

From Microscopy to Microfabrication

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Caltech President

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We sometimes forget how much technology has advanced during our lifetimes. These advances have been generated both by scientists, who are improving our understanding of the natural world, and by engineers, who create new devices, processes, and instruments in the manmade world. One example of this synergism between science and engineering is the scanning electron microscope, an instrument with which I have had some experience. It was used first for scientific investigations-to visualize objects to improve our understanding of nature. More recently, in a derivative form as an electron beam writer for mask making and direct exposure of integrated circuits, it has been used to fabricate microstructures that help to develop new technology.

First let's look at the advances in science that led to electron optics and to the electron microscope itself. De Broglie's hypothesis that particles could have a wavelike nature provided the stimulus for thinking that suitable lenses might be used to focus particles. Slightly earlier, Busch had shown that electrons could be focused by axially symmetric magnetic fields, and these two ideas allowed Ruska to develop the first transmission electron microscope in Germany in the early 1930s, a feat for which he recently shared the Nobel Prize in Physics.

Even earlier (in 1929), a German named Stinzing had filed a patent for a *scanning* electron microscope, in which a finely focused electron beam scans across the sample, but the technology to build it did not exist at that time. Knoll in Germany worked on a rudimentary scanning electron microscope in the mid-1930s, and von Ardenne, another German, actually constructed a transmission scanning electron microscope in the late 1930s. This may have been the stimulus for Zworykin, Hillier, and Snyder, working at the RCA laboratories in the very late 1930s and early 1940s, to construct a rather sophisticated scanning electron microscope. However, by having the scanning electron beam incident perpendicular to the sample surface, they were unable to get good contrast, and they abandoned the idea to pursue others that they deemed more promising.

After World War II, C. W. Oatley at Cambridge University in England and his graduate student, Dennis McMullen, developed a scanning electron microscope (SEM) that had the sample inclined at an angle to the electron beam, used backscattered electrons as the signal source, and amplified these with a beryllium-copper electron multiplier in the demountable vacuum system. This instrument used electrostatic lenses, was built of war-surplus electron tubes, and was a remarkable instrument, considering that it was put together by one graduate student in less than four years. Ken Smith followed McMullen on this instrument. He made it work better and explored the fields of application for which it might be appropriate. The third student in Oatley's group at Cambridge was Oliver Wells, who was given the task of building a second scanning electron microscope, which he used to investigate fibers, among other applications. In 1955 I arrived at Cambridge University and was the third student to use the original McMullen

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McMullen's dissertation contained this diagram (right) of a scanning electron microscope. Below is the instrument that McMullen built, which Everhart inherited in 1955, the third graduate student in Oatley's Cambridge lab to work on it.





microscope, as modified by Ken Smith. My task was to investigate contrast formation in the scanning electron microscope, a topic that was very educational and that three years later resulted in an acceptable PhD thesis. I would like to explain a bit about my experiences there and how they led to subsequent developments that have been fascinating to me and, I believe, useful for many people.

The schematic diagram of a scanning electron microscope at left is the one that Dennis McMullen actually used in his PhD thesis. The electron gun had a tungsten hairpin filament cathode, which operated well in a demountable vacuum system of 10⁻⁵ to 10⁻⁷ torr. The lenses were electrostatic; the deflecting field, which was also electrostatic, was inserted before the second lens to enable the second lens to have a very short focal length. The image was formed by signals generated by the primary electron beam, which were amplified and used to modulate the intensity of a cathode ray tube, which was scanned synchronously with the electron beam in the microscope. In this way there was a one-to-one correspondence between points on the sample surface and points on the face of the cathode ray tube. The ratio of the size of the image on the cathode ray tube divided by the size of the raster scanned on the sample provided the magnification of the microscope. In essence, the scanning electron microscope is a closed-circuit television system.

Shown at left is the instrument as I inherited it. This looks very different from one that you would buy commercially today or even from the first commercial SEM. The extra-high-tension power feed is shown at the top left. Notice the wooden dowel, which has a metal electrode and an insulated wire attached to it. This dowel was moved up to the top when you wanted the first electron lens to have the minimum focal length, and down to the bottom when you wished to ground the center electrode and remove the lens entirely from the system. You could adjust the focal length and the voltage on the center electrode of the electron lens by moving the dowel to an intermediate position. I had not learned in college-taught physics that wooden dowels were good resistors and could be used in this way to vary voltage. Dennis McMullen was ingenious, had imagination, and, because of his limited budget, used the materials that were at hand. These characteristics are important in university research even today.

McMullen believed that the signal he was detecting was produced by backscattered electrons, the primary, high-energy electrons scattered through large angles by atomic nuclei in

In the secondary electron detector known as the Everhart-**Thornley detector** (right), low-energy secondary electrons emitted by the sample are easily deflected and can be attracted to a gridded collector and there accelerated into a plastic scintillator by a small positive voltage. A light pipe carries the scintillation light to a photomultiplier, which amplifies it and produces a video signal.

Two images of an etched piece of aluminum made with primary (backscattered) electrons (top) and secondary electrons (bottom) are shown at right. The low-energy electrons' curved paths allow the microscope to "see" into the crevice.



the sample, which, because they have high energy, travel in relatively straight trajectories. On the other hand, Smith believed it likely that secondary electrons (those emitted by the sample when hit by the primary electron beam) were producing much of the signal that he collected with the secondary electron multiplier. I set about to determine which signal was the most important and what differences, if any, could be seen by using these quite different signals from a common sample.

In the detector system that I used for backscattered electrons a large solid angle was subtended between the sample and a plastic scintillator, which would produce light when struck by electrons. This light was carried by a light pipe to a photomultiplier, which amplified the light and provided the video signal from the backscattered electrons.

The diagram above shows a collector system for secondary electrons. It also used a plastic scintillator and a light pipe, which guided the light to the same photomultiplier, but here the light was produced by secondary electrons that were attracted to the scintillator by a small positive voltage placed on the copper grid and accelerated to about 10 keV to produce light. The backscattered electron image from an etched sample of aluminum is shown in the top micrograph at left. By moving the backscattered scintillator to one side, you could image exactly the same surface with secondary electrons (bottom, left). Because the secondary electrons have low energy, they are easily deflected and follow curved trajectories. They can be extracted from



The scanning electron beam and the electronic structure of a semiconductor's p-n junction interact. In a cross-section (left) through a biased germanium-indium p-n junction, the applied voltage difference across the junction translates into a contrast difference in the image, revealing the iunction's exact location. At the same time the hole-electron pairs created when the beam sweeps across the junction show up as a large spike (right) on an oscilloscope monitoring the current through the junction.

deep crevices, and you can "see" into the holes. Because backscattered electrons follow straight trajectories, a line-of-sight path did not exist from these crevices to the detector, and the holes appeared dark. Both types of signals are still used today. The secondary-electron detector, with slight modifications, has been used in most commercial SEMs and is often referred to as the Everhart-Thornley detector.

What could we do with this new technique? One of the samples we thought would be interesting to examine with the SEM was a semiconductor containing p-n junctions. Surface effects caused the locations and functioning of these junctions to be poorly understood at the time, yet they were quite important. (The transistor had been introduced in the late 1940s.) The biased germanium-indium alloyed p-n junction shown above had been polished perpendicular to the junction; by putting a voltage across the junction, we could determine exactly where the junction was, using a contrast induced by the difference in voltage between the two sides of the junction (a topic explained in my thesis). Also, when we monitored the current through the reverse-biased junction, we observed a very large current when the beam swept across the junction. This is due to electron-beam-generated holeelectron pairs, and in later work was called the electron-beam-induced current.

When I joined the faculty of the University of California at Berkeley after receiving my PhD from Cambridge in 1958, I had no desire to work on scanning electron microscopy. For one thing, I had no microscope available in the United States. A second reason was that the microscope I had used at Cambridge was not very reliable, and I didn't want to become involved with all those equipment difficulties again. And a third reason was that we had sent the micrographs of biased junctions to some semiconductor scientists at a major U.S. company and received word back that there was absolutely no interest in this technique among anyone working in semiconductors. Foolishly, I believed this.

By 1960, however, I was beginning to think that there might be some value in returning to this field because I had heard about the possibilities of integrated circuits. This idea, which, as far as I can tell, was conceived independently by Jack Kilby at Texas Instruments and Bob Noyce at Fairchild Semiconductor (who recently won the Draper Prize for this work), had the desirable feature of allowing several different electrical components to be integrated into a single circuit, so that separate electrical connections did not have to be provided between them. Our previous work with biased junctions indicated that the SEM might have very useful applications in analyzing integrated circuits. So, in 1962 I teamed up with Oliver Wells at Westinghouse Research Labs in Pittsburgh to help construct the first scanning electron microscope in an American corporate research laboratory.

Several people had assured me that passivated integrated circuits were covered with a layer of glass, which charges up under electron bombardment, and that therefore there would be no hope of observing voltage differences on the This scanning electron micrograph (top) of an early-1960s integrated circuit shows three transistors as blackbordered squares. **Applied voltage**induced contrast causes the transistor elements to appear as various shades of gray, and the junctions between them can be seen clearly as can the bonds to the electrical leads. A close-up of a mid-1960s transistor (middle) shows the isolation region (I) between it and its neighbors, its elements emitter, base, and collector—and the leads (E, B, C) associated with each element. Adding the secondary signal to that current also provides information about the surface of the integrated circuit (bottom) as well as the junctions underneath.







Our previous work with biased junctions indicated that the SEM might have very useful applications in analyzing integrated circuits.

surface of such a device by using an electron beam. But I had faith that we could do this. In Cambridge I had observed aluminum samples, and it is well known that aluminum is covered with aluminum oxide, although the oxide is only a few tens of angstroms thick. When we inspected our first integrated circuit at Westinghouse in 1962 and immediately saw voltage contrast, I had to explain this apparent paradox. The answer is electron-beam-induced conductivity through the glass layer. Later calculations proved that the primary beam had enough energy to penetrate the glass, creating conductivity in the insulator by exciting electrons from the valence band of the insulator to the conduction band. At top left is a scanning electron micrograph of an integrated circuit of the 1962 era with voltages applied, showing that one can easily determine the position of the junctions and get a very good idea of the quality of the electrical bonds as well.

After a year at Westinghouse I returned to Berkeley, where a scanning electron microscope was constructed along similar lines, using some commercial electron guns and lenses, and homebuilt magnetic deflection coils that were outside the vacuum. With Don Pederson and Paul Morton, we established the first integrated circuits laboratory at a U.S. university. The micrograph at left in the middle, made using electron-beam-induced currents, shows a midsixties transistor; you can see the isolation region (between this transistor and others in the integrated circuit), the emitter, the base, and the collector leads, as well as the junctions between



At Westinghouse Research Lab, Everhart (right) and O. C. Wells use the scanning electron microscope that was first operated in December 1962.

these regions. By mixing the secondary signal with this electron-beam-induced current, as shown in the bottom micrograph on the previous page, we could get information about the surface of the sample as well as about the junctions underneath. We had demonstrated that there was a considerable amount of information that could be determined, and thus the use of scanning electron microscopy to help in the development of integrated circuits was launched.

Berkeley later obtained one of the first commercial scanning electron microscopes in the U.S. through the efforts of Fabian Pease, and we examined many different samples in it. The original home-built SEM was connected to a computer by Noel MacDonald, and was used for early experiments on electron beam lithography. Electron beam lithography held much greater potential for miniaturization than photolithography, which was used up to the mid-1970s to create the masks for defining the patterns of the several layers of an integrated circuit. Commercially developed electron beam exposure systems for writing masks have gradually taken over much of the mask making and have led to much progress in miniaturization since then.

Indeed, Richard Feynman's prophetic speech on miniaturization ("There's Plenty of Room at the Bottom," E & S, February 1960) included a challenge to reduce a page of a book to an area 1/25,000 smaller in linear scale. This was finally accomplished by a Stanford grad student in the fall of 1985—using electron beam lithography to etch a text on an area 5.9 micrometers square (E & S, January 1986). The student, Tom

This is the way both science and engineering progress—we build on the accomplishments of one another.





Integrated circuits aren't the only application of the SEM. It's an essential tool in the study of embryonic development; the fertilization of a sea urchin egg is shown in the top micrograph. And the SEM allows geologists to "see" into meteorite inclusions. The lower picture is the first ever made of a platinum-rich nugget (called a Fremdling) cracked out of an inclusion in the Allende meteorite. These tiny balls of highly concentrated metals are thought to contain samples of the first atoms to have condensed out of the newly forming solar system. Both of these are secondary electron images.

Newman, working with the previously mentioned Fabian Pease, was involved in research to enhance electron beam lithography for writing masks for VLSI chips.

It's obvious that a great deal of technology is involved in making integrated circuits and in inspecting them. What is not generally appreciated by the public at large (or even by scientists) is to what degree technology drives science. The old reasoning that the scientist discovers new knowledge and that this new knowledge is then applied to make new technology is only partly true. Without the technology of integrated circuits and high-speed computers, many of the scientific experiments undertaken today would not be possible, and scientists would be severely limited in discovering new knowledge. Without some of the techniques of information theory that were developed because of engineers' interest in communication, the decoding of DNA would be proceeding at a much slower pace. Most of our knowledge of the biological world below the resolution of the light microscope has been achieved using the electron microscope, an instrument developed by scientists and engineers, which has provided the means to understand molecular biology and a great deal of the structure of cells and of more elementary biological units.

There have been many advances in scanning electron microscopy since the days of the early instruments I have reviewed here. We have a much better understanding of the information generated by the scanning electron beam now than we did when McMullen started his work in

1949. I am indebted to my many colleagues at Cambridge and to my graduate students at Berkeley who worked with me on some of these topics, to my colleagues at Cornell who contributed significantly to submicron fabrication, as well as to the many colleagues around the world who have worked in these fields over the last three-plus decades. This is the way both science and engineering progress-we build on the accomplishments of one another. In order for America to remain competitive, we need to invest more in building the equipment and instruments that make possible more extensive and more rapid advances in science and technology. We must also recapture the sense of urgency in this process.

Subsequent to his significant work in the development and application of the scanning electron microscope at Cambridge and Berkeley, Tom Everbart took on some administrative posts (dean of Cornell's College of Engineering and chancellor of the University of Illinois at Urbana-Champaign) before becoming president of Caltech in 1987. The Everbart-Thornley secondaryelectron detector had preceded him here, however, and it continues to be an essential part of scanning electron microscopes used on campus. This article was adapted from a talk delivered to the College of Fellows of the Institute for Advancement of Engineering in 1988.