A New Approach for the Formation of Size and Site Controlled Metallic Nano Dots Seeded by Focused Ion Beams

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We present a new approach for the generation of metallic nano pattern, which in contrast to conventional bottom up or top down processes is based on a subtractive self organization process relying on material decomposition induced by focused ion beam (FIB) exposure. Two dimensional ordered arrays of embedded as well as freestanding nanometer sized Ga dots were fabricated by a site control technique relying on preformed craters and an irradiation mediated migration and agglomeration. The formation of these dots is discussed in terms of selective etching of arsenic due to the local energy injection by the gallium ions and further minimization of the excess free energy of the surfaces.

Further we have shown that FIB bombardment of InAs produces indium crystallites. The influence of the ion dose, the beam energy, the sample temperature and the dose rate on the surface evolution has been investigated for further III/V compound semiconductors by atomic force microscopy, scanning electron microscopy, auger electron spectroscopy and X-ray diffraction measurements.

In summary, the surface topography resulting from FIB bombardment is being investigated for possible use in nano-technology applications. This technique, based on a subtractive self organization process, may lead to a new fabrication process for three dimensional metallic nanostructures.

Introduction

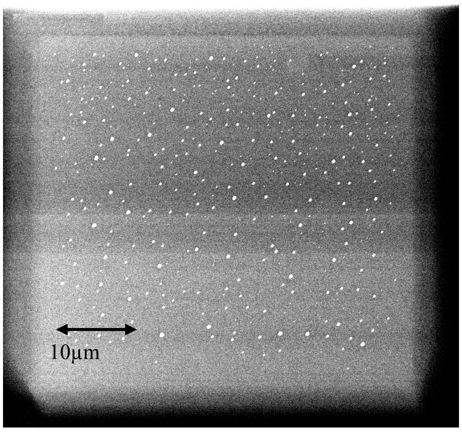
Nanoscale structuring opportunities are prerequisites for any nanoscale engineering. The main bottleneck in the application of nano dots for quantum devices [1] – [3] is the difficulty of creating ordered arrays of size controlled dots in the nanometer range with a high uniformity in size. Low-dimensional nanostructures are usually fabricated using either a top down or a bottom up strategy. The former technique is extremely flexible, but suffers from limitations in minimum feature size and uniformity. The latter one, utilizing spontaneous self-ordering effects, is limited by the broad size distribution and the lack of control of the positioning of the self-organized nanostructures. In this context the discovery of the appearance of periodic structures with dimensions in the nanometer regime induced by ion bombardment [4] – [7] has attracted growing interest due to the possibility of obtaining a self-organized formation of nanostructures. In particular resistless focused ion beam techniques are most suited for the combination of top-down structuring with selective bottom-up self-assembling techniques. To keep up with the trend of structures to shrink in dimensions, the response of ion induced material modifications will have to be controlled on a nanometer scale. A prerequisite thereof is a deep understanding of the ion beam interaction with the processed substrate material. In this paper, we present a new approach for the generation of metallic nano dots based on a subtractive self organization process, relying on compound semiconductor decomposition induced by FIB exposure.

Experimental

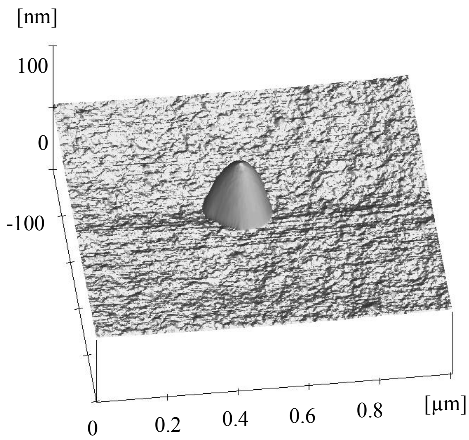
The samples exposed to the FIB were cleaved from mirror polished (100) InAs and (100) GaAs wafers. All machining experiments were carried out using the Micrion twin lens FIB system equipped with a Ga liquid metal ion source. The FIB system was operated with a 200 µm beam-limiting aperture corresponding to a nominal beam diameter of 120 nm and a beam current of 4.7 nA at an acceleration voltage of 50 kV. To investigate the morphological evolution, the samples were irradiated in a single scan with ion fluences between 6.25×10^{15} ions/cm^2 and 7.25×10^{16} ions/cm^2. After FIB irradiation, post-exposure annealing is performed at 200 °C in forming gas. For the formation of the regular Ga dot structures we generated an array consisting of 50 x 50 nominally identical 80 nm deep holes. The pattern evolution was observed by FIB-Secondary Electron Microscopy (FIB-SEM), Atomic Force Microscopy (AFM), Auger Electron Spectroscopy (AES) and X-ray diffraction (XRD).

Results and Discussion

Our FIB system enables the in-situ monitoring of the pattern evolution of the GaAs surface during FIB bombardment. Figure 1(a) shows a FIB-SEM image of a (50 x 50) µm^2 box on the GaAs surface after FIB exposure with 50 keV Ga+ ions and an ion fluence of 3.75x1016 ions/cm^2. Droplet like features appears on the GaAs surface and the diameter of the dots varies between 120 nm and 640 nm. The AFM image in Fig. 1(b) reveals a perfect calotte shape of one single dot with a diameter of 190 nm and a height of 75 nm, resulting in an aspect ratio of about 0.4 and the smooth surface surrounding of the dot.



(a)



(b)

Fig. 1: FIB-SEM image of a GaAs surface after 50 keV Ga^+ FIB exposure with an ion dose of 3.75×10^{16} ions/cm^2 (a) and the topographic AFM image of one single dot (b).

AES investigations point toward Ga enrichment in the protrusion after FIB exposure and reveal the formation of nearly pure gallium dots after a moderate annealing. We assume that the irradiation leads to decomposition of GaAs and selective etching of arsenic due to the local energy injection by the gallium ions [8]. The excess Ga atoms are produced by preferential sputtering of the arsenic atoms, and because of enhanced diffusion, the Ga atoms agglomerate into the observed Ga-rich precipitates. The development of the new heterogeneous phase leads to an increase in the total surface energy of the system. Thus, in case of preferential FIB sputtering of GaAs, the minimization of surface energy calls for a spherical shape of the small Ga dots, as we did observe. After formation of Ga dots the most crucial issue is to locate dots at predicted sites. In case of pre-patterning of the surface by milling holes in the GaAs surface the Ga-rich dots are formed at designated sites. The AFM image in Fig. 2 shows a part of the FIB generated array consisting of 50 x 50 nominally identical 80 nm deep holes, with the Ga droplets at the center of the craters.

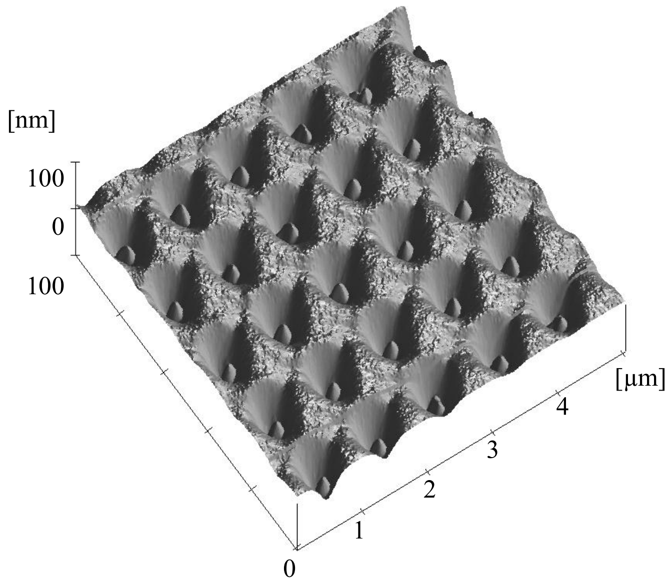


Fig. 2: AFM image of an array of embedded Ga dots generated by FIB milling of nominally identical 80 nm deep holes with a spacing of 1 µm between them.

From a section analysis of the AFM image we have measured an average diameter of the dots of 250 nm and a height of 55 nm. Besides the site controllability, the size uniformity is also improved, as each potential well in which the migrating Ga atoms are collected is uniform.

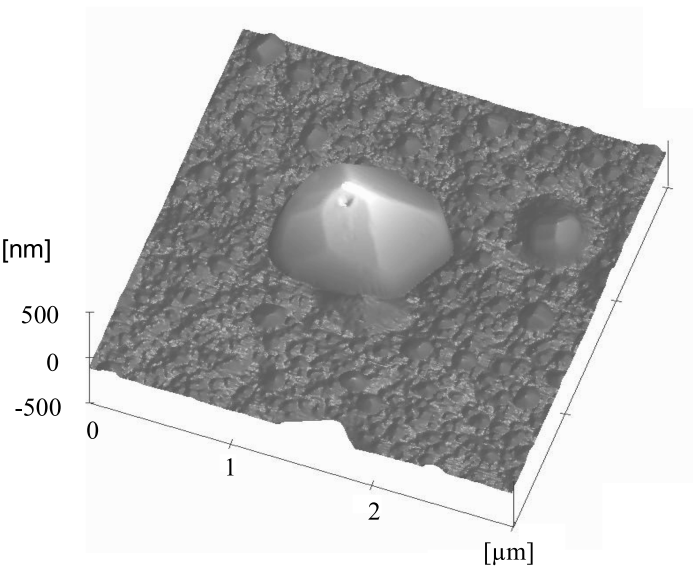


Fig. 3: AFM image of an indium crystallite on the InAs surface after FIB exposure with an ion dose of $5x10^{16}$ ions/cm^2

In case of InAs randomly distributed micro-protrusions are formed due to FIB exposure with diameters ranging from 30 nm to 2 µm. The AFM image in Fig. 3 indicates that the protrusions are nearby perfect crystallites with obvious facets, in close contact with the substrate. A two-dimensional AES mapping points towards the enrichment of indium in the crystallites, whereby the calculated composition of the ion bombarded neighborhood aside the crystallites is very close to the ratio of the undisturbed InAs surface. The XRD measurements shown in Fig. 4 prove the assumption of indium crystallite formation, as the three most intense reflections of crystalline indium are clearly visible in the X-ray diffractograms after FIB treatment. Relative intensities and *d* spacings of these reflections are in perfect agreement with reference material [8].

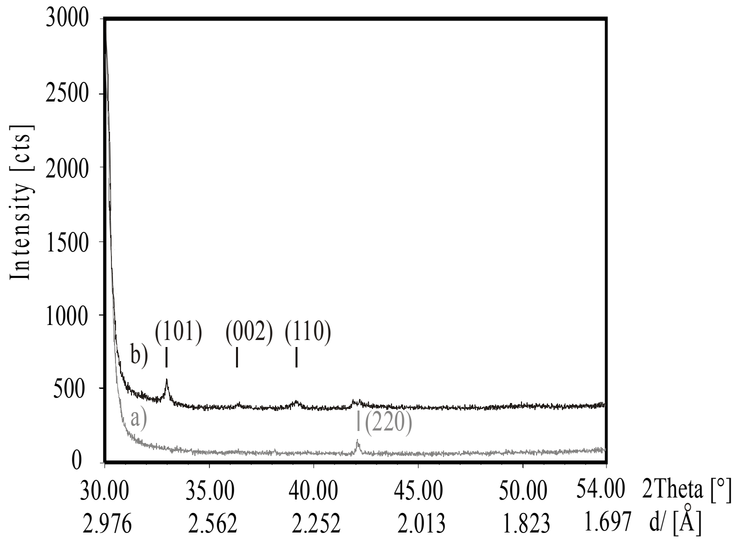


Fig 4: X-ray diffractograms of an InAs wafer before (a) and after (b) Ga^+ ion beam irradiation.

Conclusions

Concluding, we have shown that 50 keV FIB-Ga^+ ion bombardment at normal incidence produces droplet like Ga dots on a GaAs substrate and indium crystallites on the InAs surface. The formation of these dots is discussed in terms of selective etching of arsenic due to the local energy injection by the gallium ions and further agglomeration because of enhanced diffusion of the remaining species. In case of GaAs, regular patterns can be obtained by a new site control technique using ion beam surface potential modification. This technique is a promising way to obtain three dimensional metallic nanostructures with uniform dot sizes and high packaging densities. Furthermore, this method is expected to apply to various metals apart from gallium and indium.

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