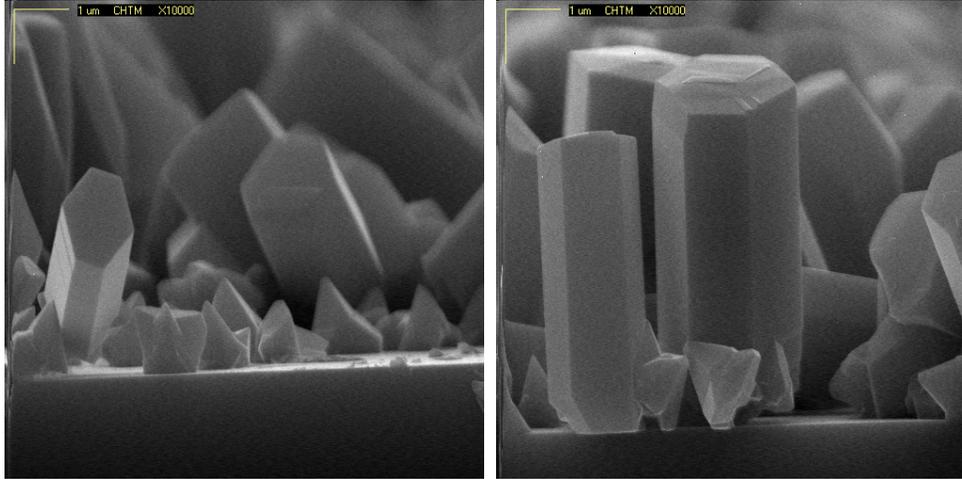
Figure 11. SEM images of carbonized growth template after 1050 C GaN growth. Template did not survive, and resulting GaN was polycrystalline.

For the second growth run, the sample was taken through the nucleation step above, followed by a subsequent growth step at the ultimate growth temperature of 1050 °C. At this temperature, the growth template did not survive, and the resulting GaN was polycrystalline as seen in the SEM image of Figure Blah.

3. Conclusion

High quality heteroepitaxial crystal growth is a high value capability, with strategic impact in defense, solid state lighting, optoelectronic and RF electronic applications. Some of the out-of-the-box approaches pursued in this Senior Council LDRD, as well as an out-of-the-box LDRD which followed preceded this research, take a different tack on this challenging problem: using self assembled and photolithographically defined structures to provide the nanoporous growth masks which are predicted to yield lower defect density epitaxial material. As one always hopes when conducting research toward a specific goal, the corridor effect played a large role in this research effort. The late-start nature of the project forced a do-or-die angle on all the experiment sets, reducing the time and number of experimental iterations, thus making every experiment "high risk." The result is that a collection of best-guess, first-of-its-kind experiments has led to several new potential research thrusts.

We will continue to pursue the primary goal of this research, nanoporous templated heteroepitaxy. As we continue to understand the nucleation and growth process in the relatively larger carbonized growth masks, we are better able to conduct research on growth in the much smaller nano-scale pores of self assembled carbon structures. Our future efforts toward this end are to 1) explore various pretreatments to the SU-8 growth masks in order to increase their resiliency under the growth conditions; 2) look into using oriented graphitic carbon mesoporous structures as a growth mask, and 3) use surfactant

templated silica films as growth masks. In addition, we will vigorously pursue the ancillary research thrusts uncovered during the course of this LDRD project.

4. References

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