Advanced Nanoscale Material Processing with Focused Ion Beams — Metallic Nano Dots Realized by a Subtractive Self Organisation Process

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Nanoscale structuring opportunities are prerequisites for any nanoscale engineering. In particular, resist-less 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.

We demonstrate surface modifications caused by focused ion beam irradiation, addressing the primary mechanisms leading to material swelling, amorphization and preferential etching. In detail, we have studied the impact of shrinking feature sizes on the sputter efficiency of focused ion beams for Si, GaAs and InAs. Milling of nano dots exhibits fundamental features for FIB patterning. Based on our experimental results, a sputter yield promoting self-focusing effect combined with a sputter rate increase at oblique angles, an opposing dose deficiency effect and material re-deposition for milling aspect ratios >1 are identified to be responsible for the complex sputter response of Si and GaAs. In addition, for GaAs the observed preferential etching of arsenic results in precipitates of mobile Ga-rich residues influencing the fundamental characteristics of FIB patterning, like sputter yield, crater bottom flatness, and ripple formation.

Fig. 1: Topographic AFM image of a Ga dot generated by FIB milling on GaAs.
Due to the high-energy injection during FIB milling and the low melting point of Ga the precipitations behave like a liquid under milling conditions. The lowest energy configuration corresponds to equilibrium determined by the minimization of the total interfacial free energy and results in spherical calotte shaped droplets (Fig. 1). Nanometer sized metallic dots can be formed in a size and position controlled fashion by combining a focused ion beam induced self-organized formation technique and a subsequent rapid thermal annealing (Fig. 2). Since GaAs quantum dot formation from Ga droplets has been reported, our technique is considered a candidate for the fabrication of highly ordered GaAs quantum dot array structures.

Fig. 2: The FIB-SEM image shows an array of freestanding Ga dots achieved by a two-step process. In the first step, we generated an array consisting of 34x34 nominally 75 nm deep holes with a spacing of 300 nm between them. Subsequently, milling of a box on the prepatterned array of holes leads to the arrangement of a regular pattern of freestanding Ga dots.

The optical microscopy and AFM analysis reveals the decomposition of the FIB modified GaAs surface layer when the temperature increases above 200 °C, whereby the dots formed during FIB exposure act as catalytic sites for thermal driven decomposition (Fig. 3). The chemical and morphological evolution of the GaAs surface due to FIB exposure and subsequent annealing has also a strong impact on the electrical properties of the GaAs surface which has been investigated by conventional 4 point van der Pauw measurement.

Further, we have shown that FIB bombardment of InAs produces indium crystallites, and the size of the crystallites increases with ion dose and ranges from 80 nm to 1.5 µm (Fig. 4). 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.
Fig. 3: The Optical microscope image shows the effect of the annealing on the surface evolution of the ion bombarded GaAs. After annealing the dots previously observed are still present, but quite different to the former now surrounded by micellar-like features incorporating straight lines (jets). These jets follow the [011] and [0-11] directions equivalent to the cleaving planes of the (100) GaAs sample. AFM investigations reveal that these micelle-like features are depressions of about 4 nm and the height of the jets piles up to more than 50 nm. We assume that annealing of the samples leads to a further destabilization of the heavily irradiated GaAs surface whereby the existing dots serve as anchor points.

Fig. 4: Topographic AFM image of the InAs surface after FIB exposure at 50 kV acceleration voltage and of an ion dose of $5 \times 10^{16}$ ions/cm$^2$. The image shows a nearby perfect crystallite with obvious facets in close contact with the substrate.