A New High Resolution Reflection Scanning Electron Microscope

A. N. Broers

Citation: Review of Scientific Instruments **40**, 1040 (1969); doi: 10.1063/1.1684146
View online: http://dx.doi.org/10.1063/1.1684146
View Table of Contents: http://scitation.aip.org/content/aip/journal/rsi/40/8?ver=pdfcov
Published by the AIP Publishing

obtained with some scattered light correction. A trace shown in Fig. 12 illustrates the order of performance achievable.

The shift in the baseline is associated with disturbance in the 20 cm effective solution path length during the photoflash. Without any photoflash, the baseline due to the measuring flash did not shift, thereby demonstrating the stability of the detecting system. The possibility of part or all of the shift being ascribed to a long lived transient is ruled out by scanning over longer times.

ACKNOWLEDGMENTS

It is a pleasure to thank L. Bradley and his group in Pure Physics Electronics for invaluable advice and for providing the printed circuits. The author also wishes to thank G. Charette for his enormous help in construction of much of the circuits, G. Kronmueller for advice and cooperation, and A. Couture for expert construction of the elliptical flash cavity. The author is indebted to Professor G. Porter, F.R.S., who contributed much to the author's understanding of the subject through many stimulating discussions.

THE REVIEW OF SCIENTIFIC INSTRUMENTS VOLUME 40, NUMBER 8 AUGUST 1969

A New High Resolution Reflection Scanning Electron Microscope

A. N. Broers

IBM T. J. Watson Research Center, Yorktown Heights, New York 10598

(Received 18 February 1969, and in final form 10 April 1969)

A new high resolution reflection scanning electron microscope has been built with a lanthanum hexaboride cathode electron gun, a contamination-free vacuum system, and in which the deteriorating effects of vibration and ac stray field interference have been reduced below significant levels. The microscope produces a minimum probe diameter of 30±7 Å with a current of 10^{-12} A at a working distance of 7 mm and an accelerating voltage of 23 kV. Point-to-point resolution of 50±10 Å has been measured in scanning reflection.

INTRODUCTION

THE reflection scanning electron microscope and its many applications have been described in a review article[1] by Oatley, Pease, and Nixon. This article includes a description of the work of Pease and Nixon (also reported in 1965[2]) who built a three magnetic lens scanning electron microscope for reflection scanning microscopy which produced a minimum probe diameter of 50 Å. The probe contained 8×10^{-13} A at an accelerating voltage of 30 kV and a working distance of 5 mm. The instrument used a tungsten hairpin cathode and a conventional oil diffusion pump vacuum system. The electron optical performance of the microscope was close to theoretical predictions, the minimum possible diameter for such a system with the same electron optical characteristics and accelerating voltage, as calculated from the formulas of Smith,[3] quoted below, being about 35 Å. One example of a point-to-point resolution of 100 Å in scanning reflection on a solid sample was shown, but in general the best point-to-point resolutions observed in scanning reflection were between 150 and 200 Å. It is possible, however, that in some cases detail below 150 Å was obscured by specimen contamination and the authors mention that contrast due to changes in secondary emission was quickly masked by contamination.

Thornton[4] has described the use of an ion pump with a commercial scanning electron microscope and noted a reduction in contamination rates. This microscope, however, has not been used for high resolution studies. Crewe[5] has reported the use of an ultrahigh vacuum scanning electron microscope with a field emission cathode. This microscope has been designed for transmission scanning microscopy though and has not been used to date for high resolution reflection scanning microscopy. A minimum probe diameter of 20 Å has been reported at a focal length of 1 mm. For a thorough coverage of scanning electron microscopy literature, see the bibliography compiled by Wells.[6]

This paper describes a new reflection scanning electron microscope which uses a lanthanum hexaboride cathode electron gun[7,8] and is pumped with an ion pump. The lanthanum hexaboride cathode has about two orders of magnitude greater life than the 0.13 mm tungsten hairpin

[1] C. W. Oatley, W. C. Nixon, and R. F. W. Pease, Advan. Electron. Electron Phys. **21**, 181 (1965).
[2] R. F. W. Pease and W. C. Nixon, J. Sci. Instrum. **42**, 31 (1965).
[3] K. C. A. Smith, Ph.D. dissertation, University of Cambridge, (1956).
[4] P. R. Thornton, in *Record IEEE 9th Annual Symposium on Electron, Ion, and Laser Beam Technology*, Berkeley (1967), R. F. W. Pease, Ed. (San Francisco Press, 1967), pp. 145–156.
[5] A. V. Crewe, J. Appl. Phys. **39**, 5861 (1968).
[6] O. C. Wells, in Ref. 4, pp. 412–438.
[7] A. N. Broers, Appl. Phys. **38**, 1991, 3040 (1967).
[8] A. N. Broers, J. Sci. Instrum. (J. Phys. E) Ser. 2, **2**, 273 (1969).

and is capable of producing an electron beam with approximately five times the maximum brightness produced by a tungsten hairpin cathode under similar conditions. In the design of the new microscope, great attention was paid to eliminating the deteriorating effects of vibration, ac stray field interference, and specimen contamination. A minimum probe diameter of 30 ± 7 Å has been measured with a current 1×10^{-12} A at 23 kV and a working distance of 7 mm (focal length 1.6 cm). Point-to-point resolution in scanning reflection of 50 ± 10 Å has been measured and many examples of resolutions of about 100 Å have been obtained on suitable specimens.

THEORETICAL ELECTRON OPTICAL PERFORMANCE

The microscope is conventional in that it uses three magnetic lenses to demagnify the crossover formed by the electron gun. For reflection scanning electron microscopy the final lens has to operate at a relatively long focal length so that the specimen can be placed outside the magnetic field of the final lens and the low energy secondary electrons collected efficiently. This long focal length, together with the poor contrast in the secondary electron signal, are the major factors that limit the resolution of a scanning reflection electron microscope compared to that possible from a scanning transmission instrument. In the new microscope the final lens is operated at a working distance of 6–10 mm and demagnifies the crossover formed by the second lens by a factor of about 20:1. This demagnification makes the aberrations of the first two lenses unimportant compared to those of the final lens, and the aberrations of the first two lenses can be neglected in making a theoretical estimate of the instrument's performance. The final lens has a design spherical aberration coefficient (C_s) of 1.8 cm[9] and a chromatic aberration coefficient (C_c) of 1.1 cm[10] at a working distance of 7 mm. Using these data and the brightness of the electron gun which has been measured at 5.6×10^5 A/cm²/sr at 12 kV[8] (corresponding to a cathode emission density of 25 A/cm² and a cathode temperature of 1750°C) the characteristics of the electron probe produced by the instrument can be calculated from the formulas of Smith.[3] Smith assumed that the square of the diameter of a focused electron probe was equal to the sum of the squares of the diameters of the disks of confusion due to spherical aberration, chromatic aberration, astigmatism, and diffraction. Smith combined this calculated probe diameter with the brightness formula of Langmuir[11] to arrive at the following expressions for the optimum operating aperture α_{opt} and the relationship between the probe diameter (d_0) and the probe current i.

[9] G. Liebmann, Proc. Phys. Soc. (London) **68B**, 682 (1955).
[10] Ref. 9, p. 737.
[11] D. B. Langmuir, Proc. IRE **25**, 977 (1937).

The diameter of the electron probe is defined as the distance between opposing points where the current density is one-fifth of the maximum value,

$$d_0^2=P/\alpha^2+C\alpha^6+Q\alpha^2$$

$$\alpha_{\text{opt}}=(Q^2+12CP)^{\frac{1}{2}}-Q/6C,$$

where

$$P=i/B+(1.22\lambda)^2,$$

$$B=5.65VJ_c\times10^3/T,$$

$$C=(\tfrac{1}{2}C_s)^2$$

$$Q=(C_c\delta V/V)^2+Z_a^2.$$

λ is the wavelength of the electrons, V the accelerating potential, T the cathode temperature, J_c the cathode emission density, δv the electron velocity spread, and Z_a the distance between the two line foci when the image is astigmatic.

For the instrument under discussion these formulas yield an α_{opt} of 4.75×10^{-3} rad and a d_0 of 27 Å for a beam current of 10^{-12} A and an accelerating voltage of 23 kV. It is assumed that no astigmatism exists and that $\delta V=0.25$ V. 10^{-12} A is the minimum beam current that can be used in the SEM if noise-free images are to be obtained in a reasonable period of time. Oatley *et al.*[1] have calculated that a noise-free 350 line picture can be formed in 5 min with this current, and periods longer than 5 min would not only be inconvenient, but would place excessively stringent requirements on the stability of the lens current and high voltage electronic supplies.

ELECTRON BEAM COLUMN

Figure 1 shows the electron microscope column, table, and vacuum system. The major components of the microscope column, the electron gun, the three magnetic lenses, the three lens spacers, the high vacuum valve, and the electromagnetic scan coils, are prealigned. External refer-

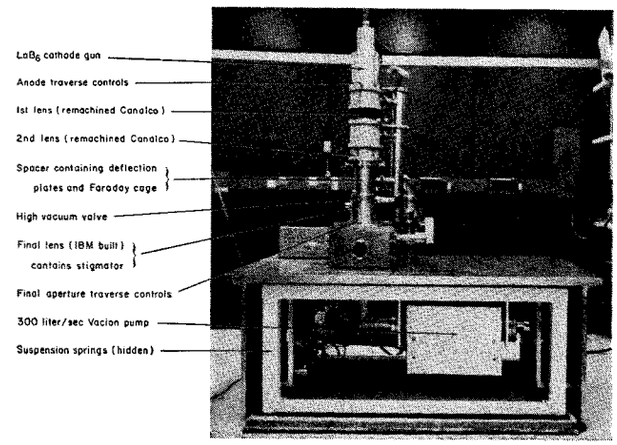


FIG. 1. New scanning electron microscope, high resolution reflection SEM with LaB_6 cathode electron gun.

ence surfaces on the electron gun and the magnetic lenses are aligned with the relevant internal surfaces to better than 0.0076 mm and on the final lens to better than 0.005 mm. The various column components fit together so that the total measured off-axis error over the full column length is less than 0.038 mm. Over-all parallelism is better than 0.02°. The first two lenses are commercially available magnetic lenses[12] which have been remachined to meet the required mechanical tolerances for the prealigned system. They have pole piece bore diameters of 2.8 mm and gaps of 3 mm. The final lens was made especially for the microscope and has a 5 cm first bore diameter, a 1 cm final bore diameter, and a gap of 5 mm. The bores of the final lens were honed and lapped until they were round to better than 0.13 μ and aligned to better than 2.5 μ. These tolerances are sufficient to ensure that the lens is anastigmatic as determined from Archard's[13] interpretation of Sturrock's[14] calculations. The final lens protrudes 12 mm into the specimen chamber allowing the electron collector to be positioned so that its field "sees" the surface of the specimen even when large flat specimens are placed parallel to the face of the pole piece. This is particularly important when the microscope is used to examine integrated circuits on silicon wafers.

The lens spacers are made from thick walled mild steel tubing and contain several concentric Mu-metal tubes for magnetic shielding. The high vacuum valve which is placed above the final lens is equipped with a Mu-metal tube which can be rolled into the bore of the valve when it is open. The over-all column shielding keeps deflection due to ac stray field interference to approximately the same magnitude as the probe diameter. At this level its effect can be eliminated, without appreciable distortion of the image, by synchronizing the line scan to the interfering frequency while recording micrographs.

Scan coils are placed in the bore of the final lens which also contains an eight pole electromagnetic stigmator. A set of deflection plates is included in the first spacer for blanking the beam. The only movable parts in the column are the gun anode and the final lens stopping aperture. Both these components are positioned from outside the vacuum with micrometers. The final lens aperture defines the operating beam aperture for the instrument. Three other 1.5 mm diam spray apertures are prealigned into the column several inches above each of the lenses to prevent the beam from striking the lens bores or the inside walls of the column.

The spacings in the column are as follows: gun anode to first lens gap 23 cm, first lens gap to second lens gap 20 cm, and second lens gap to final lens gap 33 cm. The first two lenses can be operated at a minimum focal length of about 3 mm, allowing a total demagnification of 105 000 for a final working distance of 8 mm. The gun crossover diameter is estimated to be approximately 20 μ so this demagnification is more than necessary to produce the smallest desired spot sizes.

The alignment surfaces on the top of each of the first two lenses are exactly the same diameter so the first lens and second spacer can be easily removed from the column and the column then operated with only two lenses. This is useful for larger spot sizes. The internal components of the final lens, including the final apertures, can be removed for cleaning and replacement by removing the final pole piece from inside the specimen chamber without further disturbing the column.

VACUUM SYSTEM

Specimen contamination is particularly important in the SEM because it not only produces false contrast by altering the secondary emission characteristics of the surface under examination, but the coating effect of the polymerized film can obscure fine surface detail. A layer of contamination several hundred angstroms thick can be formed during a preliminary investigation of a surface at high magnification in a typical oil pumped system, and this layer can dull the fine detail on the surface to the extent that the signal-to-noise (S/N) ratio in the collected signal falls below a detectable level and the detail can no longer be seen. This is particularly important for surface detail below 200 Å which cannot generally be seen when the microscope is operated in the "visual mode" and which only appears when the final micrograph is obtained over a relatively long period of integration.

To reduce the effects of contamination as much as possible in the new microscope, without going to the inconvenience of extensive cryogenic pumping or of a bakable system and metal gaskets, an ion pump is used for high vacuum pumping and a combination of mechanical and absorption pumps for roughing. The ion pump also has several advantages from the point of view of vibration isolation which will be discussed later. The mechanical pump is used to reduce the pressure to about 10^{-2} Torr, which takes less than 1 min, and the absorption pump to pump further down to 2×10^{-4} Torr at which point the system is opened to the ion pump. This procedure keeps the exposure of the system to the oil in the mechanical pump at a minimum and allows the absorption pump to be used many times between bakeouts. A zeolite trap is also included in the roughing line to reduce oil contamination. The ion pump has a capacity of 270 liters/sec and is equipped with a magnetic shield which reduces the magnetic field at the specimen chamber of the instrument due to the pump to less than 0.5 G. The vacuum is sealed throughout with Viton O-rings. A high

[12] Canal Industries Corporation, Rockville, Maryland.
[13] G. D. Archard, J. Sci. Instrum. 30, 352 (1953).
[14] P. A. Sturrock, Phil. Trans. Roy. Soc. A243, 387 (1951).

vacuum valve above the final lens provides access to the specimen chamber without turning off the electron gun and allows the gun to be run continually ensuring stable performance. This is particularly important with the LaB_6 cathode gun which has a relatively long warmup time. Under normal conditions, pressures of 1×10^{-6} Torr in the electron gun and 5×10^{-7} Torr in the specimen chamber are obtained. The specimen chamber can be pumped from atmosphere to an operating vacuum of 2×10^{-6} Torr in 4–5 min, and the upper column, including the electron gun, can be pumped to the same pressure in 8–10 min. Despite the relatively high specimen chamber pressure no specimen contamination is observed except in cases where the specimen itself is already coated with a layer of hydrocarbons, as, for example, with semiconductor samples still coated with the remnants of a resist layer, or in cases where the specimen itself is the source of contamination. This is often the case with organic specimens.

At specimen chamber pressures above 1×10^{-6} Torr it is necessary to use a biased mesh between the specimen chamber and the ion pump, or to close partially the high vacuum valve in that part of the system, to exclude the cloud of electrons and ions formed in the ion pump from the chamber. If this is not done, the stray electrons are collected by the secondary electron collector and can produce background noise equivalent to the detected signal.

VIBRATION ISOLATION

The microscope column, the specimen chamber, and the Vacion pump are all mounted on a heavy aluminum table which is suspended from a rigid steel frame by four steel springs. The springs extend 20 cm under load providing a resonant frequency of 1.2 Hz. The total suspended mass is about 0.9 tons (metric). Suspension springs were chosen because they can be controlled more easily than compression springs for large extensions and because they provide good lateral as well as vertical isolation unlike many of the pneumatic rubber isolation units. The Q of the suspension system is too high to leave the table undamped without the risk of random impulses generating oscillation at the resonant frequency. Rubberized horsehair is used for damping, and the damping deteriorates the vibration attenuation from 25:1 with no damping to around 20:1 damped. Excited oscillations at the resonant frequency decay in 1–2 sec. To minimize the coupling of vibration onto the suspended table from external sources, the vacuum roughing line is completely disconnected during operation, and care is taken to decouple the high voltage cable to the electron gun. The only other connections to the suspended system are the flexible air hoses used to activate the automatic vacuum valves, and flexible cables to the various beam controlling components. The Vacion pump has the advantages over an oil diffusion pump in that no roughing line is needed after initial pumpdown, no water cooling connections are required, and there are no impulses from the boiling oil.

The basic stage used for translating the specimen is a ball-slide unit which allows precise orthogonal movement of the specimen over a range of 2.5×5 cm. This stage is the most sensitive part of the microscope to vibration, however, and a second unit is used for very high resolution microscopy. With the second stage, the specimen holder rests on a tray which is bolted directly below the final lens. The holder is pushed about orthogonally on the tray by a square rod attached to the basic specimen stage. The rod fits into a slightly oversize hole in the specimen holder allowing the rod to be backed off after locating the specimen in the desired position, thus eliminating all mechanical contact between the specimen and the base of the chamber. This stage together with the spring suspension system reduces interference due to vibration below a detectable level.

ELECTRONICS

The microscope high voltage supply is a commercial unit[15] with a measured stability of better than 2 parts in 10^5 over 10 min. The lens supplies are also commercial units[12] with similar stability characteristics. In practice after complete warmup no focus drift is observed for periods up to 1 h. The scan generators, display electronics, beam deflection yoke, and stigmator are those designed for the Cambridge Instrument Company Stereoscan. To make the scan coils and stigmator compatible with the "clean" vacuum system, they were placed in a vacuum tight container before placing in the bore of the final lens.

EXPERIMENTAL PERFORMANCE

The measured electron optical performance of the instrument is in close agreement with the theoretical predictions. Figures 2 (a) and (b) show two portions of a silver calibration grid viewed in scanning transmission from which it was estimated that the probe diameter was 30 ± 7 Å. The accelerating potential was 23 kV, the beam current 1×10^{-12} A, the working distance 7 mm, and the beam aperture 4×10^{-3} rad. No astigmatism correction was needed in obtaining this result. To increase the edge contrast a stopping aperture was placed after the specimen to prevent most of the electrons scattered through the

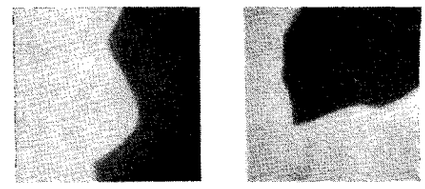
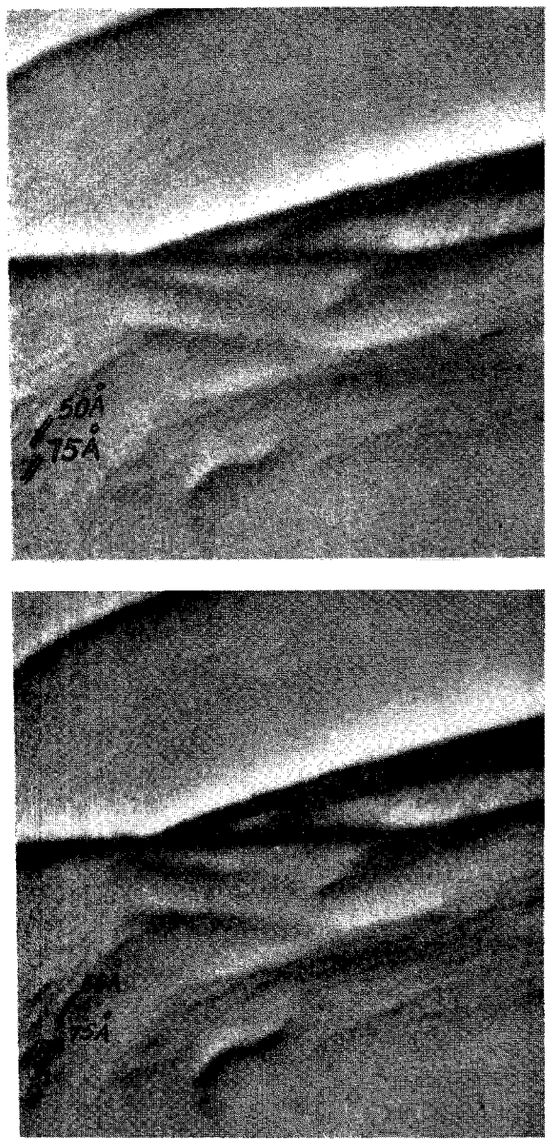


FIG. 2. Silver calibration grid in transmission, 200 000×.

[15] Brandenburg Ltd., South Croydon, Surrey, England.

edges of the specimen grid from reaching the scintillator electron detector. For this measurement, the first two lenses were operated at a focal length of 10.8 mm. The total demagnification produced by all three lenses was 8200 giving an estimated final spot size of 25 Å for a gun crossover diameter of 20 μ. The gun crossover diameter was measured from the observed spot size with only the final lens operating.

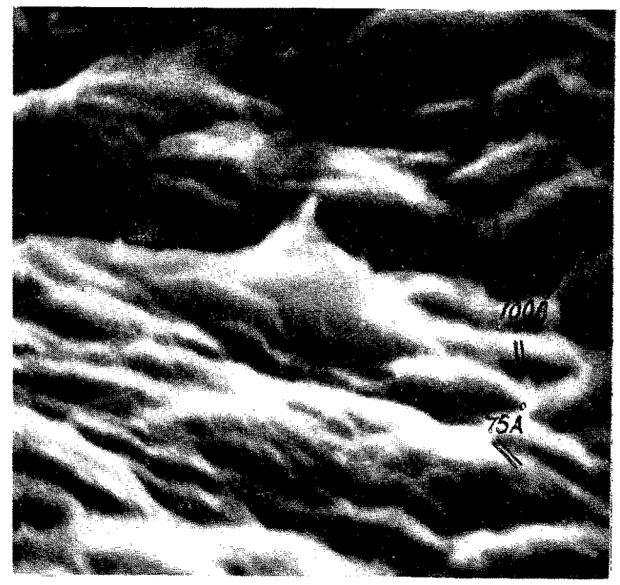
As expected the point-to-point resolution measured in scanning reflection has not been as good as would be estimated from the probe diameter and a criterion similar to that used in optics ($1.22\lambda/2$) where the probe diameter would be equivalent to the wavelength of the optical radiation. Such a relationship would predict a resolution



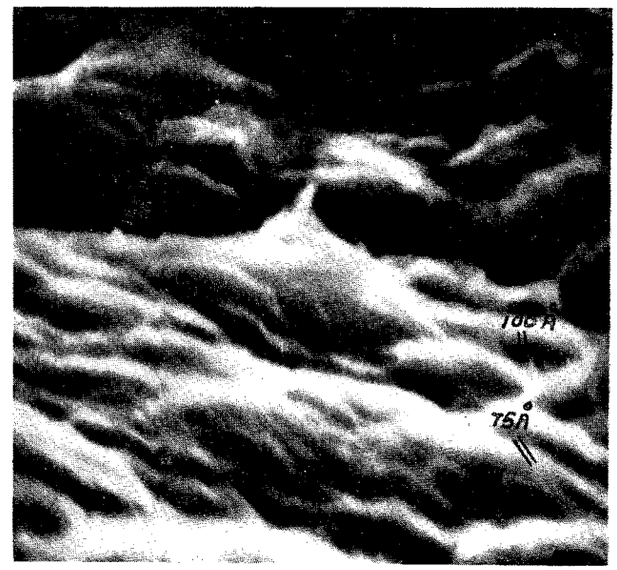
(a)

(b)

FIG. 3. Surface of silver calibration grid coated with 100–200 Å gold, exposure 70 sec, 117 000×.



(a)



(b)

FIG. 4. Surface of lanthanum hexaboride sample, exposure 90 sec, 90 000×.

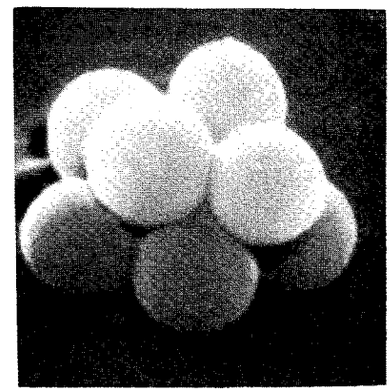


FIG. 5. 0.26 μ latex spheres, exposure 90 sec, 66 000×.

capability of 18 Å for a probe diameter of 30 Å, but would also assume that sufficient contrast was obtained between the points to be resolved and the surrounding surface. Unfortunately, in many cases this assumption is not valid in scanning reflection microscopy particularly when the points to be resolved are shallow protrusions on an otherwise smooth surface. Everhart, Wells, and Oatley[16] proposed that this deterioration in resolution was due to the secondary electrons generated as the high energy electrons reflected inside the sample move back towards the surface.

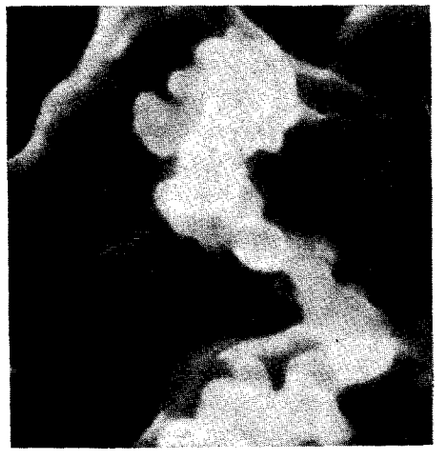


FIG. 6. Unidentified portion of human marrow specimen, exposure 60 sec, 55 000×.

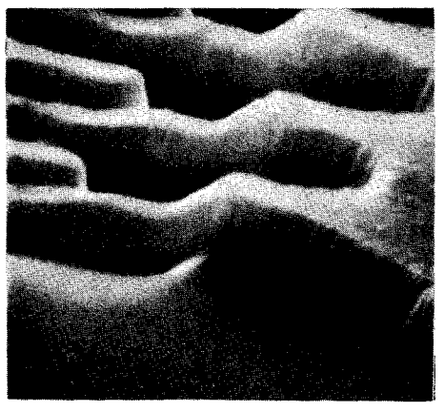


FIG. 7. Low angle of incidence view of $\frac{1}{2}\,\mu$ strip width silicon transistor structure fabricated with electron beam techniques 30 250×.

These secondary electrons emerge from a much larger area (approximately 1 μ^2 for typical accelerating potentials) than the impact cross section of the electron beam and do not contribute useful information about specimen detail smaller than 1 μ. They, therefore, reduce the S/N ratio for this fine detail and increase the contrast level required before it can be detected. This effect appears in practice as a general increase in the noise level in high magnification micrographs, where the resolvable picture element is smaller than 1 μ and is the most likely explanation for the apparent loss in resolution. The best point-to-point resolution measured with the new instrument to date is 50±10 Å as shown in Figs. 3 (a) and (b). Many cases of resolutions of 100 Å and below have been recorded and an example of such resolution is shown in Figs. 4 (a) and (b). In measuring point-to-point resolution, two micrographs were always obtained and the point separation seen in both cases, eliminating the possibility that the points were artifacts due to noise. The contamination-free environment of the microscope allows such measurements to be made with more freedom than would be possible in an oil pumped system. The magnification was checked either with latex spheres of known size (see Fig. 5), or more accurately, by moving the specimen a set distance with the specimen stage micrometers, noting the image movement at low magnification (100×) and using the scan current attenuators to determine the higher magnifications. Figures 6–8 (device shown in Fig. 7 fabricated by Hatzakis[17]) are high magnification micrographs obtained with the new instrument.

FIG. 8. Surface of plated copper film, exposure 60 sec, 30 250×.

ACKNOWLEDGMENTS

The author would like to acknowledge the assistance of W. W. Blair who was responsible for portions of the mechanical design of the microscope column and much of its precision machining, R. Lane who worked out many of the details of the vacuum system and the vibration isolation table, and J. Sokolowski whose patience and skill produced the final lens in which we were unable to detect a significant mechanical error.

Thanks are also due to the Cambridge Scientific Instruments Company for making the Stereoscan display electronics and scan coils available for use with the new microscope.

[16] T. E. Everhart, O. C. Wells, and C. W. Oatley, J. Electron. Control 7, 97 (1959).

[17] M. Hatzakis, J. Electrochem. Soc. (July 1969).