Fabrication of Semiconductor Nanostructures by Scanning Force Microscopy

K. Wiesauer and G. Springholz

Institut für Halbleiter- und Festkörperphysik, Johannes Kepler Universität Linz, A-4040 Linz, Austria

A process for fabrication of semiconductor nanostructures using scanning force microscopy (AFM) in combination with conventional optical lithography is described. It is based on the mechanical modification of ultra-thin photoresist layers with super-sharp AFM tips and subsequent pattern transfer by reactive ion etching. Minimal feature sizes of 45 nm and periods of 88 nm were achieved by this technique.

1. Introduction

The fabrication of semiconductor nanostructures for the development of quantum electronic devices has attracted tremendous interest in the last few years. In such devices, the electron wavelengths of typically 20 – 50 nm are comparable to the device dimensions. Therefore, the electronic properties are dominated by quantum phenomena. The realization of nanostructures remains a big technological challenge. Although with scanning probe microscopy controlled surface modifications have become possible down to the level of single adatoms [1], for practical applications one would like to combine the high lateral resolution of scanning probe techniques with existing semiconductor process technologies [2]. In the present work, we have developed a process sequence that combines conventional optical lithography with mechanical modifications of ultra-thin photoresist layers with super-sharp atomic force microscopy (AFM) tips [3]. This allows an efficient definition of very large, as well as nanoscale resist patterns with subsequent pattern transfer to the substrate in one single reactive ion etching step.

2. Process Description

The AFM patterning is realized by indentation of an AFM tip into a soft photoresist layer. This produces well defined structures where the resist is locally removed. The minimal feature size is mainly determined by three parameters, namely, 1) the resist thickness, 2) the size and shape of the AFM tips, and 3) the directionality of the final pattern transfer process. For optimum results the use of ultra-thin photoresist layers is of particular importance because the size of bulge of the displaced photoresist around the holes is the main limitation for the minimal feature separation.

2.1 Lithography with Ultra-Thin Photoresists

To produce an ultra-thin photoresist layer, the positive resist ‘Shipley S1805’ is diluted with a thinner (’Shipley EC Solvent’) in the proportion 1:12 to 1:15. The highly thinned photoresist is spin-coated on the sample with 5500 rpm, forming a homogeneous layer

with a thickness of about 15 nm. Conventional optical photolithography with a 'Süss Mask Aligner' was used for definition of a test pattern in the resist with various device structures with typical feature sizes of several µm (see Fig.1a).

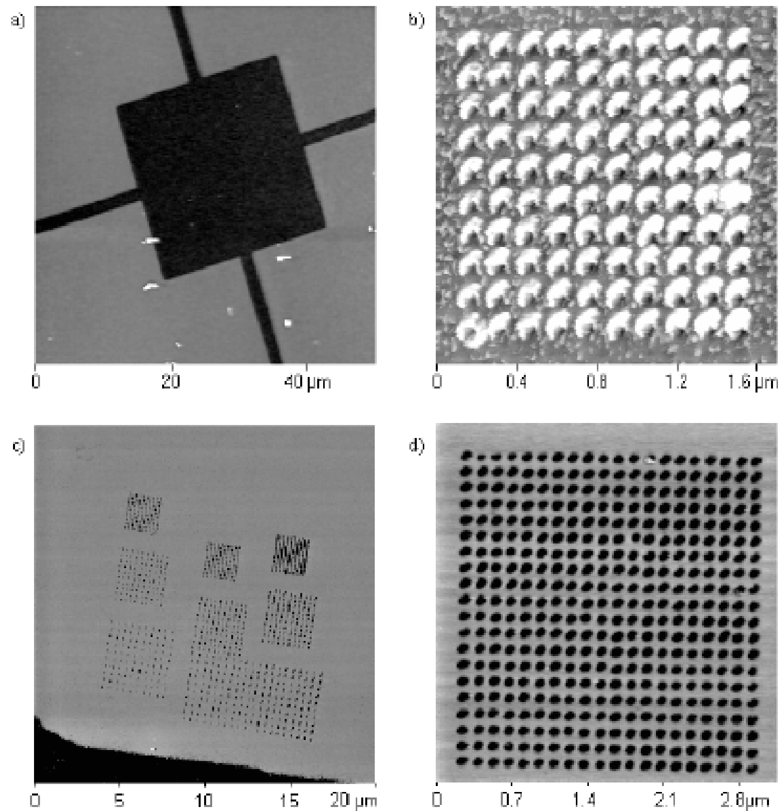

Fig. 1: AFM images illustrating the different stages of the lithography process: a) large scale resist pattern produced by optical lithography, b) hole array indented into the 10 nm photoresist the AFM, c) large scale and d) zoomed-in images after RIE pattern transfer to the Si wafer and oxygen plasma resist removal.

Ultra-thin photoresists require a careful choice of the developer since a very low solubility of the unexposed resist is required. We have tested two different developers. The developer 'Shipley MF319' shows a significant solubility of the unexposed resist as it is increasingly diluted. While the solubility of the unthinned resist is about 0.4 nm/s, it is 1.1 nm/s for a resist thinned to 1:15. Therefore, the final resist thickness of 15 nm is very difficult to control. For the 'Microposit Developer' no solubility of the unexposed resist was found. For 15 nm resists, 15 sec developing time was found to be sufficient.

The mechanical properties of the photoresist are significant for the following AFM nanofabrication process. If the resist is too soft, a reflow of the features occurs, and if it is too hard, unnecessary large indentation forces are required. This leads to increased chances of tip damage during the resist modification. The mechanical properties of the photoresist can be adjusted by a hardbake step after the developing. Here we have used a 30 min hardbake at 130° C. This hardbake also causes a shrinking of the photoresist, which becomes more pronounced with increasing resist dilution. At a 1:15 dilution, this shrinking amounts up to 30%, i.e., the resist thickness is reduced from 15 to 10 nm.

2.2 Mechanical Patterning Using Scanning Force Microscopy

Nanoscale patterns were generated by indentation of the AFM tip into the photoresist. This produces holes with the displaced photoresist left as a bulge around the holes, as is shown in Fig. 1b). The crucial parameters of this mechanical modification process are: 1) the shape and material of the AFM tip, 2) the thickness and hardness of the resist, 3) the applied indentation force, and 4) the nonlinearity corrections of the AFM piezo scanner. In order to minimize the mechanical stress on the AFM tips during indentation, the AFM was operated throughout in the 'tapping' mode, where the cantilever is vibrated near its resonance frequency with an amplitude of about 100 nm.

At first we have used focused ion beam sharpened silicon tips of 'Park Scientific Instruments'. Although these ultra-sharp tips with tip radius of around 10 nm produce very narrow holes in the resist, the monocrystalline Si tips easily break during the mechanical contact with the sample. A much better reproducibility was achieved when using carbon EBD tips that are produced by electron beam induced deposition of very hard amorphous carbon on standard silicon tips and the subsequent sharpening with an oxygen plasma [3]. This yields tip diameters in the 10 nm range. With the EBD tips many thousands of holes can be produced without significant tip degradation. Failure usually occurs only by breaking off of the carbon tip from the cantilever. The optimal tip force applied during the indentation process is reached when the tip just penetrates the layer of photoresist down to the sample surface. If the force is too strong, apart from possible tip damages, the size of the indented holes increases. At too low indentation forces the photoresist is not penetrated entirely and no reproducible pattern transfer is possible. For the 10 nm resist, we use an exposure force of about 2 μN. This corresponds to a scanner extension of 100 nm for cantilevers with a spring constant of 20 N/m.

The resist thickness is a crucial parameter for the ultimate resolution to be reached. Thicker resist layer lead to larger bulges, which limits the minimal distance between adjacent holes. On the other hand, since the resist is also attacked in the final pattern transfer by the reactive ion plasma, a reasonable thick resist is needed to allow a sufficiently deep etching of the samples. Therefore, a compromise between resolution and pattern transfer has to be found. For a resist thickness of 10 nm, a sufficient pattern transfer is possible (depth after etching about 30 nm), while the resolution is still good (minimal period about 85 nm, see Fig. 2). An important aspect for the fabrication of well defined complex structures is the compensation of the large nonlinearities of the AFM piezo scanners. This generic problem of AFM was solved using the external position control provided by the AFM of 'Park Scientific Instruments'. Test structures written without the scan correction exhibit pattern distortions as large as 10%.

2.3 Pattern Transfer by Reactive Ion Etching

For pattern transfer of the resist patterns to the Si wafers that were used as test samples, we have used an 'Oxford Instruments' reactive ion etcher with SF_6 as reactive gas. As the ultra-thin etch mask of the photoresist is attacked by sputtering, a high etch selectivity between the resist and Si is of crucial importance. We have increased the selectivity by using low RF powers (reduced sputtering rate of the resist). In addition, higher gas pressures and flow rates were found to increase the Si etch rate. A drastic reduction of the resist sputtering rate can be achieved by adding CH_4, which generates a protecting polymer layer, but unfortunately this decreases also the Si etch rate. The optimal etch conditions were found to be a RF power of 30 W, SF_6 flow rate of 50 sccm, pressure of

40 mtorr and a CH_4 flow of 25 sccm. This results in an etch rate for Si of 49 nm/min and of 6 nm/min for the resist. Finally, the photoresist is stripped from the sample in the same RIE reactor with an oxygen plasma (90W, 10 sccm, 60 mtorr). The resulting patterns after these processing steps are shown in Fig. 1 c) and d).

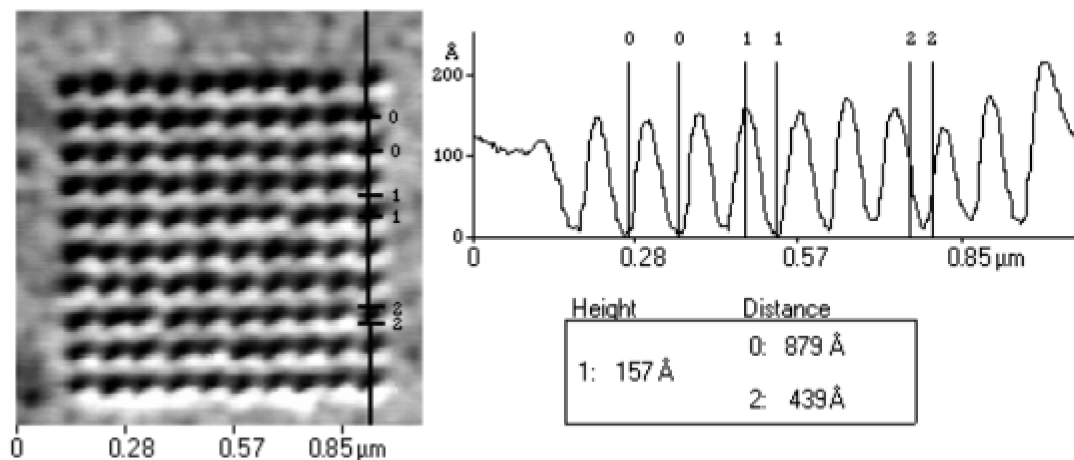

Fig. 2: Atomic force microscopy image and cross sectional profile of a periodic hole array in Si fabricated by AFM nanolithography. Array with 10 x 10 holes with a period of 88 nm, hole diameter of 44 nm and hole depth of 16 nm.

3. Summary of the Results

The minimal feature sizes that were made by the combined optical and AFM lithography process are shown in Fig. 2. The test pattern of a periodic hole array exhibits feature diameters of about 44 nm with an etch depth of about 30 nm. Minimal grating periods of 88 nm were achieved. Apart from such hole structures we have also fabricated lines with 45 nm line widths. The lines were drawn by putting a series of holes in very close proximity (less than 30 nm) to each other. In this way also more complicated structures can be made. The largest dot arrays we have produced so far consisted of several thousand individual dots and exhibited no apparent pattern distortions within fields of several micrometer in size. The big advantage of our approach is that is a mask-based lithography technique that is independent of the substrate and which allows the use of proven semiconductor processing steps. Therefore, we were able use our method for GaAs nanostructure fabrication just by modifying the final etching process.

References

[1] D. M. Eigler and E. K. Schweizer, “Positioning Single Atoms with a Scanning Tunneling Microscope”, *Nature*, Vol. 344, 1990, p. 524.

[2] E. S. Snow, P. M. Campbell, and F. K. Perkins, “Nanofabrication with Proximal Probes”, *Proc. IEEE*, Vol. 85, 1997, p. 601.

[3] M. Wendel, H. Lorenz, and J. P. Kotthaus, “Sharpened Electron Beam Deposited Tips for High Resolution Atomic Force Microscope Lithography and Imaging”, *Appl. Phys. Lett.*, Vol. 67, 1995, p. 3732.