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Nanoporous Silica Templated HeteroEpitaxy: Final LDRD Report

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Abstract

This one-year out-of-the-box 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, and the UV curable epoxy, SU-8. Use of SU-8 as a growth mask 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 patterns and pilot work on using SU-8 as a DUV negative resist, another significant potential result. While the late start nature of this project pushed some of the initial research goals out of the time table, significant progress was made.

Acknowledgements

This work was performed in part at the Nanoscience @ UNM facility, a member of the National Nanotechnology Infrastructure Network, which is supported by the National Science Foundation (Grant ECS 03-35765). Sandia is multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United Stated Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000. This work was supported under the Sandia LDRD program (Project 99405).

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Executive Summary

The purpose of this LDRD was to investigate nanoporous surfactant templated silica as a growth mask for heterepitaxial growth for Group III-V epitaxial crystal growth. The scope was expanded to include other mesoporous materials such as carbon, as well as using a patterned photocurable epoxy as a direct growth mask. A successful approach to heteroepitaxy will greatly reduce material defect density and hence improve both optical and electrical properties of the epitaxial material. Additionally, such a technology has the potential to enable highly integrated heterogenous Si/III-V optoelectronic applications.

Although the LDRD received late-start funding, significant progress was made on a number of fronts. We successfully electroplated gold into high aspect ratio holes in photoresist patterns yielding large area arrays of gold pillars with aspect ratios of ~5.5. This successful result has tremendous impact for photonic, magnetic and surface chemistry applications, which we plan to pursue aggressively in follow on work. We explored the use of sel-assembled mesoporous carbon, as well as SU-8 patterned with interferometric lithography as a direct growth mask. Although success remains elusive, these initial results are promising and will be pursued further. And finally, we performed pilot work toward using SU-8 as a deep UV negative tone resist (193 nm), which would be a significant addition to the general research community.

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.

The initial proposal of this work focused on using nanoporous surfactant templated silica as a growth mask to achieve the benefits of nano-heteroepitxay. While we have done some preliminary work on this material system, the scope of the research was expanded to a broader range of growth masks including meso-porous carbon and the UV curable photo-epoxy SU-8. 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 methodical approach to the research. Previously, we determined that the photo-epoxy SU-8 acts as a stable film up to at least 700 °C, above the nucleation temperature for GaN growth. This discovery led us to explore using SU-8 directly as a growth mask. 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 discilplines 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. Finally, pilot work toward using SU-8 as a deep UV negative resist is reported. Although designed for i-Line exposure, for films thinner than the absorption length, it may be possible to pattern SU-8 using a DUV source.

The balance of this report details our research into crystal growth on sapphire and silicon wafers with various growth masks, electrodeposition into high aspect ratio photoresist and SU-8 patterns, and patterning SU-8 with a DUV source.

2. Results and Discussion

2.1 Crystal Growth

For 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. Due to hydrophobic/hydrophyllic interactions between the solution constituents, self-assembly occurs on the mesoscale. After pyrolysis at 800 degrees C, the copolymer is removed, and the carbohydrate is converted 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 difficulties of growth in such 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.

In an unrelated research project, our group discovered that the photo-epoxy SU-8, a UV curable photo-epoxy, was stable to >700 °C. Since the nucleation temperature of GaN is ~530 °C, we decided to look into using SU-8 as a direct growth mask. This step represents a significant departure from usual patterned crystal growth approaches, where all patterning is transferred to an inorganic growth mask. This process is usually performed via high energy plasma etching with no dependable method for insuring the substrate crystal is not damaged by the energetic plasma. The ability to use SU-8 as a growth mask eliminates this potentially damaging step, providing a one-step growth mask which preserves the perfect crystallinity of the substrate.

SU-8 is designed for use at i-Line exposure wavelengths. Designed initially as a thick negative resist for use in relatively large structures such as MEMS devices with dimensions in the 100s of micrometers and thickness of 50μm or greater, SU-8 is starting to see use in higher resolution applications, with sub-micrometer dimensions. Interferometric lithography (IL) was used to perform all the SU-8 patterning. Sample substrates were coated with SU-8 using a standard spin coater. For all crystal growth samples, SU-8 2000.5 was used to achieve films with a thickness of ~300 nm, while the electroplating samples were coated with SU-8 2005 providing films with thickness ~5μm.

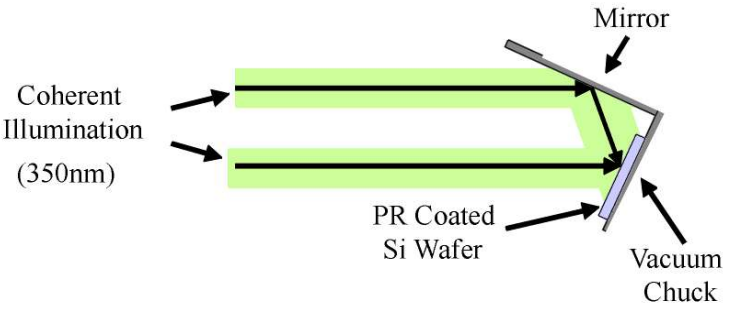

Figure 1. Lloyd mirror configuration for Interferometric lithography.

A tripled Nd:YAG (λ = 355 nm, 9ns pulses) laser was used in a Lloyd mirror configuration (Figure 1) to expose the resist in two exposures with a 90° rotation of the sample, yielding rectangular arrays of holes after post exposure bake and development. The period of the array is set by changing the incident angle of the exposure, and was varied from ~1 μm to ~376nm. Because the sapphire substrate is transparent to the exposure wavelength, the back side surface was coated with a TMAH developable polymer to act

as an anti-reflection (AR) coating. After solvent development of the SU-8, the sapphire substrate was placed in a standard TMAH developer which removed the AR coating.

Figure 2 shows a representative SU-8 resist pattern used as a growth mask. In Figure 2a, a top-down SEM image shows the resulting growth pores to be ~100-150 nm in diameter, while Figure 2b shows a cross-section SEM image of the same structure. The

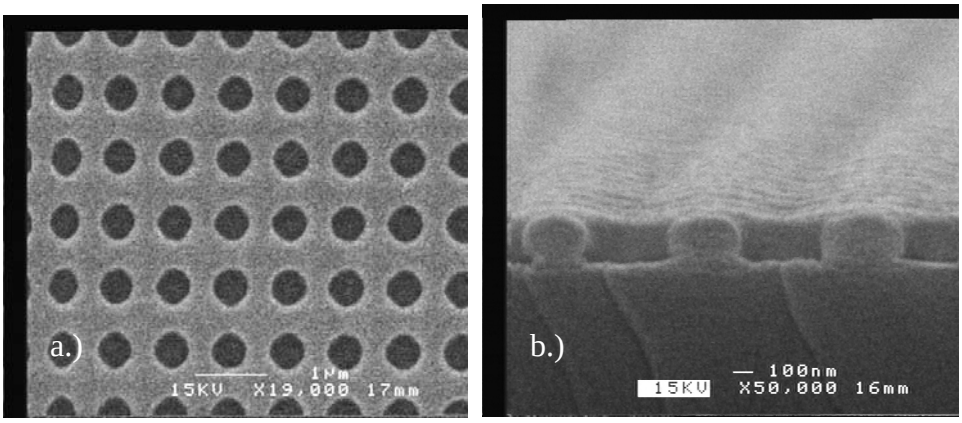

Figure 2. a. Top Down SEM image of SU-8 mask. b. Cross-section SEM Image of SU-8 growth mask.

resist holes extend through the resist to the substrate, providing access for the process gases to reach the single crystal sapphire substrate.

In the next set of growth runs, patterned wafers with both sapphire and (111) silicon substrates as well as an unpatterned control sapphire wafer were placed in the reactor and the temperature was raised to just above the typical nucleation temperature for GaN growth. After growing a standard nucleation layer the temperature was increased to near 700 °C for 45 min. of GaN growth. Both 1-D and 2-D patterns were investigated. After the samples were removed from the reactor it was found the 1-D patterned structures failed visual inspection, exhibiting peeling and delamination. The 2-D patterns had a “matte” finish, but otherwise required more analysis to determine the material

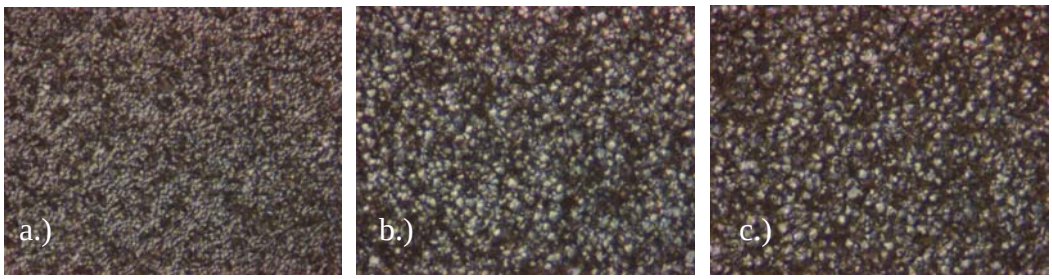

Figure 3. Namarski microscope images of a.) control sapphire wafer, b.) sapphire wafer with patterned SU-8 growth mask, and c.) (111) Silicon wafer patterned with SU-8 growth mask.

quality. While the preliminary low resolution XRD data indicated that the material was indeed GaN, the Namarski microscope photographs in Figure 3 indicate the GaN material consisted of un-coalesced crystallites of GaN. This conclusion is further strengthened by the high resolution SEM pictures in Figure 4.

The first growth run provided several useful results: 1) Due to the limited success of the 1-D structures, we decided to focus on 2-D structures to reduce the parameter space. We will reexamine 1-D structures at a later time. 2.) The growth between the control sapphire and the patterned sapphire were qualitatively the same, while the

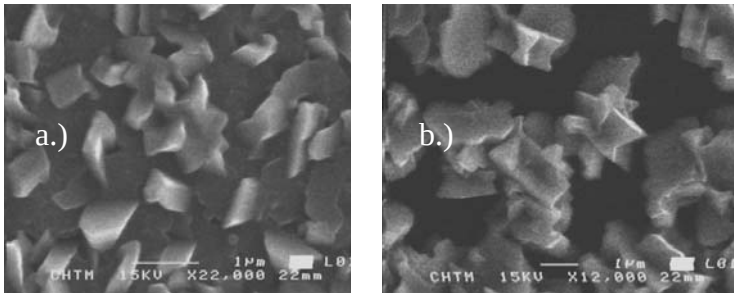

Figure 4. SEM images of a.) Control sapphire wafer showing uncoalesced crystallites and b.) patterned silicon wafer showing similar crystal morphology.

material grown on the silicon wafer was significantly different. This could be due to a variety of factors. For instance, we did not take steps to remove the native oxide on the wafers immediately prior to insertion into the reactor. Furthermore, it is possible some form of silicon nitride formed at the interface during growth. In order to avoid spending time examining these unknowns, we decided to limit immediate growth runs to sapphire to reduce parameter space, putting off exploration of the intricacies of growth on silicon till after we fully understand the impact of patterning the substrate. 3.) The fact that the control sample also exhibited un-coalesced crystallites indicated that there were some issues with the generic growth behavior.

Because the GaN film on the control wafer failed to coalesce, we decided to

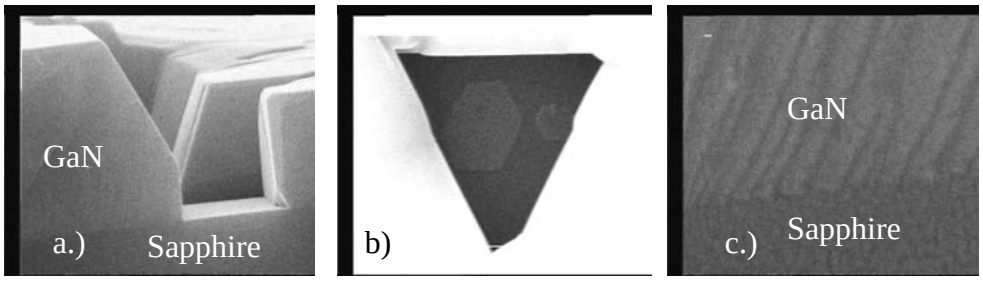

Figure 5. a.) Cross-section SEM of heteroepitaxial GaN; b.) Top-down SEM image of GaN film on top of sapphire, hexagonal nucleation is discernable in void; c.) High magnification cross-section SEM image of the interface between the GaN epitaxial layer and the sapphire substrate. No SU-8 pattern is evident.

perform the next growth run in stages, First the patterned wafer was heated to the nucleation layer growth temperature at 530 °C, the nucleation layer was grown followed by growth at 700 °C for 18 min. and 800 °C for 25 min.. Upon removal from the reactor, the second set of substrates appeared much improved over the first set. The films on both the control and patterned wafers now appeared more specular, and less matte finish.

Figure 5 shows the SEM photographs of the patterned film. This film shows a largely coalesced film, with very obvious crystalline facets. However the high resolution SEM picture of the interface between the sapphire/GaN layers exhibits no evidence of the SU-8 patterning. This led us to speculate that perhaps the SU-8, stable during anneals to ~700 °C under Ar, may be unstable under the ammonia environment of the nucleation conditions.

To investigate this, we performed one more run with a patterned wafer where the wafer was brought up to the nucleation temperature while under standard ammonia flow rate but absent the flow of Ga precursors so that no nucleation or growth occurred. At this point the temperature was lowered and the sample removed. Real time optical reflectance showed a marked change at approximately 400 °C, and upon removal, the sample showed no evidence of patterning, indicating conclusively that the as-deposited SU-8 is not stable in the GaN growth environment. Furthermore, we think the unstable SU-8 film adversely affected the control wafer, compromising the film quality on the control sample as well.

Successful growth using SU-8 as a growth mask remains elusive, however we have several approaches we plan to investigate further. Research indicates that SU-8 can be converted to a glassy-carbon like film when annealed under forming gas. We plan to explore using SU-8 patterns which have been converted to carbon prior to insertion to the growth chamber to see if such films are stable under the ammonia environment during growth. In addition we plan to look into porous graphitic carbon and mesoporous silica growth templates in a follow on task.

2.2 Electrochemical Deposition

Electrochemical deposition into patterned films was originally envisioned as a metric of pore viability, and was a key 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 hardbaked 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 6 shows a cross section SEM photograph clearly indicating some filled

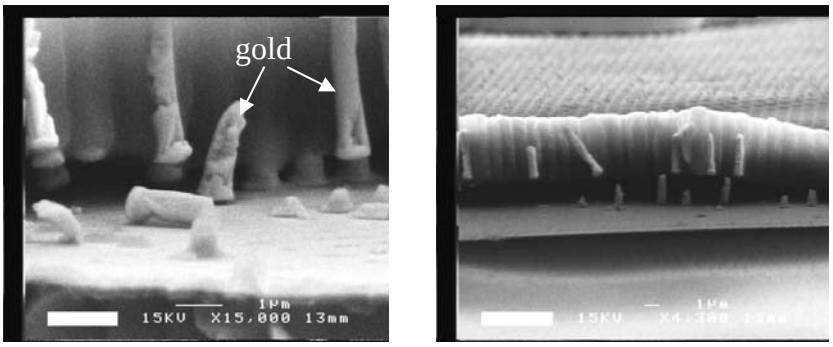

Figure 6. SEM images of filled NR-1 high aspect ratio holes before acetone removal of resist. After resist removal, no posts remained on substrate

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 7 shows some high magnification optical images of some of the features which appeared during plating. SEM photographs (Figure 8) were taken before and after soaking the SU-8 samples in

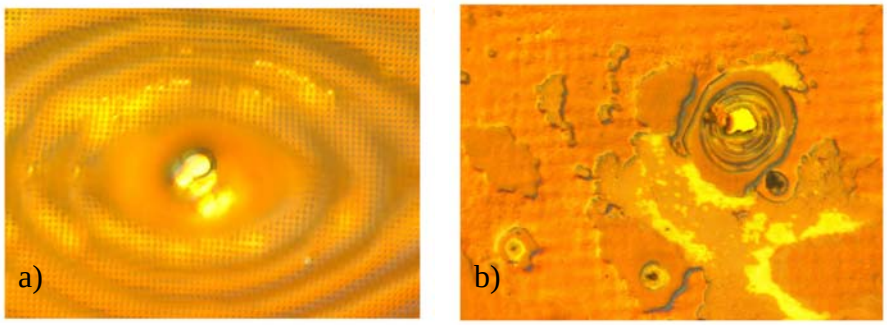

Figure 7. Optical images taken after plating at a current density of 5 mA/cm^2. a.) shows a large (>50μm) gold globule; b.) shows obvious areas of film non-uniformity.

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

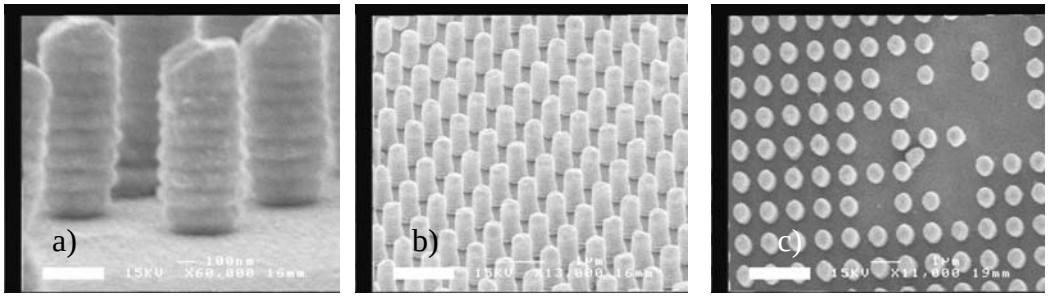

Figure 8. a.) Cross section SEM image of gold posts plated into SU-8 template at 5mA/cm2; b.) Lower magnification 45 degree angle SEM image of posts; c.) Top down SEM image of gold posts.

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 9. 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

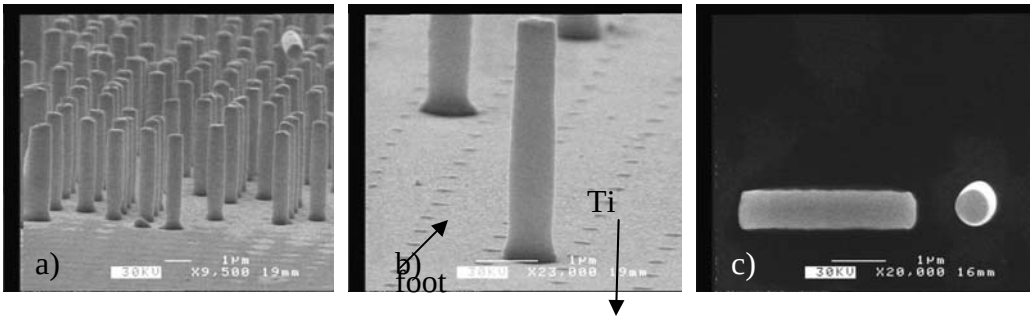

Figure 9. a. Low magnification SEM image of array of gold posts. Aspect ratio ~5; Higher magnification SEM image showing single post; c.) Top down SEM image showing one standing post and one fallen post.

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. Although all of the patterns used in this initial research were 2-D patterns, it is possible to use IL to generate 3-D structures. Figure 10 shows a face centered cubic lattice formed with three separate interference

patterns in a modification to the standard set-up used to form the purely 2-D structures shown previously.

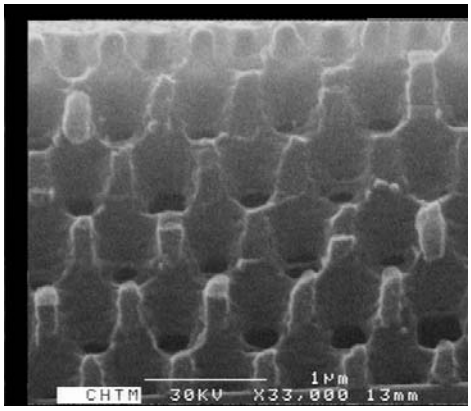

Figure 10. SEM image of a face centered cubic structure from a 3-D interferometric lithography approach

The results of this phase of the research have tremendous potential application in a wide variety of fields. First, an optimized array of gold pillars with aspect ratios such as these would be ideal platforms to study various surface chemistry applications. Thiol molecules readily bond with gold surfaces, allowing additional functionalization of the gold posts. For instance, gold posts coated with lipids could serve as intercellular probes for measuring electrical activity inside living cells [1]. Furthermore, through a combination of surface chemistry and hierarchical structure it may be possible to mimic the adhesive properties of the gecko foot by performing multiple stage plating culminating with high aspect ratio, submicrometer structures [2]. In addition to gold, magnetic material such as FePt can be plated into the features. It is theorized that if plated into holes smaller than the nominal grain size, on the order of 50-80 nm, magnetic domain alignment will occur, yielding a film with high coercivity [3]. Finally, alternative methods for filling the pores could yield interesting structures. Atomic layer deposition of tungsten followed by a polish back step would yield structures which could be used as field emitter tips, a potentially useful component in plasma and terahertz radiation generation [3].

2.3 SU-8 as a DUV Resist

Being designed for use at i-Line, SU-8 possesses a very steep absorption edge for wavelengths below ~350 nm. Because of this, the absorption depth for shorter wavelengths is extremely small. In applications where the resist thickness is substantial, this short absorption depth makes SU-8 unusable for exposure wavelengths below 350 nm. If, on the other hand, the entire resist thickness is less than the absorption length, on the order of a few hundred nanometers, wavelengths shorter than 350 nm can be used to expose SU-8. The result is that it may be possible to use SU-8 as a DUV (193 nm) negative resist, a very useful development.

Figure 11 shows some SEM images of the first attempts at patterning SU-8 with a DUV source. In this case, the source is an ArF excimer laser operating at 193 nm using a Lloyd mirror set-up and interferometric lithography. In this case, the angle of incidence is

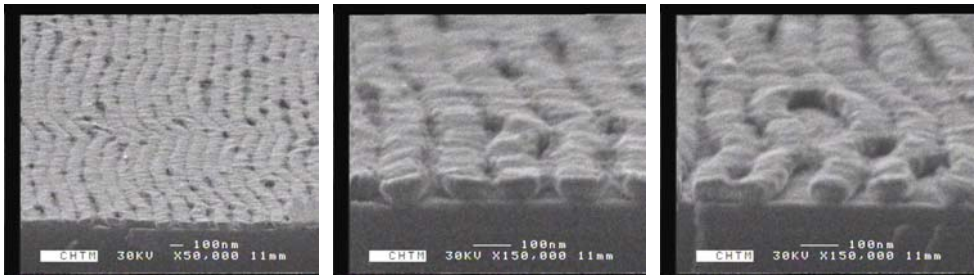

Figure 11. Three images showing patterns exposed in SU-8 with a DUV 193 nm ArF excimer laser.

adjusted so that a 1-D grating pattern is formed with a period of ~120nm. From these images, it is apparent that the resist is capable of being patterned with the DUV source, however the resulting lines suffer from a lack of adhesion to the substrate. These results are encouraging, and warrant further investigation.

3. Conclusions

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 LDRD, as well as a Senior Council LDRD which followed directly from this research, take a different tack on this challenging problem: using self assembled 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 three new potential research thrusts: 1) SU-8 as a direct growth mask, 2) large area high aspect ratio structures, and 3) SU-8 as a DUV resist.

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 SU-8 growth masks, we are better able to conduct research on growth in the much smaller nano-scale pores of self assembled silica and 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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