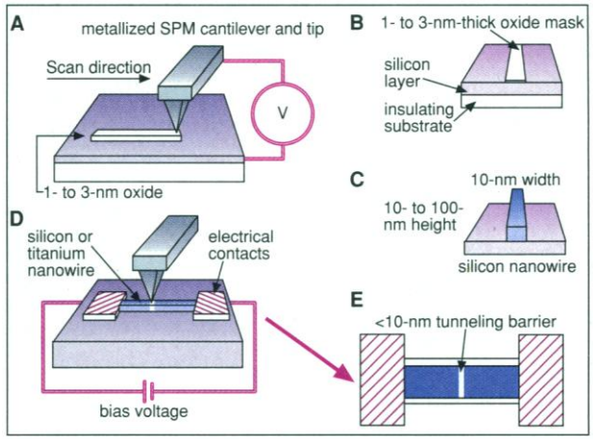
Device Fabrication by Scanned Probe Oxidation

J. A. Dagata

Nanoscience and nanotechnology have generated extraordinary interest in the past few years. This attention is largely a consequence of the invention of scanned probe microscopy (SPM) by Binnig and Rohrer (*1*). The development of SPM resonated with elements of Drexler's broad vision of nanotechnology, which itself was attracting considerable attention (*2*). The discovery that it is possible to use SPM not only for observing atomic-scale structure but also for manipulating material with atomic precision is a key element bridging SPM research activity and nanotechnology (*3*). After these initial scientific reports, significant efforts have been devoted to engineering functional nanometer-scale materials and devices by the direct use of SPM (*4*).

Progress in the case of one of these methods, the scanned probe direct oxidation technique, is particularly significant because of the remarkable level of cross-fertilization and incremental progress that has been achieved over the last few years by several laboratories worldwide. Snow and Campbell, on page 1639 of this issue, describe an important milestone in SPM fabrication of functional electronic device structures using this technique (*5*). Their work illustrates a defining feature of nanoscale engineering, that is, the integration of fabrication and measurement into a single process step is essential for attaining the desired performance. The collective experience and understanding needed to create a foundation for nanoscale engineering now exists. The profound simplicity and versatility of the direct oxidation technique has played a major role in making this possible.

Direct oxidation by SPM began at the U.S. National Institute of Standards and Technology (NIST) in early 1989 (*6*, *7*). The initial thrust of this work involved patterning single-crystal silicon and gallium arsenide surfaces. The basic idea is straightforward: Using a conducting SPM tip held either in contact with or within a nanometer of an electrically biased, stable, homogeneous substrate, one can induce a highly localized enhanced oxidation of the substrate (see figure). An oxide layer a few monolayers to a few nanometers in thickness rapidly forms in the substrate at the tip-sample junction. Typical linewidths are 10 to 20 nm, and writing speeds of up to 1 mm s^{-1} are possible, depending on the materials system, voltage bias between the tip and substrate, and available oxygen. Voltages used are 3 to 10 V, and both molecular oxygen and oxygen from adsorbed water layers, which exist under ambient conditions at the tip-sample junction, provide the reactant source. The presence of these impurities normally causes significant complications in other lithographic processes.



Thin oxide line. Scanning an electrically biased metallized SPM tip over the surface (**A**) can form a 1- to 3-nm-thick oxide line on many substrates. The pattern can then be used as an etch mask to create freestanding silicon nanowires (**B**,**C**). Further processing of the nanowire can also be carried out with the SPM tip (**D**) to create other confined structures.

The direct oxidation technique possesses remarkable features that are responsible for its success (*7*–*11*). (i) The method itself is simple to implement, relying on a process that is fairly well understood, because oxidation is a most ubiquitous chemical reaction. A number of metallic (titanium and chromium), semiconducting (crystalline, amorphous, and porous silicon; gallium arsenide), and insulating materials (SiO_2, Si_3N_4, and siloxanes) were patterned by this technique. (ii) The oxide is extremely stable relative to a typical native oxide. The conditions that exist at the junction ensure a strong driving force toward thermodynamically favorable bonding. (iii) The technique leads to sufficiently robust masks or final structures. The thermal and chemical stability required for subsequent processing by wet or dry chemical means is remarkable and, as a class, is probably unique to oxidation. The subsequent etching reactions required for forming nanostructures from the latent pattern for most materials means that a wide variety of materials are amenable to direct oxide patterning. (iv) The ability to operate at low voltage avoids proximity effects common to high-resolution electron-beam exposure. (v) Because of the method's simplicity and versatility, a diverse research base can implement these techniques, with some researchers taking an interest in the fabrication process itself and others using the process principally for device fabrication.

Steady progress has resulted in what has now become the fairly routine fabrication of functional devices. Technical improvements began with Snow and Campbell's use of metallized atomic force microscopy (AFM) tips in place of scanning tunneling microscopy (STM), which greatly improved reliability and increased writing speed, and with their demonstration that selective etching methods could be combined with epitaxial single-crystal silicon layers to form freestanding silicon structures up to 100 nm high from a latent pattern just 1 to 2 nm thick (*12*). They also demonstrated that silicon nanostructures are excellent masks for the etching of underlying heterogeneous layers, such as gallium arsenide and silicon dioxide (*13*). The next significant contribution occurred at Philips Laboratories in Europe: Researchers there extended this strategy of two-step etching by combining its use with amorphous silicon layers, which can be deposited onto a wide variety of metal and insulating films (*10*). This advance allowed the technique to be integrated into CMOS (complementary metal-oxide semiconductor) device processing, as first demonstrated by Quate's group at Stanford (*14*).

Meanwhile, Sugimura (now at Nikon in Japan) began investigating the oxidation of thin, titanium films (*8*). An advantage of this system is that when the film thickness was carefully controlled to 5 nm or less on an insulating substrate, SPM direct oxidation rendered exposed regions of the metallic film insulating. This advance made critically dimensioned device components possible in a single step through the formation of insulating constrictions in conducting titanium wires. Matsumoto and co-workers at the Electrotechnical Laboratory in Japan

The author is with the Precision Engineering Division, National Institute of Standards and Technology, Gaithersburg, MD 20899, USA. E-mail: john.dagata@nist.gov

reported device characteristics for a single-electron tunneling transistor (SET) junction formed in this way (*15*). Although the SET performance of these devices appears entirely convincing, the correlation of device size and quantum confinement has not yet been confirmed on devices based on titanium wires, which presumably reflects a contribution of grain size on performance. Problems such as these, where reliable control of electrical and dimensional properties at less than 10 nm becomes critical, demand that fabrication be coupled with in situ diagnostics. By monitoring a resistance increase in the wire during fabrication, Snow and Campbell have shown that routine and reliable control can be attained in this size regime (*5*). A distinguishing feature of SPM-based nanoscale technology, that which sets it apart from the incremental refinements of existing methods such as electron-beam lithography, is the promise of exceptional materials control. Such control, rather than linewidth control, is needed for investigations at the sub–10-nm level. Further progress will hinge primarily on the integration of fabrication with local diagnostic probes in order to fully optimize the process (*16*).

Direct oxidation by SPM, only recently at the frontiers of nanoscience, has arrived at the point where it has now become a powerful technique for the prototyping of novel device structures. This research is one aspect of a generalization of the scanned probe concept leading to its eventual role in advanced manufacturing. Multiple-tip arrays, in which large numbers of individual SPM instruments perform parallel fabrication and inspection over a large sample area, are now a reality. Quate's group at Stanford has demonstrated parallel lithography by coupling the direct oxidation process with a five-tip SPM array (*17*). As these developments continue to be explored in the research laboratory, industrial acceptance of scanned probe techniques is growing rapidly. Various types of critical measurements needed in production and process development for the semiconductor, magnetic recording, and polymers and coatings sectors have been identified (*18*). These applications have the potential to drive the creation of an integrated SPM-based technology employing both fabrication and measurement functions. The prospects for this will be determined largely by the extent to which nanoscience research and technological innovation become successful at anticipating each other's development paths.

References and Notes

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Small RNA Chaperones for Ribosome Biogenesis

Joan A. Steitz and Kazimierz T. Tycowski

Vertebrate ribosomal RNA (rRNA) genes undergoing active transcription are pictured in virtually every textbook of biology. These electron micrographs show spreads of chromatin derived from the nucleolus, the cellular compartment responsible for ribosome biogenesis. The nascent rRNA molecules that emanate from the DNA pinpoint the sites where synthesis begins and ends within the tandem repeats of rDNA (*1*). But these elegant visualizations of genes in action also convey an important message concerning the fate of the attached precursor rRNA (pre-rRNA) transcripts (Fig. 1). Because their lengths uniformly increase along each transcription unit [~13,000 nucleotides (nt) in mammals], significant RNA processing must not occur until synthesis is complete. Approximately half of pre-rRNA sequences are then discarded during the maturation of 18S, 5.8S, and 28S rRNAs (*2*). Although ciliates splice their pre-rRNAs, the processing pathway in vertebrates consists simply of an ordered series of endo- and exonucleolytic cleavages that carve away the spacer regions and trim the ends of the rRNAs. What nucleolar components orchestrate these cuts, and why do cells complete synthesis of an enormous RNA before embarking on the process?

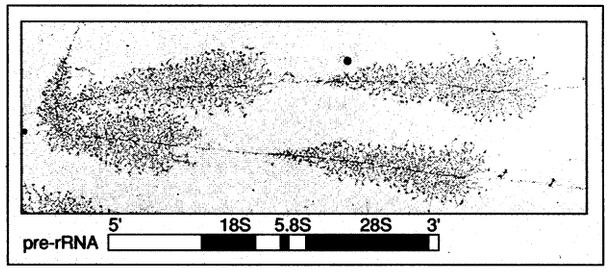

Fig. 1. Electron micrograph of actively transcribed rRNA genes from the *Triturus viridescens* oocyte. Each transcription unit shows growing pre-rRNA chains decorated at their 5′ ends with the so-called terminal knobs. Lower panel shows a schematic representation of vertebrate pre-rRNA. [Micrograph courtesy of O. L. Miller.]

Small nuclear ribonucleoproteins (snRNPs) have recently been identified as major players in eukaryotic rRNA processing (*3*). In addition to the well-studied snRNPs that assemble into spliceosomes to excise introns from pre–messenger RNAs (pre-mRNAs), cells contain distinct snRNPs localized in the nucleolus. The small RNAs in vertebrate nucleolar snRNPs range in length from 70 to more than 200 nucleotides. Many of these snRNPs contain a protein called fibrillarin, an autoantigen that is an abundant component of the fibrillar regions of the nucleolus. Current estimates of the number of nucleolar snRNPs in both vertebrates and yeast range from 50 to 100; approximately 20 vertebrate RNA sequences are now known.

So far, only three of the many vertebrate nucleolar snRNPs have been assigned functions in rRNA processing (*3*). The most

The authors are in the Howard Hughes Medical Institute, Department of Molecular Biophysics and Biochemistry, Yale University School of Medicine, New Haven, CT 06536-0812, USA.