Nanolithography with a high-resolution STEM

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A high-resolution scanning transmission electron microscope (STEM) with a beam diameter approaching 0.6 nm has been adapted for the patterning of complex fine-line nanostructures. An IBM PC XT is used as the pattern generator to direct the scan electronics from a Cambridge Stereoscan 250 which have been interfaced with the scanning coils of the STEM. A study of the ultimate resolution of the newly designed acid-catalyzed resist poly(p-t-butyloxycarbonyloxystyrene) has been carried out. The STEM has proven to be a flexible tool in the fabrication of individual nanostructure devices for quantum transport studies in mesoscopic devices smaller than an electron phase-coherence length.

Introduction

The drive to miniaturize electronic circuits has provided a strong impetus for studies of the electrical and physical behavior of materials over smaller and smaller length scales. Quantum-mechanical effects lead to a breakdown in the classical electrical behavior typically observed in large devices when dimensions are reduced to lengths comparable to an electron phase-coherence length (typically 1 μm in metals at $T = 1$ K) [1]. Device miniaturization can also be limited by the physical properties of lithographic materials being used to pattern small-scale devices. Electrons are particularly useful in studying lithographic processes on the sub-100-nm size scale because of their short wavelength. The highest resist resolution has been achieved using various metal halides [2, 3], oxides [3, 4], and contamination resist [5], and sub-10-nm structures have been formed. In its high dose, negative mode, PMMA has produced linewidths of about 15 nm [6]. The use of these materials as resists for device fabrication, however, has been limited by their high electron dose requirements and the difficulty encountered in transferring patterns in the resists cleanly to an underlying substrate. A compromise between resolution and sensitivity is achieved with resists such as PMMA processed in the positive mode, in which liftoff structures about 12 nm wide are possible [7, 8]. Structures with linewidths of roughly 23 nm made using conventionally processed negative resists have appeared in the literature [9, 10].

This paper describes the adaptation of a high-resolution scanning transmission electron microscope (STEM) to fabricate complex patterns. An IBM PC XT Personal Computer is used to control beam position, blanking, and exposure time. A preliminary description of the resolution of the newly designed acid-catalyzed resist poly(p-t-butyloxycarbonyloxystyrene, or "t-BOC" for short, is given. Finally, various ways in which the STEM has been used to fabricate nanostructure devices for quantum transport experiments are also presented.

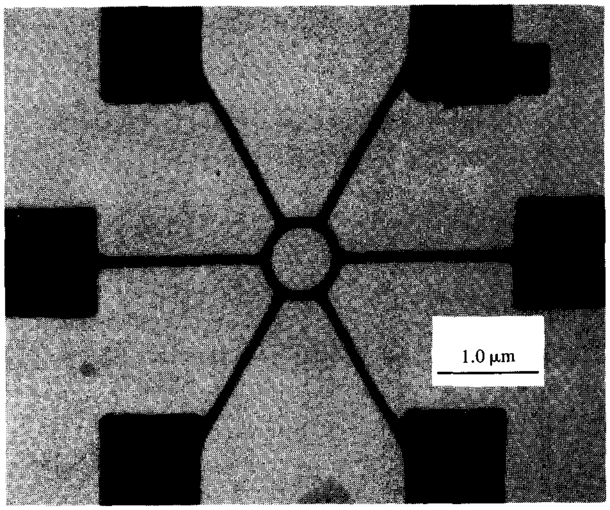
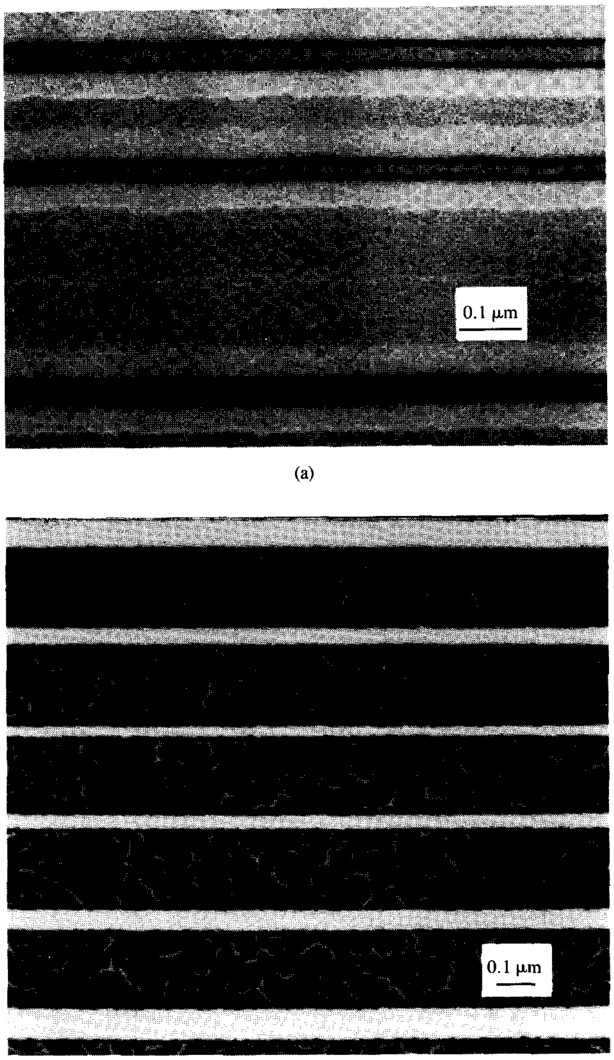


Figure 1

Nanostructure demonstrating the STEM's ability to pattern circles, angled lines, and rectangles.



(b)

Figure 2

Transmission electron micrographs showing resist developed in (a) negative mode and (b) positive mode. Both samples have been shadowed with metal incident at a 45° angle to the surface. The shadowed metal did not reach the substrate supporting the positive-mode resist.

Pattern generation with an IBM PC XT

The basic features of the STEM used in the work described in this paper have been described in detail earlier [11]. A LaB_6 cathode produces a beam of electrons which is focused by three magnetic lenses down to a minimum diameter of approximately 0.6 nm. The final lens was specifically designed for high-resolution probe applications, and has a relatively short focal length and small (approximately 5 μm × 5 μm) field size. The sample is placed between the pole pieces in the final lens. A detector positioned in the column behind the sample collects transmitted electrons. Beam blanking is accomplished by electrostatically deflecting the beam away from a plate positioned in the column above the final lens. The scan electronics from a Cambridge Stereoscan 250 were interfaced with the scan coils of the STEM. A PC XT was then interfaced with the scan electronics. A program was written to control the electron beam during exposure of a pattern. It was found that, in order to obtain maximum writing speeds, it was necessary that the PC generate pixels at its maximum rate. The program would then vary the step size between pixels written on a substrate in order to control exposure dose. In contrast, the more common method used by higher-speed pattern generators is to hold the step size fixed and to then vary the exposure time. High-speed pattern generators are frequently limited as to the shapes that they can write. In contrast, the PC program permitted writing circles, dots, and angled lines as well as rectangular shapes. An example of the STEM's patterning flexibility appears in **Figure 1**, which shows a hexagonal array of lines extending from a circle to rectangular pads.

Resolution experiments in poly(p-t-butyloxycarbonyloxystyrene)

The sensitivity of conventional resists is intrinsically limited, since a single electron can, at most, produce a single useful chemical reaction. To bypass this limitation, a new class of resists has been designed [12, 13] based on a chemical amplification scheme in which a single electron generates a catalyst which takes part in a number of useful reactions. In principle, extremely high sensitivities are possible. It might

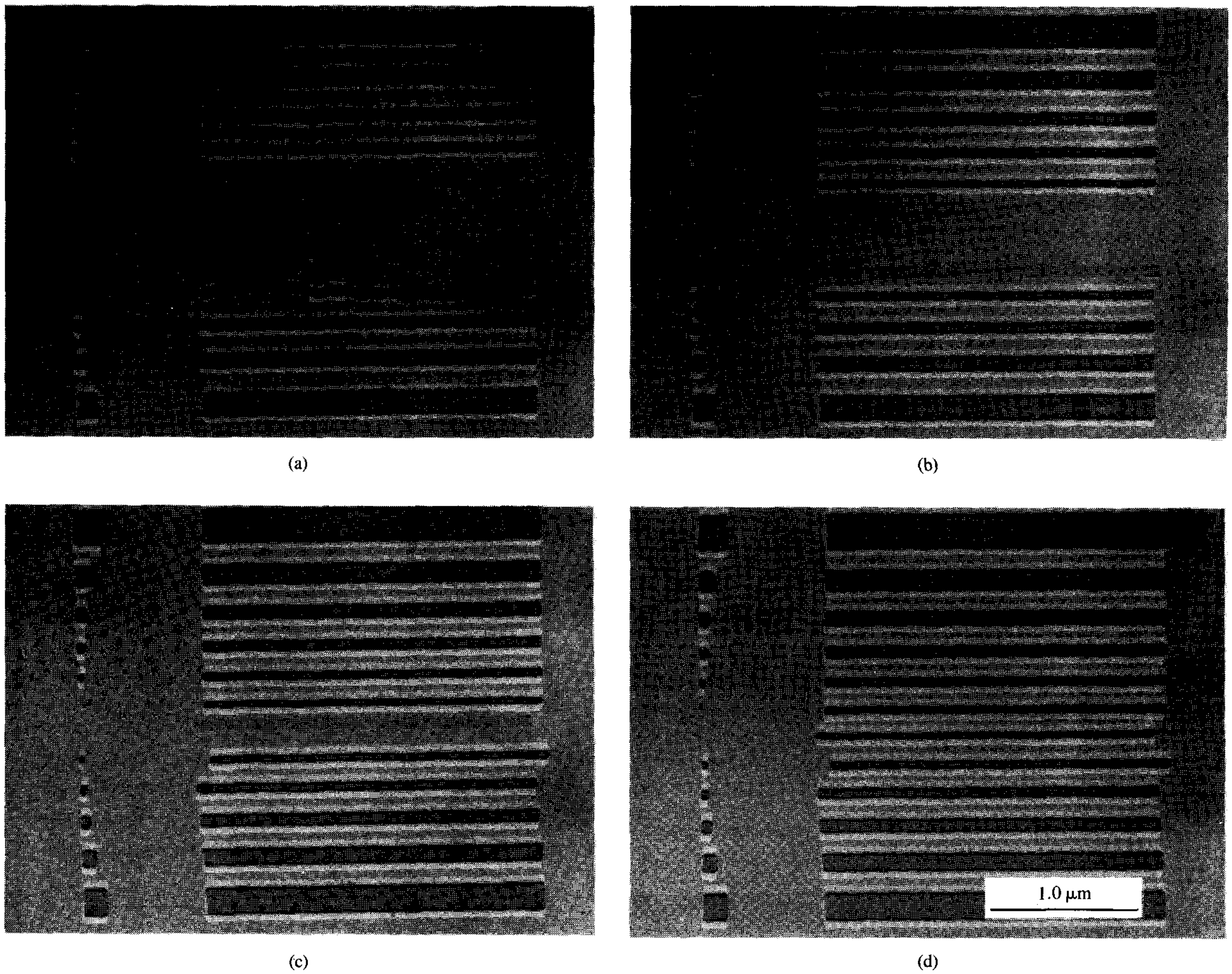

(a) (b) (c) (d)

Figure 3

Negative-mode t-BOC exposures ranging from (a) underexposed to (d) overexposed in patterns used to determine the exposure distribution. The developed resist is shadowed with metal incident at a 45° angle to the surface.

be intuitively expected that this high sensitivity would, however, be obtained at the expense of a decrease in resolution, due to diffusion of the catalyst. A resolution experiment in the STEM [14] using the acid-catalyzed resist poly(p-t-butyloxycarbonyloxystyrene), or "t-BOC," shows, surprisingly, that this is not the case.

The t-BOC resist is quite versatile in that it can be developed in both negative and positive modes simply by changing the developer [12, 13]. Exposing the resist generates a local concentration of strong acid. Baking the resist films after exposure results in acid-catalyzed cleavage and decarboxylation of the polymer side-chain functional group. The conversion of the nonpolar t-BOC functional group into more polar phenolic form results in the formation of a relatively polar polymer in all exposed areas. The proper nonpolar developer dissolves only the unexposed resist, leaving behind the exposed resist, while the proper polar developer dissolves only the exposed resist. Resists developed in both the positive and negative modes are shown in **Figure 2**.

The resolution of t-BOC was determined by exposing a thin layer of the resist in a pattern containing lines of various widths. The exposure of the pattern was repeated at a variety of doses. Analysis of the dose required to expose lines of different widths permits the determination of the exposure distribution [7]. This exposure distribution can be used to evaluate resolution in the same way that the Airy distribution is used to determine the resolution of a diffraction-limited optical microscope. This method for determining the distribution does not require measurements of linewidth. All that is required is a precise knowledge of the nominal written linewidths, which are set by the pattern generator, and a determination of which line was completely exposed at each exposure dose. The beam diameter in the STEM was always smaller than the minimum linewidths developed in the resist, and played no part in determining the minimum linewidths. Thin Si_3N_4 membranes were used as substrates to minimize backscattering effects. An accelerating voltage of 50 kV was used.

A typical exposure sequence is shown in **Figure 3** for t-BOC developed in the negative mode. Up to eight different exposure doses were used for each experimental run; four are shown in Figure 3. The lightest dose [Figure 3(a)] is slightly below that needed to barely expose the largest shapes. The heaviest dose [Figure 3(d)] was high enough to slightly overexpose the smallest shapes in the pattern. The dose to barely expose a large area of resist was approximately 5×10^{-5} C/cm^2. Under similar exposure conditions (50 keV electrons and thin substrates), positive-mode PMMA has been found to require doses roughly six times higher for development [7].

Results of an analysis of the data in Figure 3 are plotted in the exposure distribution shown in **Figure 4**. While it is clear that the distribution is not Gaussian, the half-width is the same as it would be for a Gaussian distribution with $\sigma = 15$ nm. As a check of the STEM operation, a calibration run was made using 17-nm-thick PMMA. The exposure distribution for this PMMA produced $\sigma = 15$ nm, in nominal agreement with previous PMMA results [7]. This latter result indicates not only that the PC XT-driven STEM is producing reliable exposure distribution data, but also that t-BOC has approximately the same resolution as PMMA, even though its sensitivity can be six times higher. These results suggest that resolution may be limited by something inherent in all organic resists, such as, perhaps, the range over which low-energy secondary electrons are created by high-energy electrons.

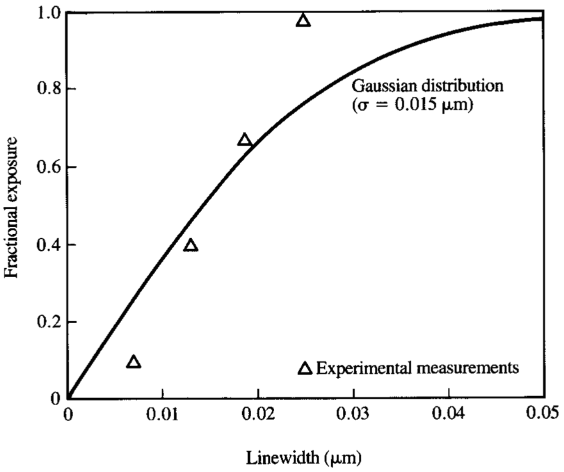


Figure 4

Exposure distribution for t-BOC resist processed in the negative mode (open points). The solid line shows the Gaussian exposure distribution expected for $\sigma = 0.015$ μm. A dose of 5×10^{-5} C/cm^2 was required to barely expose the widest lines in the exposure pattern.

Nanostructure device fabrication in the STEM

With a minimum beam diameter of roughly two atomic diameters, the STEM is an excellent instrument for studies of the ultimate resolution of resists. As a tool for nanostructure fabrication, however, it is somewhat limited because of its small (~5 μm diameter) focused-beam field size and its transmission mode detection, which requires thin-membrane samples to focus the beam. In spite of this, the STEM has demonstrated a remarkable flexibility in producing individual nanostructure devices for scientific studies.

One technique for forming nanostructures in the STEM that has been used with success involves the use of contamination resist. Since contamination resist is a negative resist [5], used to protect the underlying surface during an etching step, it is typically formed on top of a metal film deposited on top of a thin Si_3N_4 membrane. Contamination resist builds up when an electron beam strikes a layer of carbonaceous material, contaminating the otherwise clean surface of a sample. Rastering the electron beam results in the formation of a contamination resist line. The carbonaceous material in the resist can come from a variety of sources, such as dirty vacuum systems or oily vapors in the air. The rate of formation of contamination resist depends upon the "dirtiness" of a sample and on the electron-beam current and voltage, but in general is relatively slow compared to the rate for conventional resists. This lack of sensitivity permits the surface being patterned to be imaged before and after drawing contamination resist structures, in order to be certain of pattern registration. In contrast, conventional resists such as PMMA would be grossly overexposed by focusing on the area to be patterned.

Due to the lack of sensitivity of contamination resist, it would take a prohibitively long time to use the resist to pattern pads to make electrical contact between a nanostructure device and the outside world. Instead, pads are patterned using either optical lithography or electron-beam lithography in an electron microscope with a sufficiently large field size. Many copies of the pad pattern can be made at once, but when this is done the pads must be in a sufficiently general configuration to ensure their usefulness in the variety of new experiments that are invariably thought of only after the pads have been patterned. While a single pad configuration is rarely optimally suited to any particular experiment, making new samples with a different configuration for each new experiment is not practical because of the amount of time and effort involved.

An example of a device made with contamination resist is shown in **Figure 5**. Here, a ring with attached current and voltage leads has been drawn in contamination resist between PMMA pads. Ion milling removed all exposed metal, leaving a device with attached current and voltage leads underneath the resists. Before Al wires are ultrasonically bonded to the pads, the PMMA must be removed by ashing in an rf oxygen plasma.

Other resists, such as t-BOC, can be used in place of contamination resist to draw fine lines between the PMMA pads. While the use of these other resists results in much shorter exposure times and greater reproducibility than the use of contamination resist, the other resists can, depending upon the resists used, suffer from the problem of the pads partially dissolving when the top layer of resist is spun on. In addition, because of the much greater sensitivity of the top

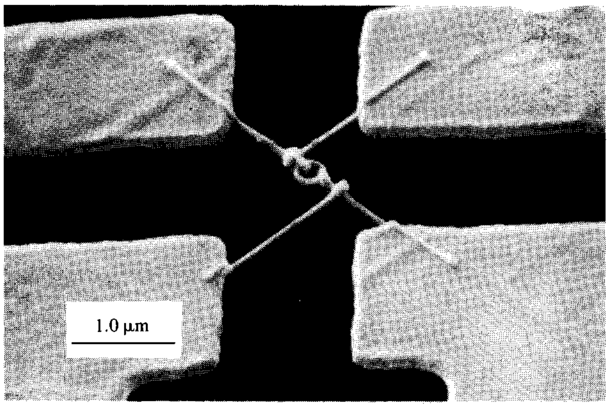


Figure 5

Scanning electron micrograph of a Au ring formed by drawing contamination resist lines between PMMA pads patterned on a Au film. Ion milling removed all exposed Au, leaving a metallic ring underneath the resists.

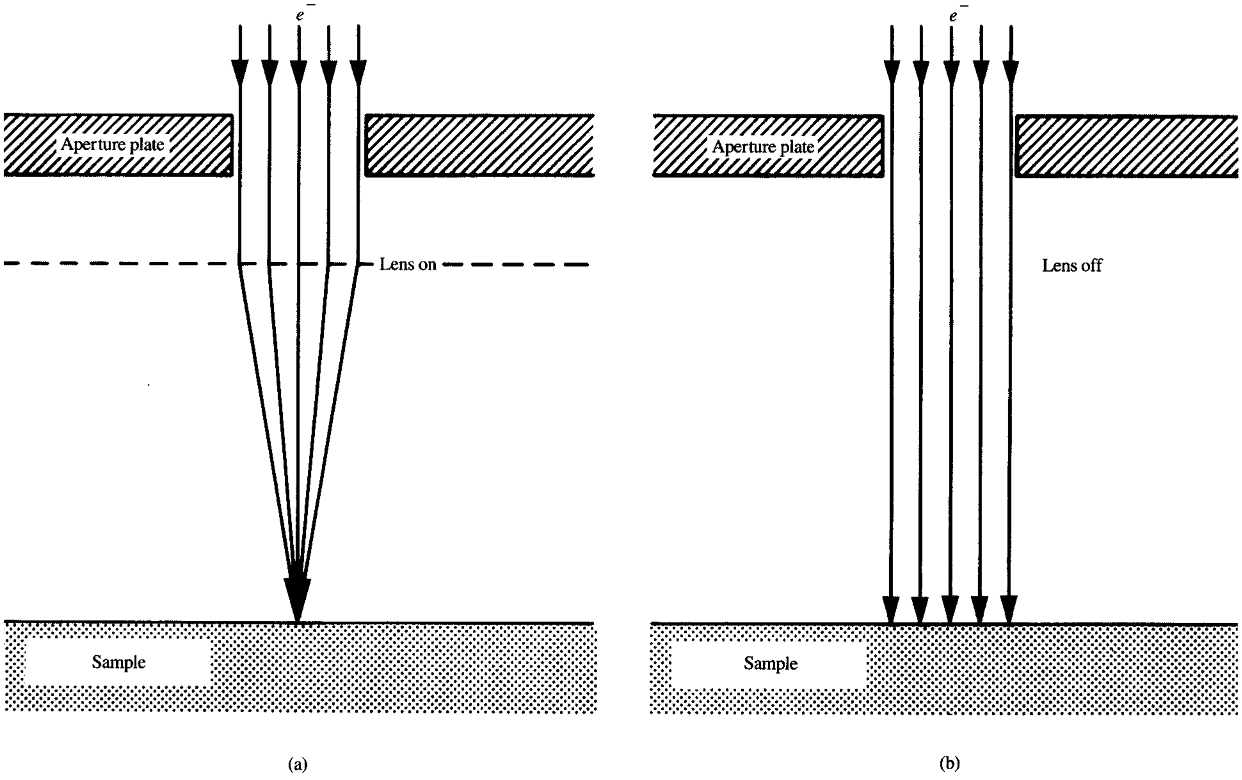


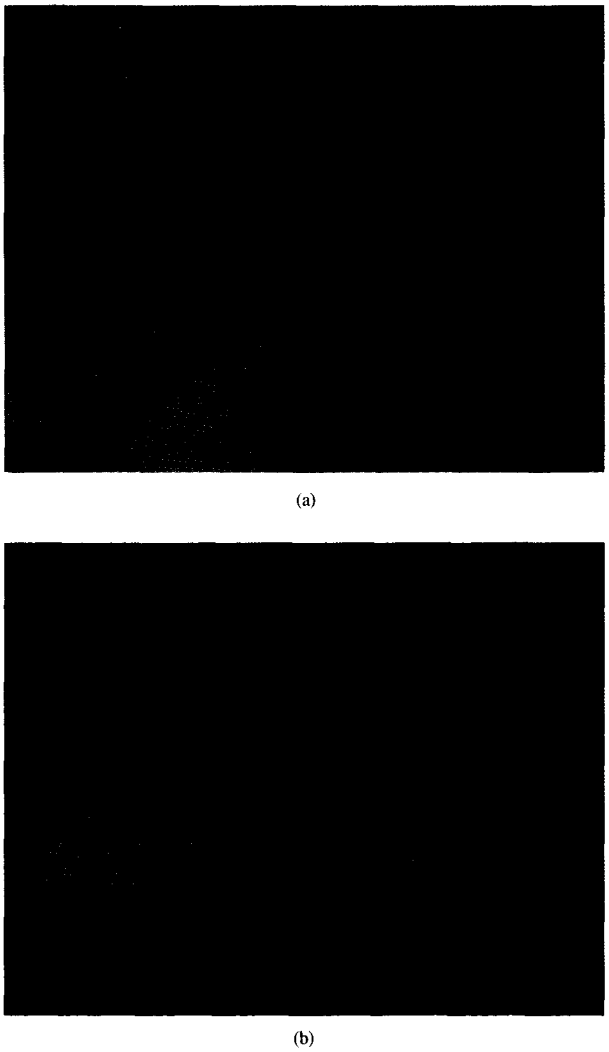
Figure 6

Schematic drawing to illustrate how the STEM can be used to pattern (a) fine lines when in a focused mode, and (b) coarse lines with widths comparable to the aperture diameter when in an unfocused mode. The drawing is not to scale.

layer resist with respect to contamination, the area on which the fine lines are to be drawn cannot be directly imaged for registration, as can be done with contamination resist.

As an alternative to procedures in which nanostructure patterns are exposed between preformed pads, a technique has been developed through which both the pads and the fine lines are patterned in the STEM (see **Figure 6**). First, with the final lens on [Figure 6(a)], the focused electron beam is used to draw a pattern out to the limits of the roughly 25-μm-diameter field that can be rastered. While the beam retains its minimum diameter only over a roughly 5-μm field, outside this region the broadened beam can still be used to expose resist. The patterning of the pads is then done by a technique similar to electron-beam proximity printing [15]. The final lens is turned off [Figure 6(b)] and unfocused electrons passing through an aperture 1.5 cm away from the sample expose the image of the aperture into the resist. The unfocused electrons originate from a 30-nm demagnified crossover image positioned in the microscope column approximately 28 cm from the aperture. From simple geometrical considerations, it is estimated that the resolution of the edge of the aperture image is about 1.6 nm. This high resolution in an unfocused image is a direct result of the short focal length of the final lens used in the STEM. When the image of the aperture is rastered to form a line, the dose received at any point on a cross section of a line is a function of the distance of the point from the edge of the line.

The number of pads that can be attached to a sample is limited by the number of aperture images that can be brought without overlapping within the 25-μm-diameter area that can be patterned with the final lens on. Since the smallest aperture that can be mechanically drilled in a 254-μm Pt aperture plate is approximately 11 μm in diameter, the number of pads is limited to eight when apertures are formed in this manner. While smaller apertures can be made by other processes, their use would be accompanied by a marked decrease in beam current as well as an increase in the minimum focused beam size due to diffraction effects. The overlap between focused and unfocused patterns is shown in **Figure 7(a)**. The field size of the STEM with the final lens off is a bit over 0.5 mm, which is sufficient to pattern pad areas large enough so that 25.4-μm-diameter Al wire can be ultrasonically wire-bonded to them. This is demonstrated in the partially bonded sample shown in **Figure 7(b)**.


(a)

(b)

Figure 7

Optical micrographs showing a sample patterned entirely in the STEM. The overlap between the pattern drawn with a focused beam and that drawn with the unfocused beam can be seen in (a). The radiating lines in (a) are 11 μm wide, the width of the aperture. Two 8-pad samples in the process of being wire-bonded with 25.4-μm-diameter Al wire are shown in (b).

The fragility of the thin membranes is a major drawback to patterning devices in the STEM, even though their use does reduce the number of backscattered electrons, leading to an improvement in micrograph resolution as well as a reduction in proximity effects in resist exposures. Thin membrane substrates have in the past been required for focusing, whether devices were being patterned on thin membranes or bulk substrates, because the electron detector was positioned for transmission-mode imaging. Recently, however, it has been shown [16] that it is possible to place electron detectors inside the final lens pole pieces above the sample and obtain high-resolution images using backscattered electrons. With this adaptation, the STEM can pattern and register on bulk substrates without requiring the use of thin membranes.

Summary

Interfacing a PC XT to control the beam positioning in a high-resolution STEM has resulted in a flexible tool for studies of the physical behavior of materials on the nanometer size scale and for the fabrication of submicron devices for quantum transport measurements. The STEM has been used to show that poly(p-t-butyloxycarbonyloxystyrene) resist, one of a new class of high-sensitivity acid-catalyzed resists, has a resolution of approximately $\sigma = 15$ nm. The use of a final lens with a short focal length, along with a relatively small final aperture, permits entire nanostructure devices, including pads, to be fabricated in the STEM.

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