Fabrication of 3 nm wires using 100 keV electron beam lithography and poly(methyl methacrylate) resist

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We report the fabrication of 3 nm NiCr wires on a solid silicon substrate. The process uses conventional 100 keV electron beam lithography and poly(methyl methacrylate) resist. The wires consist of short, continuous, lengths of metal that are attached at either end to 20 nm wide wires. Instead of exposing continuous lines in the resist, we blank the beam for several pixels to leave a gap. The resist in the gap is therefore exposed only by the secondary electrons from the neighboring regions that are directly exposed by the beam. The technique is repeatable and we demonstrate that it is possible to make 3 nm features on demand. © *1996 American Institute of Physics.* [S0003-6951(96)00403-2]

Electron beam lithography (EBL) has been instrumental in the drive toward the fabrication of ultrasmall structures. The smallest features that have been delineated were less than 5 nm, achieved by direct sublimation of several different salts on solid substrates.[1] Unfortunately it has not proved possible to transfer the written patterns from resist to a substrate and retain the resolution.

More conventional processing uses the positive resist poly(methyl methacrylate) (PMMA). Using this resist, the fabrication of 10 nm metal wires was demonstrated on membranes[2] and on solid substrates.[3] On solid substrates, the amount of electron backscatter from the substrate to the resist is small if the beam energy is high (typically 100 keV) and the pattern is sparse. Therefore the ultimate resolution of EBL on PMMA is limited by the degree to which primary and secondary electrons are scattered in the resist. The lateral straggle of the forward-scattered electrons is only a few nanometers in the resist. However, secondary electrons with energies as low as 5 eV can cut PMMA molecules (i.e., expose them)[1] and it has been demonstrated that secondary electrons may penetrate several nanometers into the resist.[4] Although this is the fundamental limit, resolution is also strongly affected by subsequent processing.

Recently, lines of 6–7 nm have been written in PMMA. This result was achieved using a highly optimized ultrasonic development process in a high contrast developer.[5] Using the Lennard-Jones potential to model the intermolecular forces in the resist, it was hypothesized that the attractive forces in the resist increased rapidly as the feature size dropped below 10 nm. This necessitated the use of ultrasonic agitation to liberate the exposed resist from the resist layer during development. Subsequent reactive ion etching with $SiCl_4$ and SiF_4 succeeded in transferring this pattern on to a GaAs substrate. Sub-10 nm metallic dots have also been made using EBL followed by the deposition and lift-off of 5 nm of AuPd.[6] However, the fabrication of metal wires through which current could be passed has not been demonstrated at this resolution. We present an exposure method that overcomes the lateral straggle and process related limitations and enables us to fabricate 3 nm wires using otherwise conventional techniques.

For this work, we used a converted JEOL 100CXII electron microscope with a spot size of 3 nm and a beam energy of 100 keV.[7] The system gives a beam current of 12±0.5 pA over the duration of the exposures. The silicon substrates were baked at 180 °C for 1 h to drive off any moisture, then spin-coated with a bilayer of PMMA. The bottom layer had a low molecular weight of 85 k and was 30 nm thick. The top layer had a higher molecular weight of 350 k and was 20 nm thick. The sample was baked at 180 °C for at least 2 h after the first coat and then overnight after the second coat. Prepared substrates were stored in the dark at room temperature.

To make the smallest features, the resist was exposed by a single pass of the electron beam, as shown schematically in Fig. 1(a). The diameter of the spot (which has a Gaussian electron intensity profile) is marked by the circles. The lines are exposed in a 100 nm period array for electron line doses in the range 0.8–2.8 nC cm^{-1}. The boundary of the region that is sufficiently exposed so that subsequent development will remove the resist is marked. The scan step per pixel is 2 nm. To control the exposure of a small portion of the line,

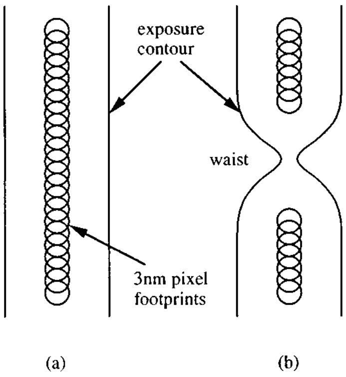

FIG. 1. (a) A conventional “single pass line.” (b) A single pass line from which several pixels have been missed out. The circles indicate the position of each pixel and the solid contour indicates the extent of the exposed resist.

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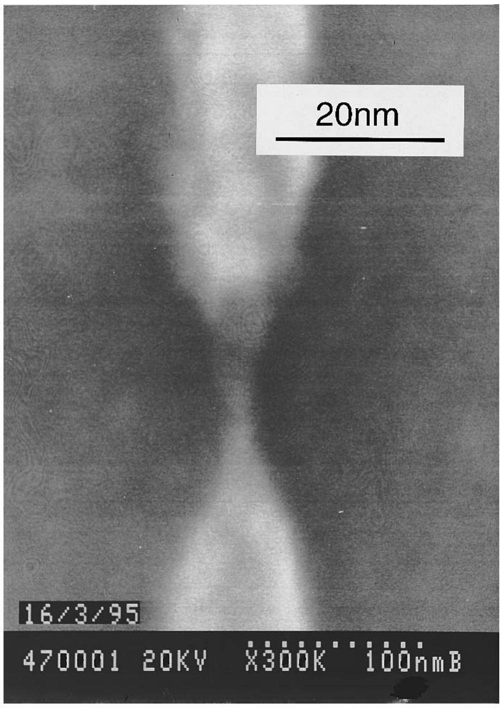

FIG. 2. A scanning electron micrograph of a 3 nm wire formed in the gap between two exposed regions. The postscript *B* on the scale marker indicates an additional magnification of $5\times$, thus the scale bar is 20 nm.

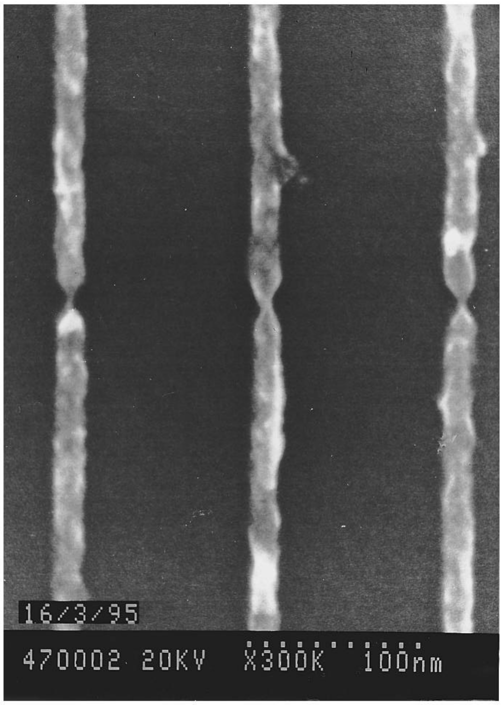

FIG. 3. The process is repeatable, allowing the fabrication of several wires simultaneously.

several (7, 9, or 11) pixels were blanked during the scan. This is shown in Fig. 1(b). The boundary of the exposed region has a waist in it due to the overlap of the radiation scattering on to the center of the gap from the neighboring regions.

After exposure the sample was developed in 3:1 IPA: MIBK for 30 s at 23 °C then rinsed in IPA and blow dried in filtered nitrogen. The sample was then mounted in an automatic dual shutter electron beam evaporator with a working distance of approximately 0.6 m. The NiCr (60:40) source was electron beam heated to its working temperature prior to opening the shutters. 10 nm of NiCr was deposited on to the sample at a rate of 0.4 nm s^{-1}. The metal was lifted-off by immersing the sample in warm acetone for 1 h followed by vigorous agitation with a Pasteur pipette. The sample was then rinsed in IPA and dried as before.

The resulting wires were examined in a Hitachi S-900 scanning electron microscope. Figure 2 is a micrograph of a single 3 nm wire. Such a wire might only have 10–20 atoms across its width. The wire width in the normally exposed regions is approximately 20 nm. In the middle region there is a narrow waist which has formed in the overlap region between the two exposed regions. This was achieved using a scan gap of nine pixels (18 nm) and a dose of 1.8 nC cm^{-1}. For the next lowest dose of 1.6 nC cm^{-1} the narrow wires are not formed and a clean break of 20 nm is made. However, at the next highest dose of 2.0 nC cm^{-1} waists are formed in the gaps with a width of approximately 6 nm. For doses above 2.4 nC cm^{-1}, the 20 nm wires are continuous. If the gap is reduced to seven pixels at a dose of 1.8 nCT cm^{-1} the 20 nm wires are continuous with no waists. Similarly, if the gap is increased to 11 pixels then a break is made.

The lateral straggle of the primary electrons at the PMMA/Si interface, established using a Monte Carlo simulator, is less than 5 nm at 100 keV. Since we need a very large dose to make the 3 nm wires (a dose of 0.8 nC cm^{-1} will expose a normal line), we believe that enough secondary electrons propagate into the region of the gap to expose the resist. The exposed material is thus removed not only from the top, but also from either side, during development. Because of this, ultrasonic agitation is unnecessary to remove the resist from the gap region. It is observed that the narrowest wires, presented in this letter, have an elongated appearance which is not consistent with the shape of the exposure contour shown in Fig. 1. We postulate that the shape of the wires is determined by the mechanism of grain formation on the substrate. On a free surface the NiCr grains are approximately circular with a diameter of 6–7 nm. When the atoms in the gap strike the substrate, they are mobile and move to join atoms on grains that are forming nearby. Atoms that remain in the gap are forced to form long rodlike grains that are pinned by other grains at each end; thus they do not follow the original profile of the resist.

This fabrication method is reproducible and Fig. 3 shows three narrow wires. To demonstrate further our capability to make 3 nm wires at will, Fig. 4 shows a wire positioned between two spars. The lithographic separation between the tip of each spar and the wire was 30 nm and the required dose was 1.8 nC cm^{-1} as before. When the separation was reduced to 20 nm the region was over exposed, due to backscattering becoming significant, and a solid metal junction was formed by subsequent processing.

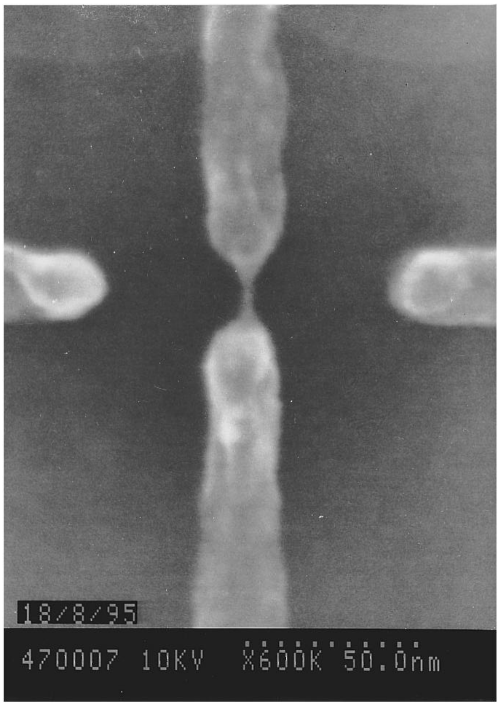

FIG. 4. The 3 nm wires can be positioned in close proximity to other structures.

More than ten chips with several hundred gap sites each have been processed. The results are consistent from chip to chip and the close examination of several chips gives a yield for the process of approximately 3%–5% for 3 nm wires. For the larger 6 nm wires the yield is significantly greater at 23%. This is not unreasonable for such small structures. We believe that the yield is limited by reliability of the aforementioned grain formation process and by the ripple that is observed on the edge of NiCr-coated lines made in PMMA. This ripple has an amplitude of approximately 2 nm over a range of 20–40 nm, which roughly corresponds to the fabrication scale.

In conclusion, we have developed a novel exposure control technique that has allowed us to fabricate 3 nm NiCr wires. The wires appear to be continuous in the scanning electron microscope, but attempts to measure electrical continuity have so far been unsuccessful. We believe that the portion of the PMMA resist in which the wire is subsequently formed is not radiated by primary electrons. Therefore the lithographic process is solely due to secondary electrons that penetrate into the critical region of resist. This is consistent with the known properties of electron beams in PMMA. The wires can be fabricated on demand and we have demonstrated that it is possible to position the wires alongside other closely spaced features.

[1] A. N. Broers, IBM J. Res. Dev. **32**, 502 (1988).
[2] S. P. Beaumont, P. G. Bower, T. Tamamura, and C. D. W. Wilkinson, Appl. Phys. Lett. **38**, 436 (1981).
[3] H. G. Craighead, R. E. Howard, L. D. Jackel, and P. M. Mankiewich, Appl. Phys. Lett. **42**, 38 (1983).
[4] S. A. Rishton, Ph.D. thesis, University of Glasgow, 1984.
[5] W. Chen and H. Ahmed, Appl. Phys. Lett. s **62**, 1499 (1993).
[6] W. Chen and H. Ahmed, J. Vac. Sci. Technol. B **11**, 2519 (1993).
[7] S. Thoms, S. P. Beaumont, and C. D. W. Wilkinson, J. Vac. Sci. Technol. B **7**, 1823 (1989).