Sharpened electron beam deposited tips for high resolution atomic force microscope lithography and imaging

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We employ the vibrating tip of an atomic force microscope as a lithographic tool to mechanically pattern a thin photoresist layer covering a GaAs–AlGaAs heterostructure. High aspect ratio electron beam deposited tips, additionally sharpened in an oxygen plasma, are used to minimize the dimensions of the fabricated quantum electronic devices. The fabrication parameters of the tips and the sharpening process are investigated. With these ultrasharp tips we are able to produce lines and holes with periods down to 9 nm in photoresist. In addition, the very sharp tips yield substantial improvements in the imaging mode. © *1995 American Institute of Physics.*

Scanning probe technologies employed for lithography have opened a new route to semiconductor device miniaturization, as they offer a large range of new lithographic techniques to the experimentalist. In contrast to other techniques such as electron beam lithography, the lithographic technique with an atomic force microscope (AFM) is not restricted to particular materials and works with very small proximity effects. An overview on various lithographic methods is given, e.g., in Ref. 1. Notable examples of recently developed lithographic technologies are the oxidation of H-passivated silicon surfaces with an AFM, followed by selective etching,[2] the direct modification of silicon surfaces and superconducting films with the AFM,[3] local modification of the conductivity in chalcogenide films by an AFM,[4] and the creation of a liftoff mask by plowing the first of two resist layers with the following development of the second layer.[5]

We have recently introduced a mechanical patterning technique of photoresist.[6] We use the structured photoresist as a mask for pattern transfer onto a two-dimensional electron system in a GaAs–AlGaAs heterostructure (HEMT). On a conventional predefined Hall bar structure we spin a highly thinned photoresist (Shipley 1805: thinner=1:70–1:15), resulting in a thickness of 3–15 nm. To reduce the viscosity of the resist to an optimal value, the resist is baked at 170 °C for about 1 h. To pattern the resist, the AFM tip, vibrating at its resonance frequency of about 300 kHz in the so-called tapping mode, is pushed towards the surface with an estimated force of about 1 μN. The force constant of the used cantilever is about 10 N/m. In this way, a mask consisting of holes or lines is created for the transfer process. The time necessary to fabricate an array of 20×20 holes is typically 2 min. Finally, the mask is transferred to the electron system by either wet chemical etching or by an ion beam irradiation technique. Details of the lithographic process,[6] the ion beam irradiation technique,[7] and the transport investigations of such produced quantum electronic devices[6,8,9] are given elsewhere.

A limiting factor in these lithographic techniques is the tip quality. Commercially available silicon tips[10] deliver excellent and constant results in the fabrication of hole arrays (also called antidot arrays) down to a period of 80–100 nm. To produce antidot arrays with even smaller periods, it is necessary to use sharper supertips, because the silicon tips are found to be of insufficient sharpness and durability to fabricate patterns with such small periods.

To overcome this problem we now employ electron beam deposited (EBD) tips for lithographic purpose. The EBD tips, used so far only for improved imaging,[11–16] are fabricated in a scanning electron microscope by focusing an electron beam onto a surface. The electron beam cracks organic molecules such as those found in pump oil and carbon containing material is deposited. Auger spectroscopy of EBD material[11] has shown that this material is mainly composed of carbon and oxygen (and possibly also hydrogen which cannot be detected in Auger spectroscopy). We deposit the EBD tips on already used AFM tips, covered with a thin (10–30 nm) gold layer to prevent charge buildup.

The most important preparation parameters of EBD tips are the electron beam current, the acceleration voltage, the deposition time, and the residual gas composition. Our results, typified in Fig. 1(a) indicate that the sharpest tips are generated at the minimal electron current (as measured in a Faraday cup) and the maximal acceleration voltage [Fig. 1(b)]. We note that electron currents larger than 1 nA do not produce tips anymore. An increased deposition time leads to a longer tip, but causes only a slight change in the tip diameter [Fig. 1(c)]. The gas pressure in the sample chamber has no significant influence on the tip topography in the investigated pressure region 1×10^{-5}–7×10^{-5} Pa. However, a higher pressure causes a higher growth rate of the tips, which is of the order of several 100 nm per minute. In contrast to other reports,[12,16] we did not find a significant dependence of the tip shape on parameters such as working distance, emission current of the cathode, or the magnification. Tests using an EBD tip as a tunneling tip indicate that our tips are insulating with resistances higher than 1 GΩ, in contrast to Ref. 11. We attribute this fact to the different residual gas composition in the electron microscope.

EBD tips optimized for lithographic application, are prepared with a beam current of 1–2 pA, a maximal acceleration voltage of 40 kV, and a deposition time of 2–5 min,

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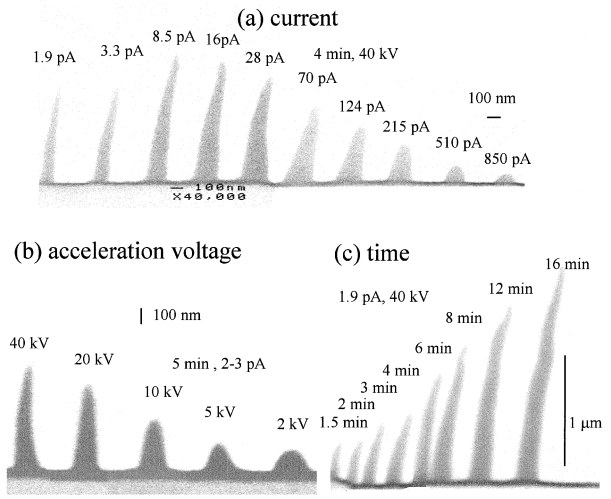

FIG. 1. Dependence of the shape of electron beam deposited tips on electron beam current (a), acceleration voltage (b), and deposition time (c). The sharpest tips are produced at the minimal current and the maximal acceleration voltage.

resulting in a length of 0.5–1 μm and a base diameter of about 100 nm. Figure 2 depicts an array of 18×18 antidots with a period of 55 nm in a 1.5 μm wide GaAs mesa, produced with an EBD tip. After preparation of the mesa and covering the device with a 5 nm thin photoresist film, the lithographic step is carried out. The sample is then etched for 8 s in the dilute wet chemical etchant H_2O:NH_4OH:H_2O_2=1000:3:1 at 0 °C, resulting in an etch depth of 3 nm, enough to deplete the electron system of a shallow HEMT structure below. The micrograph is taken after the photoresist is removed in acetone and by a dry etch

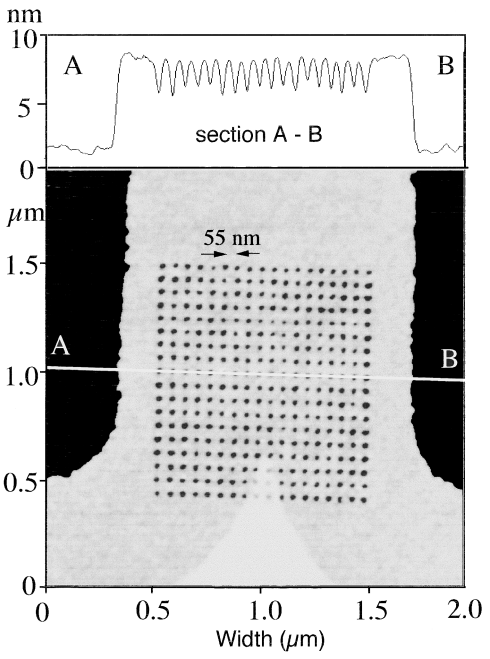

FIG. 2. Antidot array in a GaAs mesa. Depth and period of the holes are 3 and 55 nm, respectively. The mesa width is about 1.5 μm. The substrate wet etched after lithographic definition in the resist. Finally, the resist is removed and the AFM micrograph is taken. A cross section along the indicated white line is shown at the top.

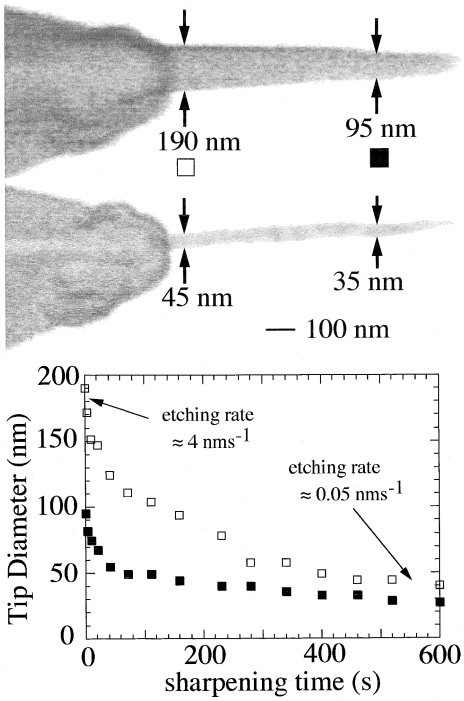

FIG. 3. Electron micrograph of an electron beam deposited tip before (top) and after (middle) 460 s sharpening in an oxygen plasma. The conical tip shape changes into a more needlelike form. The diagram below depicts the diameter of the tip, taken at the locations, indicated in the micrograph at different times during the sharpening process.

process in an oxygen plasma. The section plot along the indicated line shows that the holes are very homogeneous. EBD tips have a very good durability in the lithographic process. There was no degradation in hole quality even after the preparation of about 100 000 holes. Furthermore, a significant change in tip topography could not be observed with the electron microscope. With this kind of tip we are able to fabricate antidot arrays with periods down to 33 nm in GaAs and 15 nm in photoresist.

A further reduction in size of the fabricated patterns can be achieved by using EBD tips, which are additionally sharpened in an oxygen plasma. An electron micrograph of an EBD tip before and after the sharpening proces is shown in Fig. 3. It is obvious that the etching of the EBD tips is inhomogeneous. More material is removed from the thicker base than from the thinner end of the tip. The tip diameter measured at the base of the tip and at the end of the tip is depicted in the inset of Fig. 3. The etching process was interrupted numerous times and the diameter was investigated with an electron microscope. It can be seen that the etching rate decreases by about two orders of magnitude during the sharpening process and that the originally conical tip shape becomes more needlelike. A possible explanation of this behavior is the assumption of a rotationally symmetric material distribution in the tip in which a chemically very resistive core with a size comparable to the diameter of the generating electron beam is surrounded by a chemically ''softer'' coating. Such a shell structure could arise from a temperature gradient in the tip during the deposition process.

Although these tips seem to be very fragile they do not

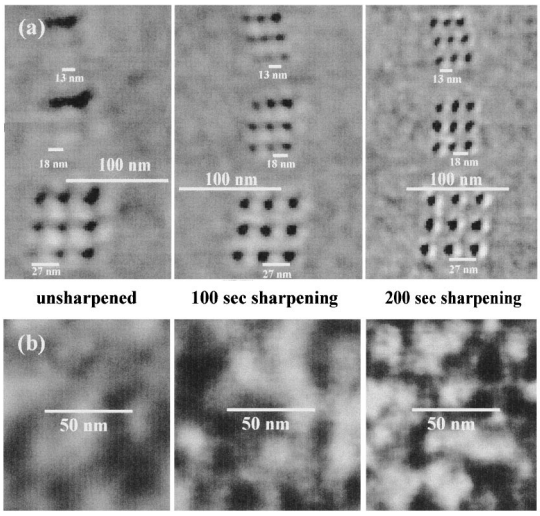

FIG. 4. Top: test structure consisting of several hole arrays with periods of 27 nm (bottom), 18 nm (middle) and 13 nm (top), written with an EBD tip before the sharpening process (a), after 100 s (b), and 200 s (c) sharpening in an oxygen plasma. The smallest fabricable period decreases by a factor of 2. Bottom: topography of a thin photoresist film taken with the corresponding tip above. More and smaller details can be resolved with the sharpened tip. The grey scale image reflects height variations of about 0.5 nm.

break during the lithographic process and deliver excellent results. Figure 4(a) depicts a test structure, consisting of three antidot arrays with the periods 27, 18, and 13 nm, which we use for the investigation of the lithographic resolution. This test structure is written into photoresist with an EBD tip before and after 100 and 200 s of sharpening, respectively. While the unsharpened tip fails in producing the arrays with an 18 and 13 nm period, the same tip succeeds in creating the array with 18 nm after a 100 s sharpening. After 200 s in the oxygen plasma even the smallest array with 13 nm period can be fabricated. The smallest period we have produced until now is 9 nm. Figure 4(b) demonstrates that, simultaneously, more and smaller topographic details of a photoresist film can be resolved in the imaging mode, indicating a decreasing tip radius. To record the images, the AFM is driven in the tapping mode to prevent soft materials such as photoresist from being damaged. A series of pictures of a thin photoresist film is shown, taken with a tip before and after sharpening. A sharper tip reaches deeper into holes and grooves in the resist. Therefore the apparent roughness (rms) of the resist layer increases from 0.30 nm for the unsharpened tip over 0.47 nm (100 s sharpening) to 0.61 nm after 200 s sharpening in the oxygen plasma.

In summary, we describe a very suitable technique for writing very small zero- and one-dimensional patterns on GaAs–AlGaAs heterostructures by AFM lithography. A substantial reduction of the device dimensions can be achieved by using electron beam deposited tips. These tips can be significantly sharpened in an oxygen plasma. The fabrication parameters of these tips have been investigated and optimized. With such optimized tips we are able to fabricate hole arrays with a period down to 9 nm in photoresist.

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