Shape control in wafer-based aperiodic 3D nanostructures

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Abstract
Controlled local fabrication of three-dimensional (3D) nanostructures is important to explore and enhance the function of single nanodevices, but is experimentally challenging. We present a scheme based on e-beam lithography (EBL) written seeds, and glancing angle deposition (GLAD) grown structures to create nanoscale objects with defined shapes but in aperiodic arrangements. By using a continuous sacrificial corral surrounding the features of interest we grow isolated 3D nanostructures that have complex cross-sections and sidewall morphology that are surrounded by zones of clean substrate.

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(Some figures may appear in colour only in the online journal)

1. Introduction

Structural control of nanoscale devices is a key issue for enhancing their performance, which depends critically on the nanostructure’s shape, size, and composition. For instance, the electrical properties of memristors [1, 2], and photovoltaic devices [3, 4]; the optical properties of metal plasmonic nanoparticles [5, 6]; and the magnetic properties of nanomagnets for magnetic memory devices [7, 8] all depend on the geometry of the device. Therefore, in order to improve the performance of nanodevices, three-dimensional (3D) nanofabrication techniques are becoming more important to nanoscience and nanoeengineering. 3D structures including rods, helices, etc have been obtained via template-assisted growth [4, 9], scanning probe lithography [10], 3D assembly of nanoparticles [11], DNA origami [12–14], multi-photon lithography [15, 16], colloidal lithography [17] and chemical synthesis [18], but these approaches are not well suited for locally fabricating 3D-shaped nanostructures on a wafer or planar surface. For instance, colloidal lithography is capable of rapidly patterning large areas, but only with periodic arrays, and only to give 2D patterns. On the other hand, scanning probe lithography can create arbitrary aperiodic structures (albeit at reduced write speeds). So there remains a pressing demand for techniques to generate 3D structures in arbitrary arrangements with rapid write times.

A promising technique to address this is glancing angle deposition (GLAD); a physical vapour deposition technique where the vapour is deposited on the substrate at a high incidence angle and the substrate can be rotated during deposition [19]. GLAD has been used to fabricate various complex 3D structures including rods, helices, zigzags, etc where the structural dimensions are directly controlled by the incident molecular flux angles, the molecular deposition rate, and the rotation rate of the substrate. It has the advantage of being a simple and fast fabrication process appropriate for a...
variety of materials including insulators, semiconductors, magnetic materials and conductors [20–22]. Seeding the substrate before the GLAD exposure has proven to be an effective technique to homogenize the resulting structures by restricting extinction, and controlling the symmetry of the array [23]. Recently, we have shown a rapid and scalable approach to obtain 3D nanocolloids using GLAD on substrates seeded with ~10 nm gold nanodot arrays grown with block-copolymer micellar nanolithography [24]. To date however, studies of GLAD seeding have focused on the role of highly symmetric, periodic, and quasi-infinite seed arrays [25–27]. Thus they are not appropriate for growing the isolated or aperiodic 3D nanostructures that are required for many technological applications.

In this work, we use electron-beam lithography (EBL) to write nanoscale seed layers having complex seed shapes, and isolated aperiodic arrangements. We start by exploring the growth properties of nanostructures on periodic patterned seed arrays and demonstrate that the lattice spacing of the array influences and thus limits the dimensions of the nanostructure. To address this we introduce a new geometry, the sacrificial corral, to protect the main structure and allow more precise control of the structural dimensions. We present examples of GLAD grown nanorods and nano-helices, grown from oxides and metals; in all cases the structures grown within corrals exhibit better uniformity, and show superior fidelity for the underlying seed geometry and the growth programme than isolated seeds or periodic seed arrays.

2. Theory

In the absence of diffusion, when adsorbate atoms hit and stick where they land, the morphology of GLAD grown structures is dominated by ballistic shadowing by adjacent structures. In the presence of seeds the diameters of the grown structures are dependent on the seed diameter \(d_s\), and the gap between adjacent seeds \(g\) [28]. The maximum effective gap between seeds \(g_{\text{max}}\) is defined by the geometrical relation,

\[ g_{\text{max}} = h_s \tan(\alpha), \]

where \(h_s\) is the seed height, and \(\alpha\) is the polar angle of the incoming flux. When the seed gaps are wider than the maximum effective gap distance \(g > g_{\text{max}}\), unseeded nanostructures grow between seeds due to the absence of the shadow effect in these regions. On the other hand, columnar competition, the complete shadowing and stochastic extinction of some structures by their neighbours decreases the uniformity of growth when the seed gaps are in the range of \(d_s \leq g \ll g_{\text{max}}\). The centre-to-centre spacing of seeds in an array is simply \(s = g + d_s\), and the maximum effective spacing is \(s_{\text{max}} = g_{\text{max}} + d_s\).

3. Experimental

The fabrication scheme for one of the designed nanostructures is depicted in figure 1. Nanopatterned seeds based on hydrogen silsesquioxane (HSQ) negative tone electron-beam resist were fabricated by electron-beam lithography (Jeol, JBX-6300FS). HSQ resist was spin coated onto Si wafers at speeds of 6000 rpm for 1 min to yield a 30 nm thick resist layer. After spin coating, the samples were baked at 90 °C for 1 min. The custom seed patterns were written in a JBX-6300FS lithography system at 100 kV acceleration voltage and 1 nA beam current. The resist were developed by immersion in MF322 for 80 s, and rinsed in deionized water for 30 s.

Nanostructures were grown on such seeded substrates in a GLAD system based on electron-beam evaporation at room temperature with a base pressure of \(5 \times 10^{-7}\) mbar. The substrate manipulator provides independent control over the azimuthal direction \(\varphi\), and molecular flux direction \(\alpha\) during deposition. The flux angle \(\alpha\) and the azimuthal rotation rates per unit thickness \(d\varphi/dt\) were constantly updated with closed-loop feedback based on measurements of material deposition rates on a quartz crystal monitor and controlled by a computer running code developed in the lab. In order to examine only the geometrical effect of the designed structures, other deposition parameters including the temperature, the material, the deposition rate, and the incident molecular flux were kept constant between different samples. In particular, the flux angle was fixed to 85°, because it is the primary factor...
determining film density [27]. At 85° the polar angle is high enough for shadowing to produce high fidelity structures, but not so high that roughness in the mask layer plays an important role in shadowing. A typical growth process consisted of Ti deposited on top of the nanopatterned seeds as an adhesion layer with a deposition rate ∼0.1 nm s⁻¹ at α=10°, \( \frac{d\rho}{d\theta}=210° \text{ nm}^{-1} \). This was followed by alumina (Al₂O₃) which made up the bulk of the nanostructure, deposited to give either nanorods by fast azimuthal rotation (\( \frac{d\rho}{d\theta}=(18\pm0.2)^° \text{ nm}^{-1} \) at 0.1 nm s⁻¹ deposition rate and \( \alpha=85° \)) or helices using slow rotation (\( \frac{d\rho}{d\theta}=(1.8\pm0.1)^° \text{ nm}^{-1} \)).

4. Results and discussions

The ability of EBL to write arbitrarily shaped seed layers of small sizes allows the effect of those seed layers on the resulting GLAD-grown nanostructures to be studied systematically. We start by examining circular seeds arranged in periodic square lattices. This was followed by GLAD deposition of about 250 nm of Al₂O₃ at high substrate rotation rate to give columnar nanostructures as shown in figure 2. The seeds increase in diameter (\( d_s \)) from top to bottom, and increase in centre-to-centre spacing (\( s=g_x+g_y \)) from left to right. Each zone of seeds is 10 μm×10 μm but the images show only the central 1.3 μm×1.3 μm to avoid edge effects. The diameters of the resulting nanocolumns were measured by analyzing the SEM images. Ideally, the diameter of the nanorod should be equal to that of the seed upon which it grows. However, figure 2(B) shows that for a given seed diameter, the diameter of the nanorod increases with spacing. When \( d_s \leq g_x \ll g_{\text{max}} \), the uniformity of the nanorods decreased due to columnar competition. Conversely, when the seed gap exceeded that established by the geometrical relation \( g_{\text{max}} = \frac{h}{g_y} \tan (\alpha) \) (e.g. for \( d_s=50 \text{ nm}, h=30 \text{ nm}, s=550 \text{ nm} \)), unintended nanoslots were formed within the gaps of the patterns due to incomplete shadowing. Therefore, in periodic lattices, the nanorod diameter is a function of both the seed diameter and spacing at the given seed height incident flux angle. This poses a problem for aperiodic arrangements where a fixed column diameter is desired, but the spacing between columns, or their arrangement is not uniform. This is an important consideration in structure design that has received little attention, because GLAD has traditionally been applied to the growth of periodic, quasi-infinite films.

We therefore introduce a new motif for the controlled growth of isolated structures: the sacrificial corral, which manipulates the shadow effect locally and protects the target structures within. Our model structure for testing the efficacy of corrals is a pair of circular seeds \( d_s=50 \text{ nm} \) in diameter and separated by 100 nm (see figure 1(b)). These represent the electrodes of any simple two-terminal device. The corral is a continuous closed path drawn around these seeds at a constant minimum distance. We used EBL to draw 50 nm wide corrals at different distances (100, 250, 400, and 550 nm) from the central structure, as shown in figure 3(a). These patterns are used as seed layers for growing Al₂O₃ by GLAD to yield the nanostructures in figures 3(b) and (c). The top down SEM images (figure 3(b)) show that shadowing from the corral produces a denuded area on both its interior and exterior. The pair of nanocolumns grown in the centre of the corrals increase in diameter as the corral gap increases, similar to the effect seen with periodic arrays. For \( \alpha=85° \) the maximum effective gap is expected to be \( g_{\text{max}}=342 \text{ nm} \). This is consistent with the appearance of unintended deposition near the nanodot pair for the corral with 550 nm gap. Thus, within the limits imposed by geometrical shadowing, the corral acts as a ‘resistless mask’ that maintains a clear area in the vicinity of the double dots, without the need for additional mask steps.

As a control system to confirm the role of the sacrificial corral, the nanorods were fabricated on double dots arrays (see figures 3(d) and S1) using lattice spacings between the dot pairs the same as the gaps used for the corrals in figure 3.
One additional layout was used with very large interpair spacing corresponding to isolated double dots. The nanorods’ heights and lengths were measured and plotted in figure 4(a), for comparison against those of the corral structures. The measured values of the nanorods’ heights were calibrated by geometrical relations between the projection viewed by SEM and nanorods’ geometry (see supplementary information for details, available at stacks.iop.org/NANO/25/235302/mmedia). This shows a strong trend for larger diameter dots as the spacing is increased in both corrals and arrays. In the limit of large spacing they converge towards the measured values for the isolated double dots (blue and red bars), as expected. Corralled double dots consistently have substantially smaller diameters (by approximately 20%) than their array counterparts. This results in an enhanced aspect ratio (figure 4(b)) for corralled double dots for spacings less than $s_{\text{max}}$ (it was not possible to measure the height of corralled structures for $g_s = 100$ nm at 45° tilt because the corral itself obscures the base of the nanorod during imaging. Instead, these were measured at 20° tilt). At the lower end of the gap range ($g_s = 100$ nm) the aspect ratio for corralled structures is no better than that of the periodic ones. This arises because densely spaced seeds admit fewer gaps between them, and so are shadowed for proportionally more of the azimuthal sweep (see supplementary information for details). A simple single ray analysis, shows that the $g = 100$ nm structures are shadowed for twice as much of the deposition cycle as for 250 nm spacing. The ratio of $d/d_s$ for corralled dots is a function of the gap size, but is insensitive to the spacing between corrals (figure S2). Thus, the corral motif decouples the shape of the nanostructures from the distance between them.

Adding an additional corral surrounding the first one creates a double corral (see figure S3). Doing so has no effect on the dimensions of the target inside the first corral, as seen in figures S3(b) and (c). The utility of including extra corrals is that they provide additional annular regions of clean substrate around the target structure.

The greatest strength of the GLAD technique is its ability to grow structures that have 3D shapes, and are sculpted perpendicularly to the substrate normal [19, 20, 24]. We now demonstrate that, in addition to controlling the aspect ratios of nanorods, the sacrificial corrals enhance the fidelity of nanostructures with 3D-shapes. In this process, 500 nm thick Al$_2$O$_3$ was deposited by GLAD with slow substrate rotation corresponding to 200 nm per turn. This growth programme should produce left-handed helices with 2.5 turns. Figure 5 shows the results when grown on an isolated dot doublet (a), a double dot array (b), and a double dot enclosed in a corral (c). Neither the structures grown on the isolated double dot, nor
the double dot array have a helical shape. However, the nanostructures grown within the corral demonstrate clear helicity as expected from the growth programme, with a total height of $(179 \pm 9)$ nm and pitch of $(64 \pm 9)$ nm. This can be attributed to the homogenizing effect of the constant shadow cast by the corral, versus the irregular variations in shadow length experienced by the periodic seeds. A structure within a periodic array is shadowed only periodically whenever the azimuthal angle coincides with the direction of one of its neighbours (see supplementary information). Between these angles the structure is not shadowed, and receives flux on its flanks, as well as its top surface. This introduces a shadowing modulation in addition to the deliberate modulation that comes from the substrate rotation.

Ultimately, the goal is to construct 3D structures grown on seeds with complex shape. For this it is necessary to preserve the cross-section of the seed shape as it is swept through the 3D profile grown by the GLAD process. Previous work on GLAD seeding has been restricted to spherical, hemispherical, and square shaped seeds [23, 24, 27–33]. Figure 6 shows this for an ‘A’ shaped seed, using growth conditions identical to those for figure 5. The growth was performed on a patterned substrate consisting of isolated ‘A’ seeds, an array of such seeds, and a corralled seed. The isolated ‘A’ yields grainy and overgrown structures with poor fidelity for the underlying seed and weak helicity. Similarly, the array structures suffer from unintended deposition throughout the spaces between the seeds, with particularly strong growth between the strokes of adjacent ‘A’s at their baseline. Conversely, the corralled structure shows close reproduction of the original seed, clean substrate within the corral, and good helicity. It remains afflicted by some graininess, but this can be mitigated by further optimizing the growth processes such as the rotation speed, the incident flux angle, or the materials [33]. In figure 7, we introduce one possible example to fabricate 3D nanostructures with a smooth surface on complex shaped seeds. Here we grow 500 nm of Ag on the seed patterns at $d\phi/d\theta = (1.8 \pm 0.1)^\circ$ nm$^{-1}$. The seeds are covered with good continuous surface morphology of Ag nanostructures which contain a surrounding clear area within the corral. Metals, of course, play a crucial role in lithographic processes by acting as conductive interconnects and traces. Here we have used constant growth conditions to focus on exploring the role of our seed configuration. However we have shown previously...
that, when combined with nanoscale seeding, substrate cooling during growth permits fine control of GLAD-grown features in a wide variety of materials, including metals and semiconductors. We anticipate that corralled growth can be applied to any material which has already proven GLAD compatible. So a wide range of materials, including challenging ones like the noble metals, are suitable for the growth of aperiodic structures using corrals.

5. Conclusions

The use of periodic seed arrays for growing nanoparticles by GLAD is a powerful technique subject to some limitations: it is only useful for geometries that require periodicity, and where the dimensions of the nanostructures are controlled by the periodicity of the array in which they grow rather than being independent. The corrals introduced here serve three important roles: (a) they permit the growth of aperiodic structures so that the dimensions of the structure do not depend on the spacing to adjacent structures; (b) they improve the structural quality of the features they contain, with higher aspect ratios for nanorods, and better helicity for nanohelices; and (c) they create a clean area free from unintended deposition around the structures of interest thus acting as a type of ‘resistless mask’.

We anticipate that these corrals will offer new possibilities in GLAD based growth of 3D nanostructures. Possible applications include chemical-bio-sensors based on surface plasmons [34, 35], single point lithography [36, 37], and imaging [38, 39]. In particular the dimensional control offered by corrals creates the opportunity to place nanoscale features in arbitrary arrangements with the knowledge that a feature’s structural dimensions are not influenced by the spacing to its neighbours. Furthermore, the clear area within the corral that surrounds the features at the centre will be particularly important in nanoelectronic applications involving metals where the unintended deposition that normally occurs would lead to short circuit in the structures [40–42]. Recent work in the field of single molecule surface enhanced Raman scattering has shown that our geometry—two closely spaced posts on a clean region within a ring—makes a very effective optical antenna allowing single molecule detection [34]. Fabricating such devices with the techniques reported here would be not only simpler, requiring substantially fewer fabrication steps, but also offers the opportunity for shape control of the post structures. In particular, GLAD grown posts could be chiral which provides the opportunity for enantiospecific sensing.

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