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PHOTOENGRAVING PROCEDURES FOR SILICON DEVICE FABRICATION

C. H. Klute
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M. L. Jones

23 February 1962

DIAMOND ORDNANCE FUZE LABORATORIES
ORDNANCE CORPS • DEPARTMENT OF THE ARMY
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FOR THE COMMANDER:
Approved by

Philip J. Franklin
Chief, Laboratory 900

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ABSTRACT

Photoengraving procedures are especially adaptable to the fabrication of solid-state electronic devices from silicon because they provide a convenient method for using the naturally occurring oxide layer to control the areas into which impurities can be diffused. A precision printer has been devised to register the so-called step-and-repeat transparencies, used in the photoengraving process, with appropriate areas of the silicon surface. By means of this device and selective etchants, it is possible to remove the oxide layer or to make shallow or comparatively deep etches into silicon over small and accurately specified areas. Examples of the results achieved thereby are discussed as they relate to silicon device fabrication.

1. INTRODUCTION

The fabrication of transistors and related solid-state electronic devices by photoengraving procedures is assuming increasing importance in both research and commercial production. Despite the success of this approach to silicon device fabrication, aside from references 1, 2, and 3 there has been comparatively little discussion of this process in the literature. In the photoengraving of a semiconductor, the lapped and polished surface of a single crystal wafer is coated with a layer of photosensitive polymer or "resist" (Eastman Kodak Co.). The coated wafer is then exposed to a light source, rich in short wavelengths, through a transparency bearing the pattern to be developed in the resist. Where the light strikes the resist, it is rendered insoluble and the pattern can be developed by dissolving the unexposed portions of the resist layer in an organic solvent or "developer" (Eastman Kodak Co.). Silicon can be made to grow a dense, amorphous layer of oxide that can be masked with the resist in this manner and selectively etched, where not protected by the resist, without attack on the elemental silicon underneath. Such a selectively etched oxide layer can be used as a barrier to prevent the diffusion of impurities into the silicon except in those areas from which the oxide has been removed (ref 4). The patterns involved are microphotographs with dimensions of a few thousands of an inch. Because multiple diffusions and related processing steps must frequently be performed within overlapping and accurately specified areas of the silicon surface, a mechanical and optical system providing high resolution and relative ease of operation is required for the registration of successive microphotographs. A detailed description is given of a precision registration printer used to register step-and-repeat transparencies, such as those described by Hellmers and Nall (ref 5), with the areas critical for diffused device fabrication. With the equipment described it is possible to remove relatively thick oxide layers from very small and accurately specified areas of the silicon surface. Shallow etches into silicon may be performed with equal accuracy. Deep etches into silicon may be achieved with somewhat less control of the geometry of the etched area. A brief discussion of these etching procedures with illustrations of the results achieved is also given.

2. PRECISION REGISTRATION PRINTER

2.1 Description

At the start of this investigation no device such as the precision registration printer to be described was available commercially; accordingly, a standard metallurgical microscope was converted into a precision registration printer by making three major modifications: (1) the vertical illuminator was modified to provide both incandescent light (filtered to remove the short wavelengths) for viewing and registering the patterns, and high intensity short wavelength light for making the exposure, (2) in addition to the rotating mechanical stage used to position the transparency, a mechanical substage was provided to rotate and translate the wafer beneath the transparency, and (3) a vertical motion (along the optic axis) was added to the substage so that the wafer can be brought into intimate contact with the transparency after registration. The completed precision registration printer and associated electrical equipment are shown in figure 1.

As the figure shows, a carbon arc light source is connected to the vertical illuminator through a conical tube, terminating in an electrically controlled shutter at the position normally occupied by the incandescent lamp, which is relocated so that it enters the vertical illuminator through the side at 90 deg to the optic axis. The incandescent bulb and its holder slide in a tube press-fitted into the illuminator body, so that the bulb may be withdrawn from the light path when the carbon arc source is used.

The platform of the microscope was modified to accomodate two rotating mechanical stages as mentioned previously. An exploded view of the modified arrangement is shown in figure 2. In addition to the upper stage for manipulating the transparency and the lower stage for providing the rotation and translation of the wafer holder, a rack and pinion are connected to, and the rack projects down from, the lower mechanical stage. This rack and pinion provide the coarse and fine vertical motion for the wafer holder. The structural connection between the two rotating stages was the milled aluminum plate, designated "rotating stage holder" in the figure. The lower stage was capable of a 70-deg rotation.

The wafer holder is shown diagrammatically (exploded view) in Figure 3. This subassembly was built around a monocular microscope body tube that engages the rack and pinion of figure 2. This was an element added to the metallurgical microscope during the modification and is not to be confused with the body of the latter. A brass barrel was machined to fit the upper end of the microscope body. Through the center of the body and the barrel, the micrometer shaft was inserted. The shaft supported the hemispherical anvil at its upper end in a seat provided with a ring of small ball bearings. This arrangement provided a limited tilting of the upper flat surface of the anvil. A micrometer dial

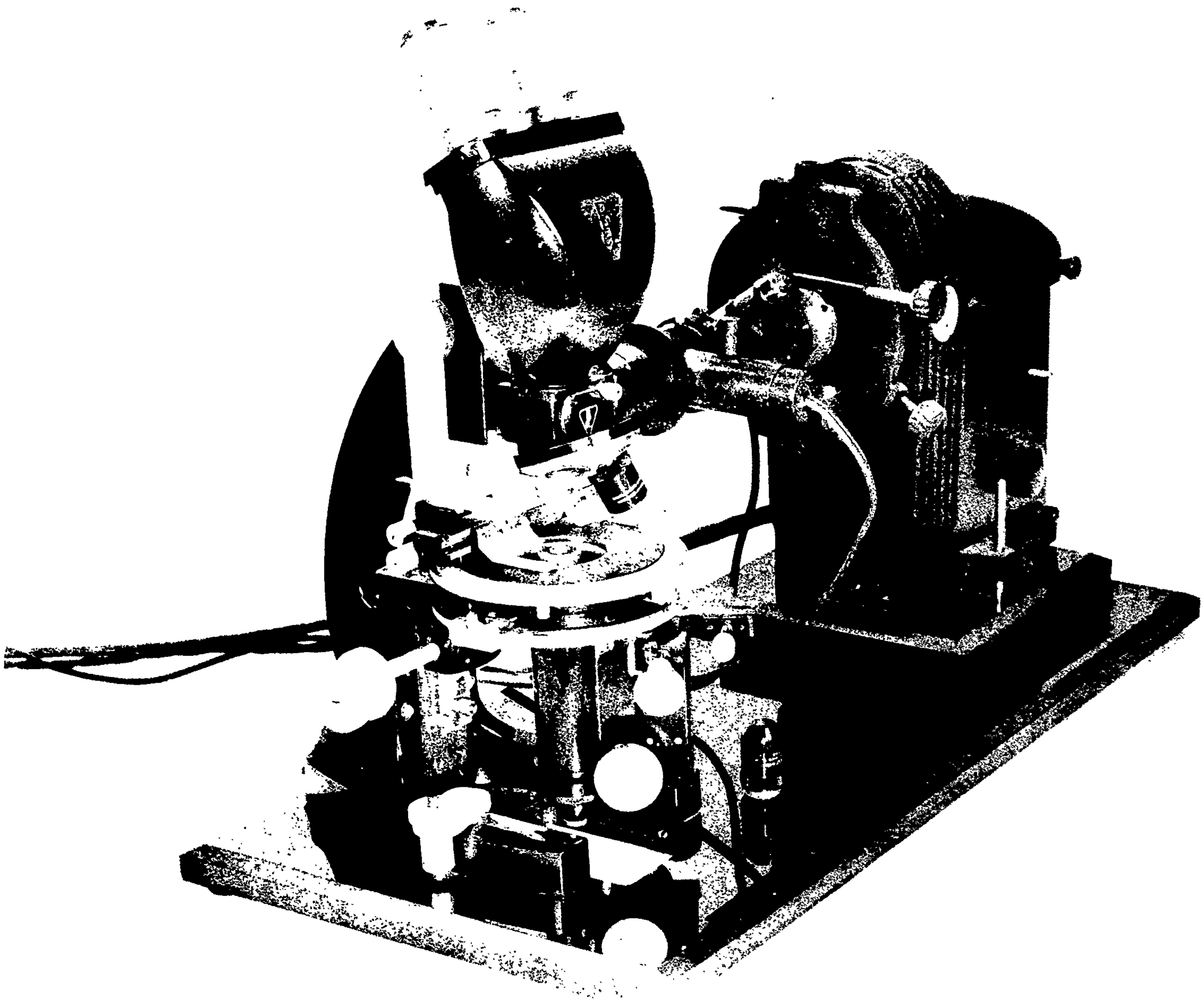

Figure 1. Precision registration printer for microphotographs. 1902-61

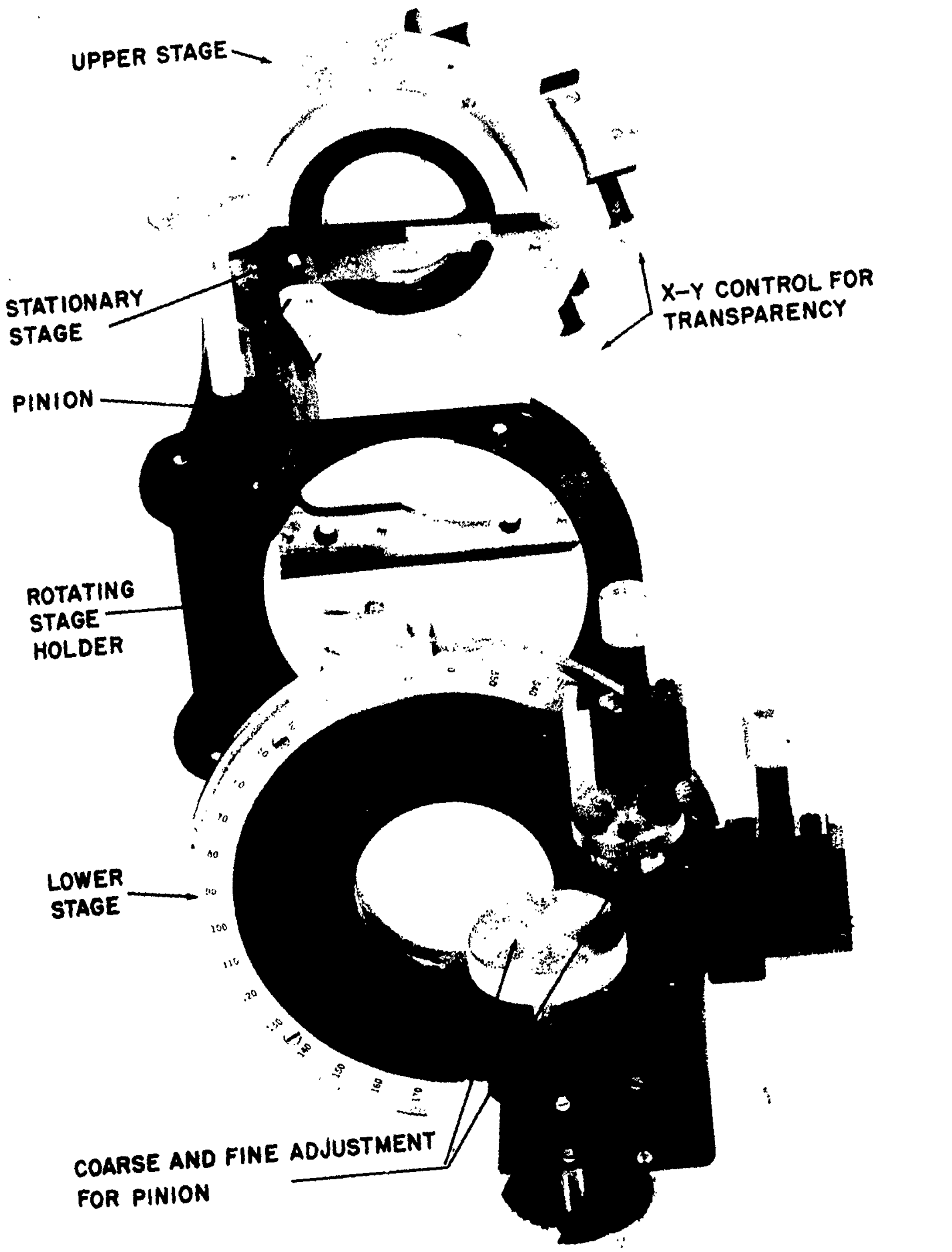

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Figure 2. Exploded view of microscope substage assembly.

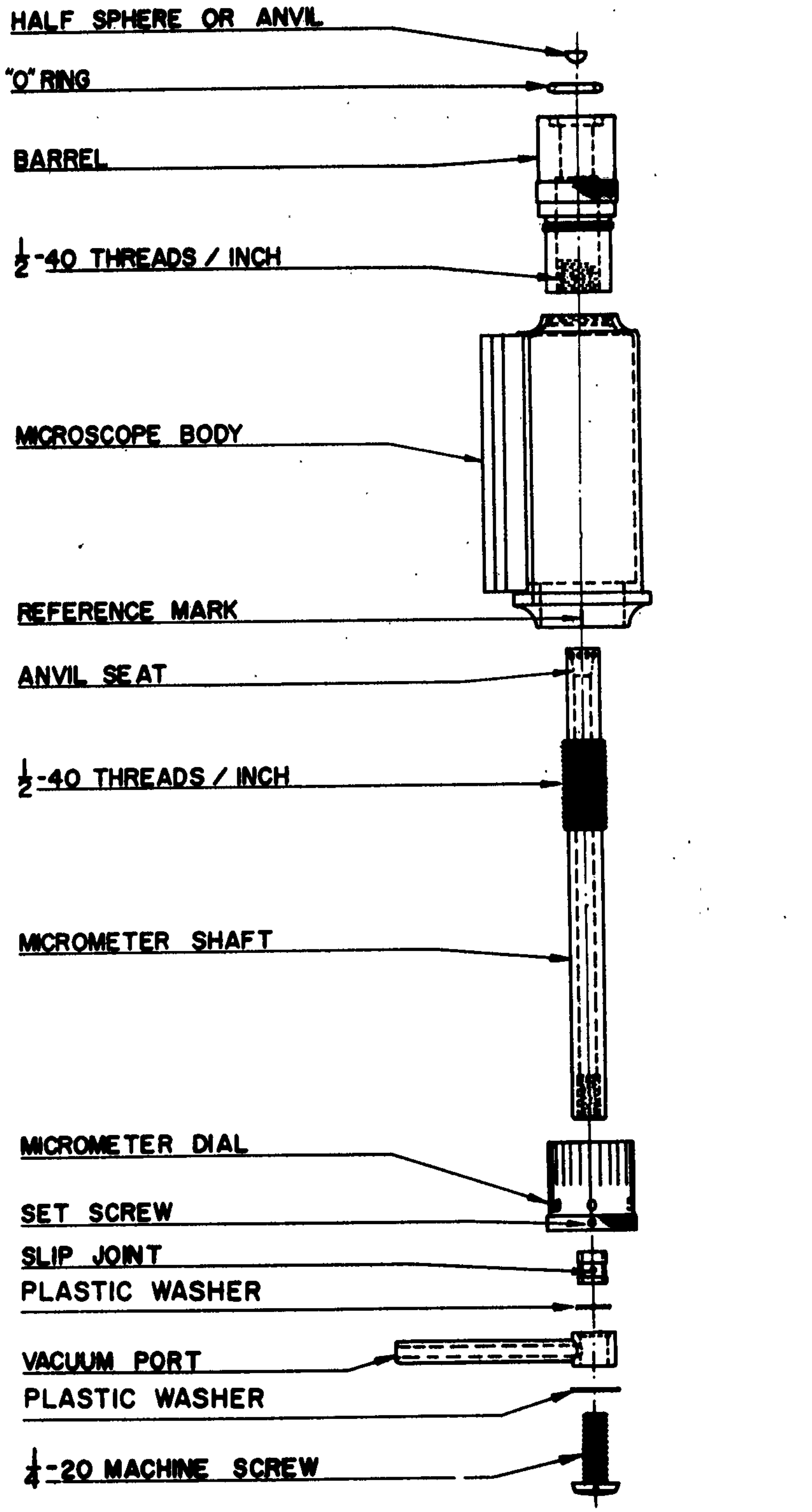

Figure 3. Diagram of wafer holder subassembly.

was secured to the lower end of the micrometer shaft. When these parts were assembled, the flat face of the anvil projected above the anvil seat slightly. It could be positioned relative to the upper recess in the barrel by a rotation of the micrometer dial. A rubber 0-ring, also placed within the recess, completed the upper part of the wafer holder subassembly. The lower part of the holder subassembly comprised the slip joint, two fluorocarbon washers, the vacuum port and a 1/4-20 machine screw. The latter had a hole down its center with an opening on the side which connected with the duct in the vacuum port. When these parts were assembled on the micrometer shaft and secured by the machine screw, a vacuum channel was provided through the vacuum port to the recess in the barrel. If a semiconductor wafer is placed upon the anvil, and the holder subassembly raised to make contact between the transparency and the 0-ring, application of a vacuum to the vacuum port compresses the 0-ring, permitting intimate contact between the pattern and the semiconductor wafer. It is apparent that the tilting motion of the anvil is necessary for making this intimate contact. The apparatus was designed to hold wafers up to 3/8 in. square used in laboratory experiments. Minor modification would allow it to accommodate the circular wafers approximately 3/4 in. in diameter obtained by cross-sectioning melt grown single crystals as supplied.

It was also necessary to raise the microscope platform above its usual position to accomodate the added substage components, and this in turn required raising the microscope optics. This was accomplished easily by securing vertical aluminum bars to the microscope frame and relocating the rack and pinion movements of the platform and the microscope optics to new positions on these bars. The assembly of the precision registration printer was completed by fastening the modified metallurgical microscope and arc lamp to a heavy base.

2.2 Operation

In the operation of the printer, the resist-coated wafer is placed on the anvil of the wafer holder and, by manipulation of the substage controls, is brought to the center of the field of a low-powered objective. The 1 in. by 3 in. glass transparency is placed, emulsion side down, into the slide holder of the upper mechanical stage. Viewing with the incandescent light, the operator brings the transparency into registration with the markings on the wafer which were produced by previous processing steps. Such markings generally arise from differences in thickness, and hence in the interference colors, between the oxide layers regrown over a previously etched area and those not previously etched. These differences are visible through the resist. Next the wafer holder is raised to make contact between the 0-ring and the transparency. Depending upon the thickness of the wafer, it may be necessary to raise or lower the anvil, with respect to the barrel, before applying the vacuum to the wafer holder to bring the wafer into intimate contact for the exposure.

To make the exposure, the incandescent light and filter are removed from the optical path and, with the shutter closed, the arc lamp is started. While the arc is stabilizing itself, the microscope objective is swung aside so that the intense light from the arc may strike the wafer directly after reflection from the mirror of the vertical illuminator. The shutter is then opened for a predetermined period by a timer-controlled solenoid. With resist materials presently in use, exposures vary from 45 to 75 sec.

3. ENGRAVING PROCEDURES

Of the three types of resist materials employed in semiconductor processing, photoresist, photosensitive lacquer, and metal etchant resist, the latter is the most resistant to the etchants used in silicon processing and was used in the examples discussed below. All of these materials are products of Eastman Kodak Co. The silicon wafers used in these experiments were lapped and polished along the (111) face of the crystal. The oxide layers, which were generally about 500 mμ thick, were produced by heating the wafers at 1100°C in a stream of wet nitrogen. The metal etchant resist as supplied was diluted with at least half its volume of the thinner (Eastman Kodak Co.) supplied with the resist, and this solution, made fresh daily, was spread over the wafer and the excess spun off. The wafer was next baked in the dark for 5 min to remove most of the thinner. The temperature of the bake was not critical; temperatures as low as 60°C and as high as 120°C were used successfully. The wafer was registered with the transparency and exposed as previously described. The development of the resist pattern required several minutes in the developer at room temperature. It is most important that the unexposed areas be absolutely free of resist because even a thin residue of resist will impede the action of the oxide etch to be used later. For this reason the wafer with the developed pattern was given a rinse in fresh developer after the main development, and before the developer could evaporate from the surface, it was flushed away with a small stream of water. A final bake at 120°C dried and hardened the resist. Where oxide only was to be etched later, a 10-min bake sufficed; if a deep etch into silicon was anticipated, the baking period was extended to 1 hr to impart added durability to the resist pattern. After etching, the resist layers were always easily removed with methylene chloride. The resist layer is delicate, particularly when swollen by the developer, and must be handled carefully.

Oxide masks through which impurities could be diffused into the desired areas of the silicon were prepared by etching the oxide where it was not protected by the resist. Etches for the oxide are based upon hydrofluoric acid. However, free HF in aqueous solution is extremely penetrating to the resist and will invariably undercut and lift it from the wafer before the desired areas are completely etched. By having ammonium fluoride in the solution and by having the concentration of the active components sufficiently low, this can be avoided. The oxide etchant used in this work was a mixture of 48 percent hydrofluoric acid, ammonium fluoride and deionized water in the approximate weight ratio 3:6:10. When used at room temperature, it penetrated 500 mμ of oxide in 4 min.

A typical example of a small pattern etched through an oxide layer is shown in figure 4. The etched rings are nominally 50μ wide, with inside diameters of 250μ. The center spot has a nominal diameter of 50μ. The dimensions of the etched pattern are not necessarily precisely equal to those of the transparency. The etched pattern may be slightly larger or slightly smaller than that of the transparency, depending upon the extent to which the etchant undercuts the resist or the extent to which light diffuses underneath the edges of the opaque regions of the transparency. The shifts of the edges of the etched pattern from those of the transparency can be kept to within ±2.5μ without difficulty and are consistent from pattern to pattern when made with the same transparency and etching procedure. The randon (i.e. statistical) uncertainty in the placement of a particular edge of repeated oxide patterns has been estimated to be within ±0.8μ. These dimensional variations are sufficiently small so that at present they do not represent a limitation upon the size of devices being manufactured. A more annoying source of dimensional disturbance arises from ragged edges that are somtimes observed. These are a reflection of the fragile nature of the resist while still wet with developer. If the resist is not carefully processed at this point, particles several microns in diameter may be dislodged from the pattern edges. Whenever this occurs, the defect is faithfully reproduced in the pattern subsequently etched in the oxide.

The patterns etched in oxide layers can be used to control the etching of silicon as well as the diffusion of impurities. Andrus and Bond (ref 1) have described an etchant for removing small amounts of silicon. With slow silicon etchants of this sort, etches several microns deep could be performed in which the random positional variation of the edges was somewhat greater than in the previous case, but still did not exceed ±2.0μ. The edges were characteristically smooth. Deeper etches into silicon could be achieved with more vigorous etchants. The etches shown in figures 5 and 6, for example, were accomplished by a 3-min immersion of the resist-covered wafer in 8:1 concentrated HNO_3 — concentrated HF after first removing the oxide in the oxide etch. This etchant penetrated the resist in about 1 min after which time the oxide mask concentrated the attack on the areas desired. At the end of 3 min, a thin film of oxide remained over the protected areas, whereas the silicon had been penetrated to a depth of 39μ elsewhere.

4. DISCUSSION

The procedures and equipment described above have been used in DOFL; similar procedures and related equipment find industrial application in the production of planar diodes and transistors and even more complicated structures. The engraving of patterns in the oxide layer is frequently used in silicon device fabrication. Figure 7, for example, shows a silicon wafer containing 25 planar transistors prepared by the procedures discussed, except that the more common photoresist was used instead of the metal etchant resist mentioned earlier. Oxide masking was used to localize the emitter and base diffusions to areas 150 and 450μ in diameter, respectively, as well as to locate the vacuum-deposited

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Figure 4. Oxide mask engraved on silicon wafer.

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Figure 5. Photomicrograph showing the end of a moat deeply etched in silicon.

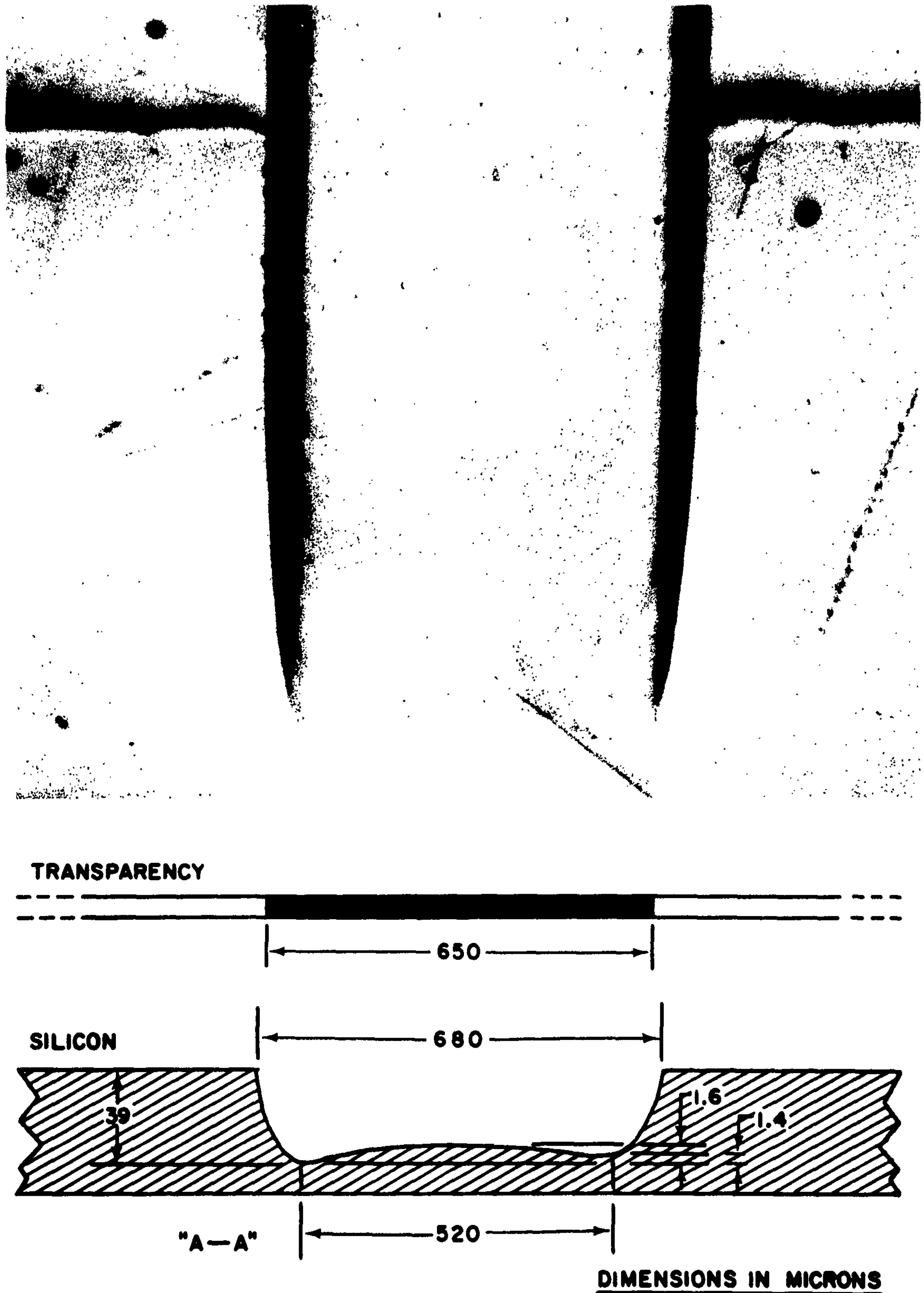

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Figure 6. Moat deeply etched in silicon lapped at 3 deg bevel to show cross section. The line drawing shows the relationship between the engraved pattern and the transparency from which it was derived.

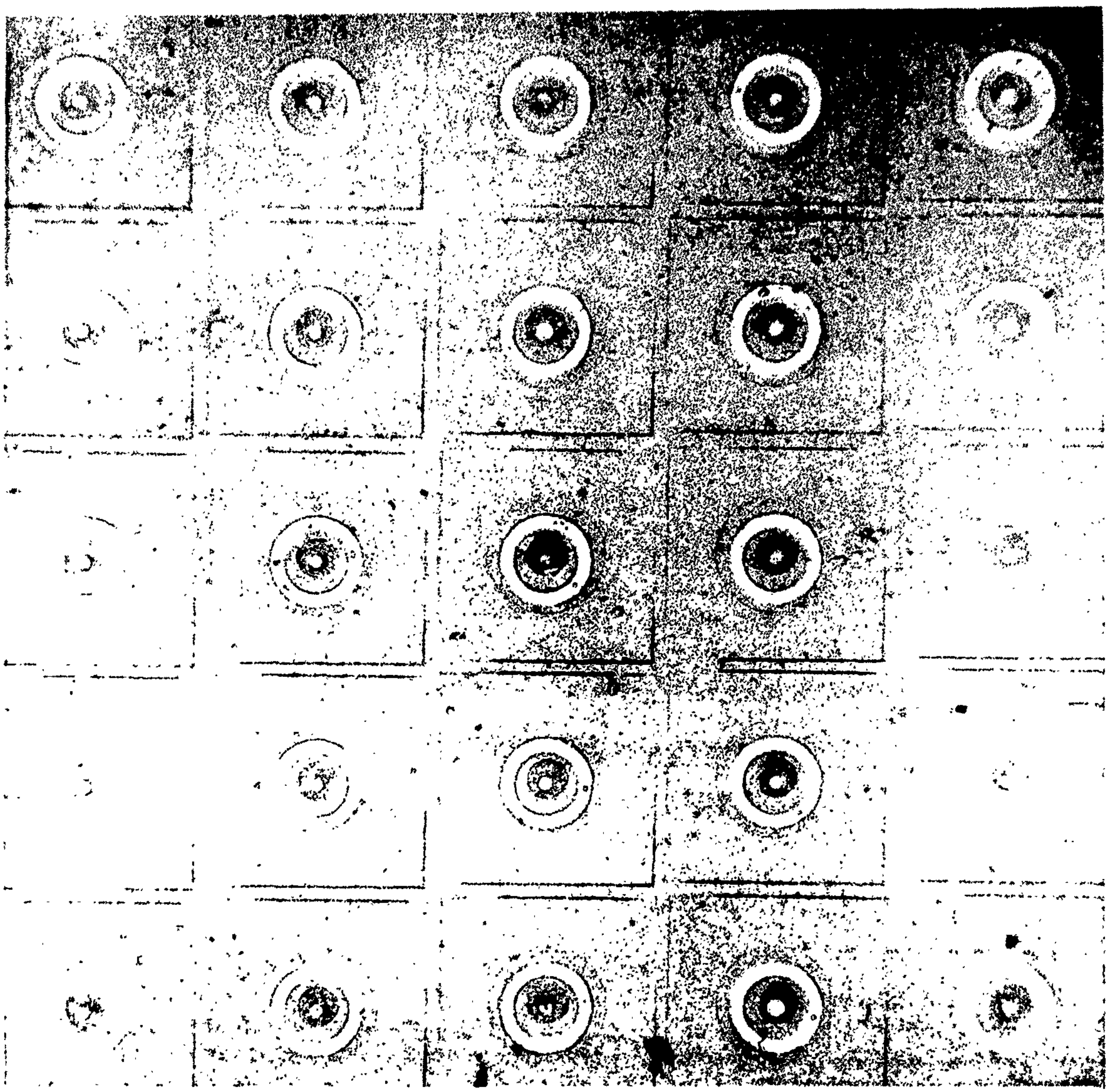

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Figure 7. Silicon planar transistors fabricated by means of photoengraving.

aluminum emitter and base contacts within these areas. Procedures for etching into the silicon itself are encountered less frequently but will certainly become more important as epitaxial structures become employed in so-called functional-block circuitry in which the desired electronic function is designed in a single piece of multiple-junction semiconductor material. The entire process depends upon good adhesion between the wafer and the resist, and this is best secured by coating the wafers promptly after oxidizing.

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ACKNOWLEDGMENT

J. R. Nall, formerly of DOFL, originated the design of the precision registration printer.

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TR-1019 23 February 1962 6 pp. text, 7 pp. illustrations
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2. Solid-state devices-Photoetching
3. Precision registration printer--Design
4. Precision registration printer--Application

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Photoengraving procedures are especially adaptable to the fabrication of solid-state electronic devices from silicon because they provide a convenient method for using the naturally occurring oxide layer to control the areas into which impurities can be diffused. A precision printer has been devised to register the so-called step-and-repeat transparencies, used in the photoengraving process, with appropriate areas of the silicon surface. By means of this device and selective etchants, it is possible to remove the oxide layer or to make shallow or comparatively deep etches into silicon over small and accurately specified areas. Examples of the results achieved thereby are discussed as they relate to silicon device fabrication.

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1. Photoengraving-Methods
2. Solid-state devices-Photoetching
3. Precision registration printer--Design
4. Precision registration printer--Application

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AD ____________ Accession No. ____________

Diamond Ordnance Fuze Laboratories, Washington 25, D. C.

PHOTOENGRAVING PROCEDURES FOR SILICON DEVICE FABRICATION
C. H. Klute, G. B. Wetzel, R. J. Anstead, and M. L. Jones

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