Self-organization of large-area periodic nanowire arrays by glancing incidence ion bombardment of $CaF_2(111)$ surfaces

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The fabrication of well-ordered nanowire arrays over large areas is a challenge with many potential applications. Here we report a novel glancing incidence ion beam assisted self-organization approach to form periodic wire arrays over large areas on a $CaF_2(111)$ substrate. Preferential erosion of fluorine by the 4.5 keV Ar⁺ ion beam creates a surface enriched in calcium. The calcium self-organizes in elongated island structures of preferential width and separation. If the sample is irradiated along a fixed azimuth we observe nanowires with ~10 nm periodicity and wire lengths of at least several micrometers oriented along the azimuthal direction of ion beam incidence. Electrical conductivity measurements reveal an anisotropy in conductivity of at least three orders of magnitude. © 2001 American Vacuum Society. [DOI: 10.1116/1.1349722]

I. INTRODUCTION

Efficient methods for the fabrication of nanoscale structures have attracted increasing attention in recent years. Selfassembly phenomena offer an alternative to lithographic methods and are particularly attractive for fast nanoscale surface patterning of large areas. Some systems minimize their free energy by self-organizing into structures with welldefined characteristic length scales. Such systems spontaneously form large arrays of nanostructures. Strain fields play an important role in most self-assembled quasi-twodimensional surface systems. The interfacial stress caused by the lattice mismatch between island and substrate can impose an upper limit on the cluster size, leading to the formation of clusters with a narrow size distribution. In semiconductor heteroepitaxy this technique is widely used to form quantum dots.¹ Many applications demand both a narrow size distribution and a high degree of spatial ordering of the resulting structures. These characteristics are not necessarily related, however, it is known that strain fields at surfaces can lead to spatial ordering.² Periodic stress domains have been observed, for example, on the partially oxidized Cu(110) surface³ and in two-component thin films grown on an immiscible substrate.⁴ Many other regular surface patterns may be explained by stress domains, examples being the Au(111)"herringbone" surface reconstruction⁵ and the formation of pseudomorphic Fe islands with long range periodicity on Cu(311).⁶

The self-formation of spatially ordered one-dimensional structures (periodic nanowire arrays) over macroscopic areas is even more challenging than the formation of spatially ordered quasi-zero-dimensional (dot) structures. One promising technique is the decoration of step edges on vicinal

surfaces.^{7–9} In this article we present a different approach for the self-assembly of periodic nanowire arrays. In contrast to most other techniques for self-organized nanostructure fabrication, no material is deposited. Instead, we preferentially erode one component of a two-component material by glancing incidence ion irradiation. In our experiments, structural reorganization of this depleted surface, to minimize the free energy of the system, is responsible for formation of ordered nanowire arrays. This is not the first time that ion beams have been used to structure surfaces at the nanoscale. Selforganization of hexagonal ordered dot structures on GaSb following normal incidence ion irradiation has recently been demonstrated.¹⁰ Off-normal ion beam irradiation has also successfully been utilized to nanopattern single component $metal^{11-14}$ and $semiconductor^{15,16}$ crystal surfaces. Ripple structures have been reported along preferred crystallographic directions and oriented normal and parallel to the azimuthal direction of incidence of the ion beam. Their formation is believed to be related either to competition between erosion and kinetic restrictions on the diffusion of adatoms,11-14,16 or to a surface instability arising from a surface-curvature dependent sputter vield.^{15,17} The nanowires formed in our experiments are of another origin. They are of a different composition to that of the substrate and exhibit a well-defined periodicity. We explain our results as arising from self-organization of the surface into periodic stress domains.

II. EXPERIMENTAL METHODS

Nanostructured samples were prepared in a modified version of the multipurpose UHV apparatus developed earlier for ion beam polishing and thin-film deposition applications.¹⁸ It consists of a load lock attached to a vacuum chamber equipped with a 0.5–5 keV ion gun, rear view low-energy electron diffraction optics, quadrupole mass spectrometer, sample transfer and manipulation devices, and a commercial Beetle-type scanning tunneling microscope.

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FIG. 1. Contact mode AFM micrographs of CaF₂(111) surfaces, unirradiated and irradiated with a 4.5 keV Ar⁺ ion beam under different incidence angles and under continuous azimuthal sample rotation. Image (a) is that of a cleaved CaF₂(111) surface, image area $1 \times 1 \ \mu m^2$. The cleavage steps are ~0.3 nm in height, equivalent to the separation of individual cleavage planes. Image (b) shows a sample irradiated at 4° glancing incidence angle, with an ion fluence of 2.6×10^{14} ions/mm² (image area $400 \times 400 \ nm^2$). Image (c) is of a sample prepared under the same conditions as (b), but the image area is $1 \times 1 \ \mu m^2$. Images (d)–(f) depict samples which were irradiated with an ion fluence of $1.3 \times 10^{15} \ ions/mm^2$, but at glancing incidence angles of 6°, 10°, and 16°, respectively (image area $400 \times 400 \ nm^2$).

Two electron beam evaporators have been added to the apparatus described previously. The evaporator axes lie at a polar angle of 13° to the sample surface normal. In order to perform conductivity measurements, macroscopic silver contact patches separated by 10 μ m were deposited on previously nanostructured samples. This was achieved with the aid of a shadow mask consisting essentially of a thin nylon fiber brought into close proximity to the sample. In this way, vacuum measurements of the electrical conductivity of wire arrays over a 200 μ m width spanning a 10 μ m region of the surface could be performed. A biased preamplifier was used for measuring the conductance of the samples. After measuring the sample resistance in vacuum, the samples were examined in air with an optical microscope for a high density of cleavage steps or other defects in the gap region. Nanoscale examination of the sample surface topography was performed under ambient conditions using contact mode atomic force microscopy (AFM) and ultrasharp cantilevers with a nominal tip-end radius of less than 10 nm.

Calcium fluoride crystals were cleaved in air and mounted on the sample holder. The crystals were inserted into the load lock within 30 min and transferred into the UHV chamber within another 30 min. There the sample was mounted on a stepper motor controlled, combined linear and rotational manipulator. Adjustment of the sample position and the tilt angle of the ion-gun allowed ion irradiation of the sample at glancing angles between 0° and 16° . During ion irradiation the azimuthal angle of incidence of the beam on the sample could be varied continuously, in preprogrammed steps, or held constant. A 4.5 keV Ar⁺ ion beam was used. The ion beam was focused to a spot size of several hundred microns and scanned across the sample. The scanning area exceeded the sample size, so that secondary electrons were produced by ions hitting the aluminum sample holder. This was important to avoid excessive charging of the CaF₂ crystal during ion bombardment. Contamination of the sample surface with material sputtered off the sample holder was avoided by mounting the sample surface out of sight of the sample holder. The ion fluence was estimated from the ion beam current measured on an aluminum plate placed in the same position as the samples. We have made no compensation for secondary electron emission and therefore expect the quoted ion fluence values to be systematically overestimated.

After irradiation, the sample was either removed from the vacuum system for ambient AFM topographic characterization, or kept in vacuum for conductivity measurements. AFM measurements were performed within a few hours of exposing the irradiated sample to air. Evaporation of contacts



FIG. 2. Sample irradiated at a fixed azimuthal orientation with a 4.5 keV Ar⁺ ion beam and an ion fluence of 2.6×10^{15} ions/mm² at a glancing angle of 10°. (a) Contact mode AFM image, area $1 \times 1 \ \mu m^2$. (b) Enlarged portion of the same surface, $260 \times 100 \ nm^2$. (c) Cross section through the line indicated in (b).

for conductivity measurements was completed within 45 min of ion irradiation of the sample. The base pressure in the vacuum chamber was $\sim 4 \times 10^{-8}$ mbar.

III. RESULTS

The cleaved $CaF_2(111)$ surface exhibits large (~100 nm wide) flat terraces, separated by occasional cleavage steps [Fig. 1(a)]. These steps are one or a multiple of the separation of individual cleavage planes of the CaF_2 crystal. CaF_2 cleaves along the (111) face between two fluorine layers, leading to fluorine terminated surfaces. Samples were ion beam irradiated under a range of fluences, incidence angles, and azimuthal orientations. In the following sections we describe (A) the dependence of the surface morphology on the ion beam glancing incidence angle under conditions of continuous azimuthal rotation and constant total ion fluence; (B) the surface topography of samples that have been irradiated at a fixed azimuthal angle; and (C) the electrical conductivity of samples irradiated at a fixed azimuthal angle.

A. Samples rotated during irradiation

In this section the influence on the sample surface morphology of ion beam irradiation under a variety of incidence angles but continuous azimuthal rotation is described. Samples irradiated at a 4° incidence angle were exposed to an ion fluence of 2.6×10^{14} ions/mm². All other samples described in this section were irradiated with a nominal ion fluence of 1.3×10^{15} ions/mm².

A labyrinthine island structure (i.e., elongated, meandering, and sometimes ramified islands) was observed in AFM images of samples irradiated under a glancing angle of 4° [Fig. 1(b)]. The apparent island height measured in the AFM images is 0.6 ± 0.2 nm and the apparent island width is 6-10nm. Tip size effects, however, could cause an overestimation of the island width and an underestimation of the island height. Two-dimensional fast Fourier transforms of such images reveal a characteristic length scale of 12 ± 3 nm. No distinguished directionality can be seen in such transforms. The characteristic length scale appears to be associated with the mean separation of adjacent islands. In addition to the



FIG. 3. Surfaces irradiated under different ion beam glancing incidence angles and fixed azimuthal orientation. (a) AFM topograph of a wire array formed at a glancing incidence angle of 10° , area 390×390 nm². (b) Autocorrelation image of (a). Such images reveal up to 50 oscillations, indicating the high degree of phase coherence and periodicity of the nanowire arrays. (c) and (d) AFM topographs of samples irradiated at glancing angles of 14° and 16° , respectively (image area 500×500 nm²).

island structure, large-scale two-dimensional bubble features are observed on these surfaces [Fig. 1(c)]. These features have been recently associated¹⁹ with a phase segregation of the surface into fluorine terminated and color (F-) center terminated regions. A similar labyrinthine island structure was observed on samples prepared under an ion beam incidence angle of 6° [Fig. 1(d)]. The bubble features, however, were not present at this slightly increased incidence angle.

Samples prepared at an incidence angle of 10° did not exhibit any elongated islands. Instead, compact islands are seen on the surface [Fig. 1(d)]. The island height is again $\sim 0.6 \pm 0.2$ nm. Two-dimensional fast Fourier transforms of these AFM images reveal a preferential island separation of 10.6 ± 2.0 nm. No long range spatial ordering is observed. A large lateral scale (~ 100 nm) surface undulation is superimposed on this 10.6 nm scale island structure. The height fluctuations associated with these larger scale undulations are ~ 0.3 nm, the interlayer separation of the (111) planes in CaF₂.

A further increase in the ion beam incidence angle to 16° results in a surface topography with no clear preferential structure size [Fig. 1(f)]. The structure heights are found to be 0.5 ± 0.2 nm; similar to that found on samples prepared at lower incidence angles.

B. Samples held fixed during irradiation

In this section the influence on the sample surface morphology of ion beam irradiation under a variety of incidence angles but fixed azimuth is described. Figure 2 shows an AFM image of a sample that was irradiated at an incidence angle of 10° (fluence $1.3 \times 10^{15} \text{ ions/mm}^2$). The surface is covered by a dense array of nanowires of height ~ 3 nm and periodicity 9.5 ± 1 nm [Fig. 2(c)]. This wire array structure is superimposed on a large-scale lateral surface undulation of a few angstroms. The nanowires, however, do not seem to be influenced by this surface modulation.

A systematic study of the dependence of the surface topography on the ion beam incidence angle was conducted for an ion fluence of 2.6×10^{15} ions/mm². At glancing incidence angles less than 8°, no ordering into nanowires was observed at this fluence. For incidence angles between 8° and 10°, nanowire arrays with periods between 8 and 12 nm [Fig. 3(a)] are formed. Autocorrelation images derived from the most ordered real space AFM images reveal a remarkable coherence of the wire period, with at least 50 oscillations visible perpendicular to the wire axis in some images [Fig. 3(b)]. A well-defined wire orientation is revealed in twodimensional fast Fourier transforms of such AFM images. All components are found to lie in a 1° sector, implying that the wires do not deviate more than 0.5° from their mean orientation.

Samples prepared under ion beam incidence angles of 14° and 16° exhibit a lesser degree of long-range order. At 14° incidence [Fig. 3(c)], features are observed that span pairs of wires. The height of such features exceeds that of single wires. Figure 3(d) shows a sample that was prepared at an incidence angle of 16° . In this image the ~ 10 nm periodicity is still dominant, but pairing of adjacent wires clearly occurs. Such wire pairs exhibit a smaller corrugation across the pair compared to the corrugation between pairs.

The orientation of the nanowires always coincides with the azimuthal direction of ion beam incidence (within the experimental uncertainty of \pm 5°), independent of the crystal orientation. Furthermore, the orientation of the wire array could be changed after its formation by irradiating the sample at a different azimuthal angle. This second irradiation caused the wire array to change its alignment to that of the new azimuthal direction of the ion beam. The degree of longrange order, however, decreased with each successive irradiation step.

C. Conductivity measurements

Samples described in this section were prepared with an ion fluence of 1.3×10^{15} ions/mm² at a glancing incidence angle of 10° and fixed azimuth. The contact patches were deposited on the substrate in such a way that the nanowires ran either parallel or perpendicular to the electrodes. On unirradiated samples, the conductance was smaller than $10^{-12} \ \Omega^{-1}$ across the 200 μ m length, with a 10 μ m gap between the electrodes. On irradiated samples, the conductance when the wires were oriented parallel to the electrodes was smaller than $4 \times 10^{-11} \ \Omega^{-1}$, and thus only minimally larger than for unirradated samples. Samples with wires oriented normal to the electrodes (i.e., spanning the gap between the electrodes) had conductance values in the range $(1-6) \times 10^{-8} \ \Omega^{-1}$. In both cases a linear I-V characteristic

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was observed in a voltage range of ± 1 V. A strong electrical anisotropy therefore exists for these samples, with the conductance in the direction of the wires at least three orders of magnitude higher than the conductance parallel to the wires. This anisotropy is exceptionally large if one compares it, for example, with the factor of ~20 measured for thin layers of aligned carbon nanotubes.²⁰

IV. DISCUSSION

Convolution effects during AFM imaging of surfaces exhibiting structure sizes of the order of the AFM tip end radius can significantly influence the apparent width and height of such features. Structures usually appear wider, and consequently the separation between features is underestimated. It is therefore impossible to deduce accurate values for the widths of the wires reported here. A reliable value for the wire array periodicity, however, is obtainable, as the measured periodicity is not influenced by tip size. Clearly observable defects in individual wires suggest that the resolution of our AFM images exceeds that of the wire separation. We are therefore confident that the AFM images give a realistic impression of the real surface structure.

The increased conductivity of the samples following ion irradiation indicates a structural and/or stoichiometric change in the surface layers. A simple increase of the ionic conductance of the CaF₂ crystal due to defects induced by ion bombardment cannot explain an anisotropy in the conductance. Depletion of fluorine by potential sputtering causes a calcium enriched surface layer. Calcium can only be removed by kinetic sputtering events or thermal desorption. Calcium enrichment in the surface layer/s may therefore explain the increase in the conductivity. The coincidence of the electrical and topographic anisotropy of the surface strongly suggests that the nanowires are composed of calcium, separated by a largely unchanged "substrate" of CaF₂. This interpretation is consistent with the observation that AFM images exhibit only a small surface undulation (of the order of the CaF₂ interlayer separation) on large lateral scales, indicating that ion irradiation causes little damage to deeper layers of the substrate. Combining these observations with the knowledge that potential sputtering preferentially erodes fluorine from CaF₂, we conclude that the nanowires we observe are either composed of calcium, or are at least calcium enriched.

The formation of a periodic structure usually arises from a balance between short-range attractive and long-range repulsive forces. In our case, bonding interactions between the calcium adatoms can be identified as the attractive force. The necessary repulsive force between wires may be related to stress fields in the substrate.²¹ Such stress fields are often associated with coherent heteroepitaxial islands, and are due to lattice relaxations which accommodate the lattice mismatch between the adlayer island and substrate. Overlap of the strain fields in the substrate near the islands leads to a repulsive interaction between islands.²¹ Calcium possesses a $\sim 2\%$ larger lattice constant than CaF₂. Consequently, we expect tensile stress in the substrate beneath the adatom islands (nanowires) and compressive stress between the wires.

Such stress fields and the associated repulsive forces between islands can lead to ordering of such systems into periodic stress domains.²²⁻²⁴ Such stress fields can extend several layers into the substrate. Their range considerably exceeds interatomic distances and allows the orientation of domains to be independent of particular crystallographic directions in the crystal or surface. It is thus not surprising that in our experiments, the orientation of the wires can be arbitrarily defined by the ion beam direction. For samples continuously rotated during ion irradiation, no preferred direction is present, and kinetic restrictions on the calcium atom mobility presumably lead to the observed meandering island structures. Their preferred separation is, however, still determined by the same stress fields responsible for wire formation. We observed the formation of compact islands instead of elongated islands when the ion beam glancing angle was increased (rotated samples). This is not inconsistent with the behavior of stress domains, which undergo a transition from a stripe phase to a droplet phase as the ratio between compressive and tensile stressed areas changes.²⁵ We may speculate that at steeper ion incidence angles, the amount of material in the adatom islands changes, leading to a change in the stress fields to one favoring the droplet phase. A similar transition from elongated, meandering islands to compact, round islands has already been reported for Ag islands embedded in a Pt(111) surface.²⁶

Nanowires formed at 14° and 16° incidence angles exhibit a lesser degree of long-range order both along and perpendicular to the wire axes, compared to the wires formed at lower $(8^{\circ}-10^{\circ})$ incidence angles. However, the occasional pairing of nanowires formed at these steeper incidence angles is an intriguing feature. We speculate that this feature can also be explained by the concept of stress domains. At lower ion fluences, the wires appear to possess a smaller separation (periodicity) at all incidence angles. Only when the fluence was increased was the pairing effect observed. Zeppenfeld *et al.*²⁴ have shown that the minimum periodicity for stress domains occurs at a coverage of 50%, i.e., for the same percentage of compressive and tensile strained substrate areas. At this coverage, the free energy of the system is also a minimum. As the ion beam fluence increases, the amount of fluorine removed from the surface increases and we expect the adatom (calcium) island coverage could change. The work of Zeppenfeld et al.24 implies this would necessarily lead to an increased wire periodicity, provided that the domains (adatom islands) do not change in height and are mobile enough to readjust continuously with the change in coverage. Kinetic restrictions may prevent this optimal reduction of the total system energy. Instead, an increase in the number of Ca adatoms may be associated with an increase in height and width of the wires, without the system being able to minimize its free energy by readjusting the wire periodicity. Eventually, the wires may reach a critical width/height and it becomes energetically favorable to "pair" two neighboring wires together, an action which involves far less material transport than continuous adjustment of the array period during wire growth. Following these considerations, we propose that the wires we observe first form close to the thermodynamically favored coverage of 50%, where the system's energy is lowest. The observed two atomic layer height wires imply that in this situation, the ion beam has removed essentially all fluorine in the first F-Ca-F surface trilayer. At this coverage the periodicity is 10 ± 1 nm. This is a stable surface structure that is maintained at a low incidence angle between 8° and 10° even at higher ion fluences. Consequently, an equilibrium between mobile calcium formation and calcium removal from the surface must exist at these incidence angles. At steeper incidence angles, a higher density of Ca-adatoms are produced, and consequently the wire width/height increases with ion fluence. Eventually, pairing of neighboring wires occurs in order to reduce the free energy of the system.

Our measurements clearly show that the azimuthal direction of ion beam incidence defines the orientation of the wires. It appears that the ion beam straightens otherwise meandering islands. Ions will have steeper incidence angles on those sections of a meandering wire which deviate from the azimuthal direction of ion beam incidence. Such sections of a meandering wire will be more prone to kinetic sputtering. This will lead to a natural "streamlining" of wires. The most stable wires will be those orientated along the azimuthal direction of ion beam incidence. The ion beam is therefore primarily responsible for orienting the wires, and therefore the stress domain pattern, while stress influenced adatom diffusion²⁷ may help stabilize the pattern and make it more uniform.

V. CONCLUSION AND OUTLOOK

We have demonstrated the formation of ordered nanowire arrays with a periodicity of ~10 nm over macroscopic sample areas. We propose a stress domain model for nanowire formation using concepts developed by Marchenko,²² Alerhand *et al.*,²³ and Zeppenfeld *et al.*²⁴ The wire separation and periodicity are a simple consequence of minimization of the system's free energy, while the long-range orientational order of the wires is simply related to the fact that wires orientated along the surface in the direction of the ion beam are less prone to kinetic sputtering. The ion beam thus has a "combing" effect, straightening otherwise meandering islands.

The technique we have described is a novel, rapid method to pattern large areas with arrays of nanowires having a welldefined periodicity and orientation. Such surfaces may find application as templates for further growth or onedimensional surface functionalization.

Calcium fluoride has been suggested as a substitute for silicon oxide as the insulator material in microelectronic devices. Its wide band gap and the fact it can be grown layer by layer on most Si faces makes it a promising candidate. With this perspective, the growth of one-dimensional metallic nanostructures on CaF_2 is of particular interest for semiconductor-insulator-metal devices. Metal nanostructures could potentially find application as gate electrodes to confine electrons in a potential well in the semiconductor.

Furthermore, it seems feasible to employ a number of etching and lift-off processes to transfer a nanowire pattern on a CaF_2 surface to a silicon substrate.

Finally, the ordering of islands into regular, periodic structures may not just be limited to the system described in this article. It may be possible to use glancing ion beam irradiation as a general means of external symmetry breaking and to impose long-range order in other heteroepitaxial growth systems.

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