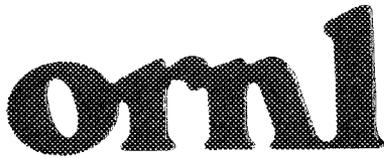




3 4456 0277124 9

ORNL/TM-10590



OAK RIDGE
NATIONAL
LABORATORY



New Applications of Silicon Micromachining

R. J. Lauf
R. F. Wood
P. H. Fleming
M. L. Bauer

OAK RIDGE NATIONAL LABORATORY
CENTRAL RESEARCH LIBRARY
CIRCULATION SECTION
K100A ROOM 113

LIBRARY LOAN COPY
DO NOT TRANSFER TO ANOTHER PERSON
If you wish someone else to see this
report, send in name with report and
the library will arrange a loan

UNCLASIFIED

OPERATED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

Printed in the United States of America. Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
NTIS price codes—Printed Copy: A03 Microfiche A01

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

ORNL/TM-10590
Distribution
Category UC-37

Metals and Ceramics Division

NEW APPLICATIONS OF SILICON MICROMACHINING

R. J. Lauf
R. F. Wood
P. H. Fleming
M. L. Bauer

Date Published: June 1988

NOTICE: This document contains information of preliminary nature. It is subject to revision or correction and therefore does not represent a final report.

Prepared by the
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-84OR21400



3 4456 0277124 9

TABLE OF CONTENTS

	Page
LIST OF FIGURES	v
ABSTRACT	1
INTRODUCTION	1
RUGGED ALPHA DETECTOR	4
FLUID-SAMPLE CELL FOR SAXS	5
EXPERIMENTAL	7
WAFER PREPARATION	7
ETCHING PROCEDURE	8
RESULTS	12
RUGGED ALPHA DETECTOR	12
FLUID-SAMPLE CELL FOR SAXS	16
SUGGESTIONS FOR FUTURE WORK	18
CONCLUSIONS	18
ACKNOWLEDGMENTS	19
REFERENCES	19

LIST OF FIGURES

	<u>Page</u>
Fig. 1. Solid state absolute pressure transducer using a micromachined silicon chip	2
Fig. 2. Cross-section of a boron-doped silicon wafer, masked with SiO ₂ , before and after etching with a mixture of ethylenediamine, pyrocatechol, and water	3
Fig. 3. Proposed design for a rugged α -detector using a chemically micromachined silicon wafer. Note that thicknesses are not to scale	5
Fig. 4. Cross section of a silicon wafer coated with a layer of CVD ceramic before masking and etching with a mixture of ethylenediamine, pyrocatechol, and water. No boron-diffused etch-stop layer is needed, because etching stops at the ceramic layer, which then forms the window	6
Fig. 5. Fluid sample holder for small-angle X-ray scattering. Note that a hole could also be drilled from the bottom to facilitate filling, draining, and cleaning	7
Fig. 6. Photomask patterns used in silicon etching with EDP mixture. (a) Pattern for initial tests and for SAXS sample cell, (b) Pattern for full-sized prototype α -detector	10
Fig. 7. Etched window in sample 1--3. Note undercutting of the mask as the etch follows preferred crystallographic directions	13
Fig. 8. Edge of fractured window showing that boron diffusion profile follows irregularities in the bottom surface of the wafer	13
Fig. 9. Wafer etched 7.5 h, showing silicon precipitated on window. (a) View of one window. (b) Detail showing that precipitates are pyramids bounded by $\langle 111 \rangle$ surfaces	14
Fig. 10. Thin, perforated window in sample 3-2, attributed to inadequate boron doping	15
Fig. 11. Sample 7-1, showing square windows with smooth bottoms and well-defined corners. This wafer was implanted on the polished side, so the (as-received) etched side of the wafer is up. Compare the surface texture on the supporting rib (inset) to the textures in Fig. 8	15
Fig. 12. Scattering cross sections of single-crystal silicon and Mylar tape as a function of scattering angle	17

NEW APPLICATIONS OF SILICON MICROMACHINING*

R. J. Lauf, R. F. Wood, P. H. Fleming, and M. L. Bauer

ABSTRACT

The use of photolithography and anisotropic etching of silicon wafers to make strong, thin membranes has created a large family of miniature sensing devices such as pressure transducers and accelerometers. This report describes several entirely new devices in which silicon membranes are used for their strength and for their transparency to certain kinds of radiation. Two applications are described: a rugged alpha detector and a fluid sample cell for small-angle X-ray scattering.

INTRODUCTION

Single-crystal silicon is used in a wide variety of miniature sensing devices that have become commercialized over the last fifteen years. These devices rely not only on the electronic properties of single-crystal silicon but also on the outstanding mechanical properties of silicon.¹ One of the earliest and most successful mechanical applications of silicon is the solid state pressure transducer, illustrated schematically in Fig. 1. In this device, the silicon wafer is deeply etched to form a thin, square membrane, which deflects under a pressure differential. The deflection is measured by a piezoresistive strain gage with an integrated signal conditioning circuit, all fabricated directly on the silicon.² Devices are commercially available to measure vacuum, differential, absolute, and gage pressures as high as 69 MPa (10,000 psi).

*Research sponsored by the Exploratory Studies Program of the Oak Ridge National Laboratory under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc., and by the U.S. Navy RADIAC Project.

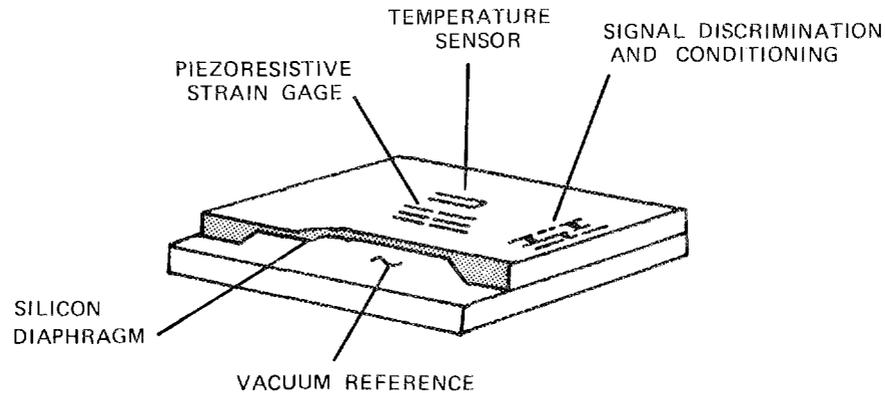


Fig. 1. Solid state absolute pressure transducer using a micromachined silicon chip.

Similar technology has been used to create a miniature accelerometer,³ a micromechanical switch,⁴ and a miniature gas chromatograph.⁵ In each case, a critical aspect of device fabrication is the controlled etching of a silicon wafer to produce membranes, grooves, channels, cantilever beams, and similar components. Chemical etchants for silicon can be isotropic or anisotropic, dopant dependent or not, and have varying degrees of selectivity to silicon, which in turn determines the appropriate masking material.¹

One commonly used etchant is a mixture of ethylenediamine, pyrocatechol, and water (EDP). The advantages of EDP are: (1) it is anisotropic, making it possible to fabricate unique geometries; (2) it is highly selective to silicon and so can be masked by a variety of convenient materials such as SiO_2 , Si_3N_4 , chromium, and gold; and (3) it is dopant dependent, exhibiting virtually zero etch rate on silicon that is highly doped with boron.^{6,7}

To make a thin silicon membrane, boron is diffused into the back side of a $\langle 100 \rangle$ silicon wafer to a depth corresponding to the desired membrane thickness. A mask is then developed on the front side of the wafer by conventional photolithography. Etching of the unmasked area proceeds downward to the point where the boron concentration reaches about $7 \times 10^{19}/\text{cm}^3$. At that point, etching virtually stops, leaving a smooth, flat-bottomed pit bounded on the sides by $\langle 110 \rangle$ and $\langle 111 \rangle$ planes (Fig. 2).

ORNL-DWG 87-1991

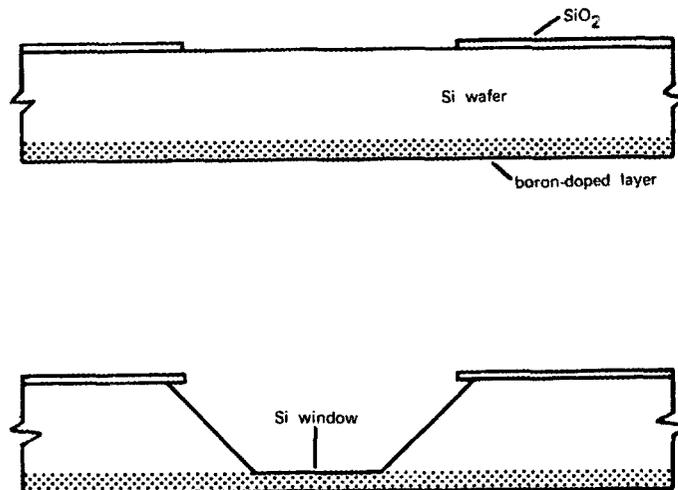


Fig. 2. Cross section of a boron-doped silicon wafer, masked with SiO₂, before and after etching with a mixture of ethylenediamine, pyrocatechol, and water.

The technology for silicon micromachining is sufficiently mature that membranes as thin as $\sim 5 \mu\text{m}$ can be formed by this process. Because a silicon membrane of this thickness should be reasonably transparent to many kinds of radiation, two applications were examined, namely, a rugged α detector and a fluid sample cell for small-angle X-ray scattering (SAXS).

This report describes the initial experiments intended to demonstrate the concept of micromachining to achieve radiation transparency while maintaining adequate mechanical strength.

RUGGED ALPHA DETECTOR

Conventional α -radiation detectors typically employ a scintillator/photomultiplier system in which the energetic α -particles cause the scintillator to emit light, which is detected and amplified by the photomultiplier (P/M) tube. Because the P/M tube is sensitive to visible light, the device must be shielded by a window that is opaque to ambient light yet "transparent" to α -particles. The commonly used window material is a double layer of aluminized Mylar. Unfortunately, the Mylar window is quite fragile and is easily punctured by particles such as dirt and sand; light can enter through the puncture and overwhelm the detector. Consequently, the use of these detectors in the field to survey α -contamination is very costly because of their short useful life.

Attenuation of α -particles by candidate window materials can be estimated by the total mass per unit area. The mass of nominally 0.00025-in. aluminized Mylar is ~ 1 mg/cm², whereas the range of 4-MeV α -particles is ~ 3 to 4 mg/cm². (Note that the range is not linear with energy. In copper, for example, the range is ~ 1 mg/cm² per MeV at 1 MeV but increases to ~ 2 mg/cm² per MeV at 5 MeV.) Using the density of silicon, one can calculate that 1 mg/cm² represents a silicon window 4.3 μ m thick. The penetration depth of α -particles in silicon is shown in the following table for several energies:

<u>α-Particle energy (MeV)</u>	<u>Penetration depth (μm)</u>
1	4
3	15
5	25

Therefore, a silicon membrane 4 μ m thick will attenuate α -particles with initial energies less than ~ 1 MeV but will admit most of those with higher energies. To achieve the desired mechanical strength and abrasion resistance, as well as to simplify manufacturing, the following design is proposed (Fig. 3). A silicon wafer would be etched to provide the maximum thin area consistent with mechanical strength. A very thin layer of aluminum would be deposited on the etched side to ensure optical opacity, followed by a layer of scintillator material (silver-activated zinc

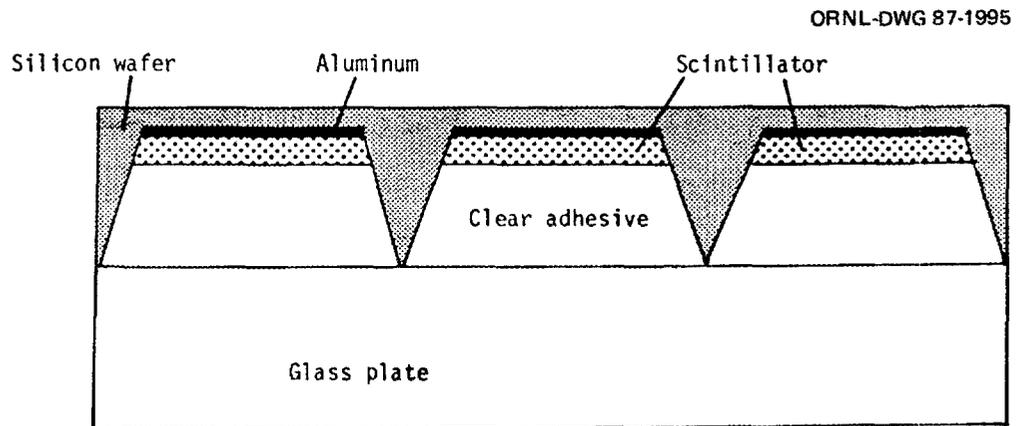


Fig. 3. Proposed design for a rugged α -detector using a chemically micromachined silicon wafer. Note that thicknesses are not to scale.

sulfide) also deposited by evaporation. The wafer would then be attached to a thick glass plate with transparent adhesive that would fill in and support the thin areas to create a solid, robust assembly. The smooth side of the wafer faces to the outside to facilitate cleaning in the field.

For greater abrasion resistance, a possible improvement would be to deposit a thin layer of hard ceramic such as Si_3N_4 , SiC , or AlN on the silicon wafer and then completely etch the silicon away in the window areas, as shown schematically in Fig. 4. This approach has the added advantage that chemical vapor deposition is faster and easier to control than boron diffusion.

FLUID-SAMPLE CELL FOR SAXS

The ORNL 10-m SAXS camera⁸ is a powerful tool for examining materials such as solids, powders, and polymer films by X-ray scattering through angles from ~ 1 to ~ 100 mrad. It is difficult to examine liquid samples because the X-ray beam line is evacuated and the liquid must therefore be contained in a sealed capsule with thin windows. The window material, in addition to having low absorption, ideally should not scatter strongly over the angles of interest. Beryllium sheet, for example, has unacceptably high scattering, presumably from dispersed BeO particles. Mylar has

ORNL-DWG 87-1990

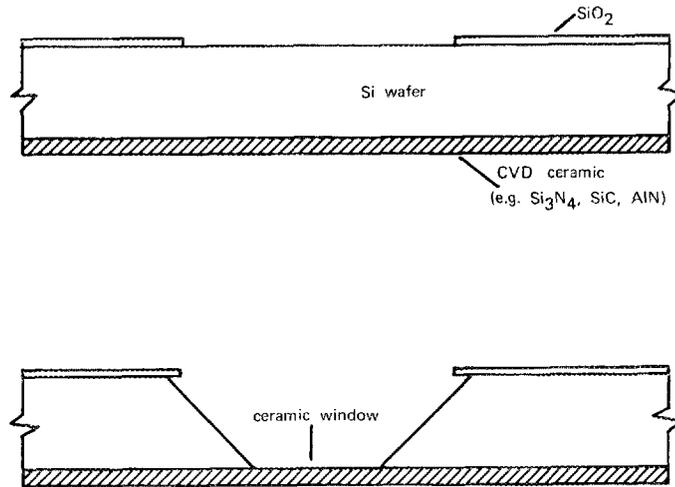


Fig. 4. Cross section of a silicon wafer coated with a layer of chemically vapor deposited ceramic before masking and etching with a mixture of ethylenediamine, pyrocatechol, and water. No boron-diffused etch-stop layer is needed, because etching stops at the ceramic layer, which then forms the window.

relatively good transmission and little scattering, but it is so weak that the windows tend to balloon outward, changing the effective sample thickness. In addition, many organic liquids can react with Mylar, it is difficult to seal reliably, and it is permeable to many solvents.

A fluid sample cell designed to use micromachined silicon windows is shown schematically in Fig. 5. In this design, a wafer with many windows would be diced so that each die has a single window surrounded by a thick rim for support, much like the solid state pressure transducer. The windows would be soldered into a machined holder of Kovar or other low-expansion alloy (to match the expansion coefficient of silicon) using gold-silicon or gold-germanium eutectic solder.

The fill hole shown at the top of the holder (Fig. 5) could be supplemented by a drain hole at the bottom to facilitate filling, draining, and cleaning. Furthermore, it is conceivable that tubes could be fitted to the openings to actively pump the fluid through the chamber so that flow processes could actually be studied dynamically by SAXS.

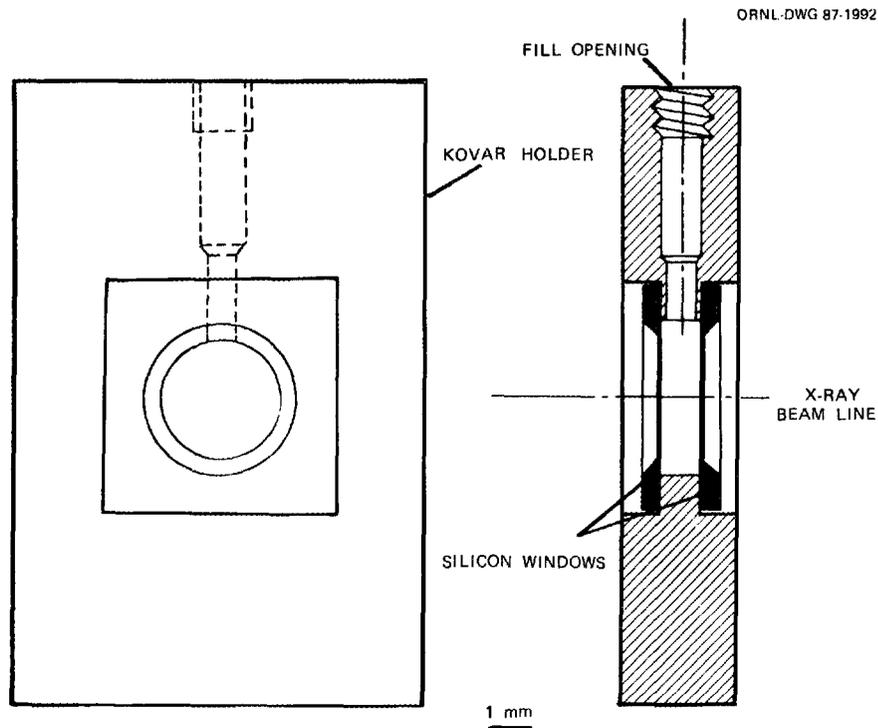


Fig. 5. Fluid sample holder for small-angle X-ray scattering. Note that a hole could also be drilled from the bottom to facilitate filling, draining, and cleaning.

EXPERIMENTAL

WAFER PREPARATION

The $\langle 100 \rangle$ silicon wafers used in this study were 7.6 cm (3 in.) in diameter and $\sim 300 \mu\text{m}$ thick, obtained commercially from Wacker Siltronic Corp. The wafers were float-zone refined, but Czochralski material should work equally well, and indeed for this application the quality of the silicon does not seem to be a very important consideration. Base resistivities of $\sim 0.3 \Omega\text{-cm}$ in both n-type (P-doped) and p-type (B-doped) silicon as supplied by the manufacturer correspond to doping levels of less than $10^{17}/\text{cm}^3$ and are not expected to alter the etch rates reported in the literature.^{6,7} The wafers had been polished on one side and bright etched on the other.

The wafers were ion implanted with boron to a dose calculated to yield $\sim 5 \times 10^{19}$ to 1×10^{20} B/cm³ after diffusion to a depth of ~ 5 μ m. Initially, both commercially implanted wafers and wafers implanted in an experimental dc glow-discharge apparatus were investigated. The glow-discharge implantation proved so satisfactory that it was used in all subsequent work involving boron doping.

After implantation, diffusion of the implanted boron was carried out at 1200°C for about 90 min in a commercial diffusion furnace. Not only does the diffusion drive the boron in to the desired depth, but it also removes the lattice damage associated with the implantation process. During the initial stages of the work, when the processing conditions were being optimized, two wafers were subjected to each set of implantation and diffusion conditions and then cut into quarters to provide eight identical samples for the etching experiments. Also during this period, implantation, diffusion, and etching to form the thin windows on the bright-etched back sides of the wafers were tried. As will be shown, the texturized surface produced by the bright-etching process created an uneven surface on the 5- μ m-thick windows. Consequently, all later work was restricted to forming the windows on the polished side of the wafers. Wafer preparation is summarized in Table 1.

ETCHING PROCEDURE

Photomasks were made in two different patterns (Fig. 6). Both were made to cover a nominal 3-in. wafer, and for each pattern a positive and a negative photomask were made on 4 \times 5 in. glass plates. (By making both positive and negative photomasks, we retained the flexibility to use either positive- or negative-acting resist, depending on the needs of the etching process.)

The first pattern is an array of 3-mm round holes spaced 9.5 mm center to center. This pattern served two purposes. First, thin windows would be available for testing their transparency to α -particles, with a substantial amount of thick material remaining to facilitate handling. Second, the holes were spaced so that, if successfully etched, the wafer could then be diced to make the SAXS sample holder shown in Fig. 5.

The second pattern consists of square holes 3 mm wide spaced 5 mm center to center. This pattern was designed with two factors in mind.

Table 1. Silicon wafer preparation

Wafer	Etch stop layer	Side
1, 2	Boron, $5 \times 10^{19}/\text{cm}^3$; implanted at 3 KeV; annealed 1.5 h at 1200°C	Etched
3, 4 ^a	Boron, $5 \times 10^{19}/\text{cm}^3$; implanted at 3 KeV	Polished
5 ^b	Aluminum nitride, deposited by CVD at 300°C	Polished
6 ^b	Silicon nitride, deposited by CVD	Polished
7, 9	Boron, $5 \times 10^{19}/\text{cm}^3$; implanted at 3 KeV, annealed 1.5 h at 1200°C	Polished

^aWafers 3 and 4 were the first to be implanted on the polished side. They were implanted at -3 KeV, laser annealed, and then implanted again in an attempt to ensure a high boron dose. The results were unsatisfactory, and the much simpler treatment used with wafers 7 and 9 subsequently became standard.

^bEtch tests on ceramic-coated wafers have not been completed at this time.

First, as will be shown, the EDP mixture is crystallographically anisotropic, so round holes on a <100> wafer will undercut the mask to become square holes anyway (actually irregular octagons) bounded by <110> and <111> planes. Thus, by making the holes in the mask square and orienting them parallel to the <110> directions (indicated by a flat on the edge of the wafer) we could make a very crisply defined square window. Second, this pattern has much more thin area and is better suited to building a full-sized prototype α -detector.

To prepare the wafers for etching after the implantation and diffusion steps, a 4500-Å-thick SiO₂ layer was grown on the wafers by wet oxidation at 1000°C for 2 h. This layer prevents etching by the EDP mixture except in the areas defined by photolithography using the photomasks described above.

Guided by information in the literature,^{6,7} we designed, constructed, and tested an etching apparatus. An extended series of etching runs (Table 2) was made to study the etching process and to establish and gain control of the most important variables of the entire fabrication process. Fortunately, the very first attempts established that our basic approach was sound, and subsequent runs were made primarily to improve the process to provide the best samples possible for testing.

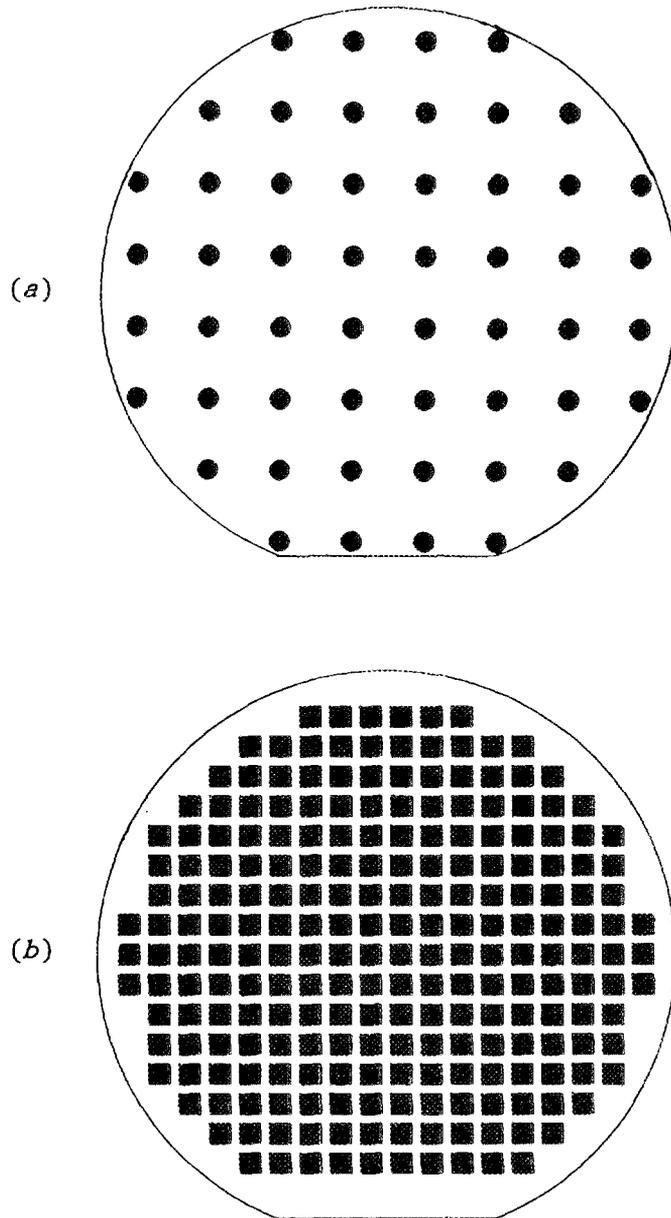


Fig. 6. Photomask patterns used in silicon etching with ethylenediamine, pyrocatechol, and water mixture. (a) Pattern A for initial tests and for SAXS sample cell, (b) Pattern B for full-sized prototype α -detector.

Table 2. Results of etching silicon wafers

Wafer sample ^a	Pattern	Total etch time ^b (min)	Window characteristics
1-1	A	315	Fairly smooth, ~10 μm thick
1-2	A	450	Thick in most places, rough
1-3	A	300	Very good, ~5 μm thick
2-1	A	455	Fairly uniform, ~3.5 μm thick
3-2	A	450	Very thin, full of holes
3-3	A	450	Very thin, full of holes
3-4	A	435	Very thin, full of holes, some small pyramids present
7-1	B	320	Very smooth, uniform, no pinholes, ~6 μm thick
7-2	B	305	Smooth, uniform, thin
7-3	B	335	Smooth, uniform, a few pinholes present
9-1	B	305	Fairly thin, uniform, a few pyramids present
9-2	B	335	Very thin and uniform

^aFirst digit identifies the wafer number.

^bEthylenediamine: pyrocatechol: water (225 ml:45 g:120 ml) refluxed at 116°C; masked with SiO_2 .

The etching procedure that evolved can be described as follows. The etchant was a mixture of 225 ml of ethylenediamine, 45 g of pyrocatechol, and 120 ml of distilled water. The ingredients were mixed and heated in a quartz reflux system resting on a hot plate, with provisions for continuous stirring and the slow bubbling of nitrogen to prevent oxidation. The mixture was held at the boiling point of 116°C during the entire etching run. Immediately before inserting the SiO_2 -masked wafer into the etching chamber, it was dipped in buffered hydrofluoric acid, rinsed in distilled water, and dipped in ethylenediamine. Under the conditions of our experiments, the total etching time as ~5 h, but the etching apparatus was designed so that the sample could be easily removed for visual inspection (5- μm silicon membrane appears slightly reddish under transmitted white light) and reinserted for longer etching if needed.

The results of all the etching experiments are given in Table 2. In general, the EDP etch performed as reported in the literature.^{6,7}

A typical window made from Pattern A (round holes), Fig. 7, is an octagon with four long sides bounded by $\langle 111 \rangle$ and four shorter sides bounded by $\langle 110 \rangle$ surfaces. One window was punched out to better visualize its thickness and surface texture (Fig. 8). The thickness was well controlled by the boron doping, although the surface texture of the bottom side of the wafer influenced the shape of the boron diffusion profile. Consequently, the inner surface of the window had a texture similar to the back side of the wafer.

When the wafer was etched for too long a time (7.5 vs 5 h), the windows became thick and rough (Fig. 9). We attribute this to precipitation of some of the silicon back onto the etched surface. The silicon deposits consist of intergrown pyramids bounded by $\langle 111 \rangle$ surface.

The first two wafers doped with boron on the polished side (wafers 3 and 4) did not etch properly. The windows became extremely thin and full of holes (Fig. 10). After several attempts with similar results, it was concluded that the doped layer had too little active boron, perhaps because of solid-phase precipitation, and/or because the boron had possibly diffused too deeply so that a well-defined etch-stop layer was not created. Later wafers (e.g., 7 and 9) implanted only once from the polished side and thermally annealed without the laser surface treatment performed quite well. Excellent results were obtained using the square-hole mask (Pattern B) on wafers implanted from the polished surface (Fig. 11).

RESULTS

Selected etched samples were tested for their transparency to α -particles and to X-rays as well as their X-ray scattering over small angles typical of the range used in the ORNL 10-m SAXS camera.

RUGGED ALPHA DETECTOR

The scope of this exploratory study did not include the actual building of a-detector as envisioned in Fig. 3. However, a preliminary test of the transparency to alpha-particles was conducted using sample 1-3. For this, a ^{239}Pu source was used, which has a half-life of 24,400 years and emits alphas with an average energy of ~ 5.14 MeV. The source was ~ 3 mm in diameter, set into the surface of a metal disk.

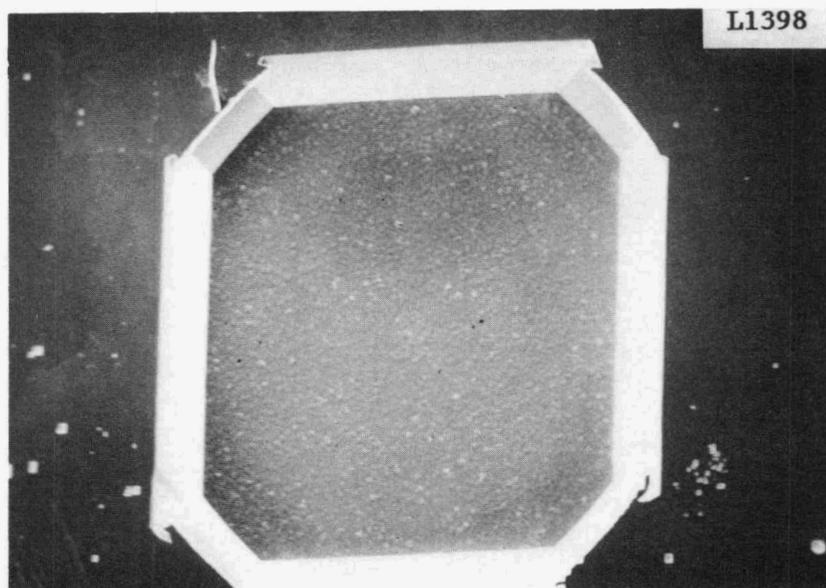


Fig. 7. Etched window in sample 1-3. Note undercutting of the mask as the etch follows preferred crystallographic directions.

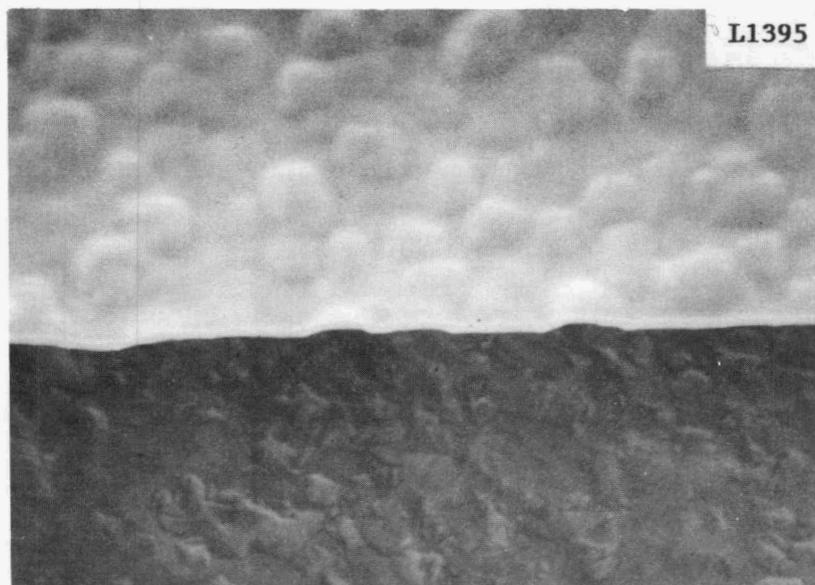


Fig. 8. Edge of fractured window showing that boron diffusion profile follows irregularities in the bottom surface of the wafer.

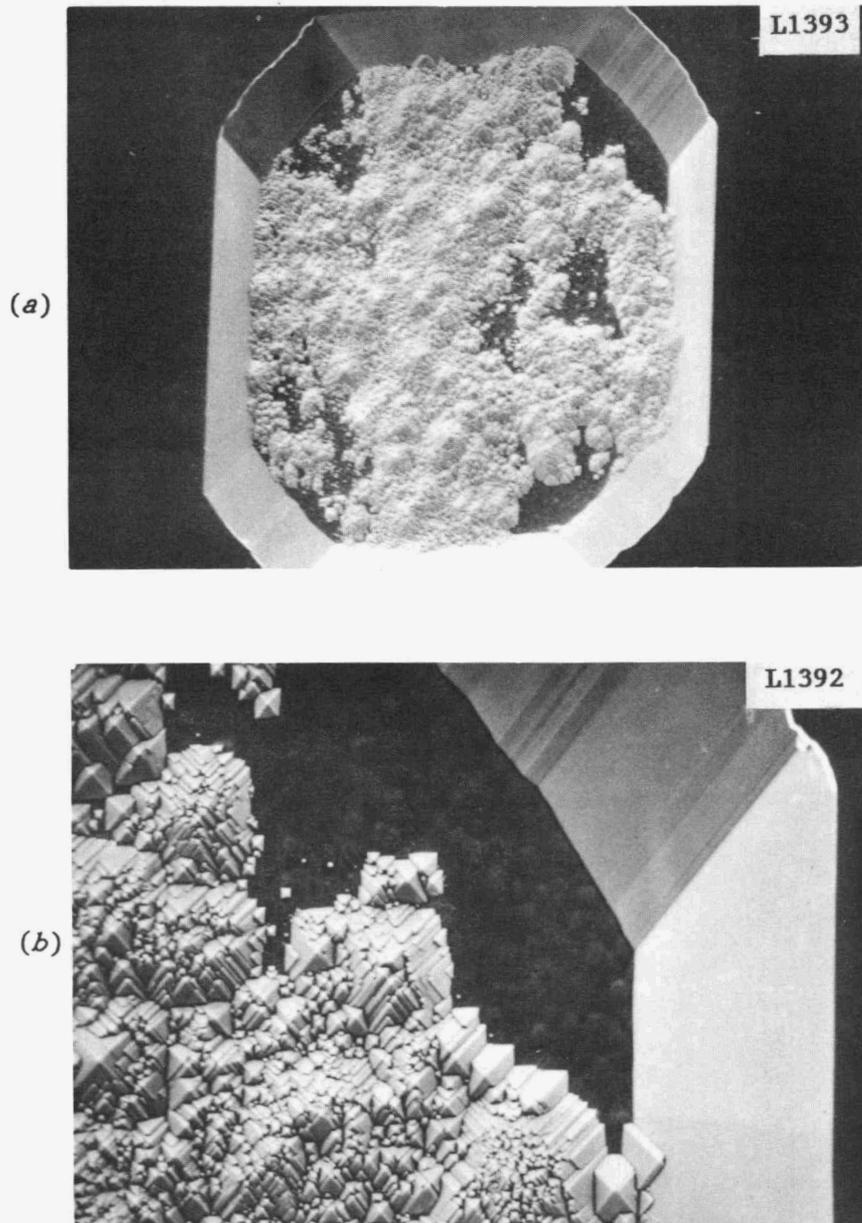


Fig. 9. Wafer etched 7.5 h, showing silicon precipitated on window. (a) View of one window. (b) Detail showing that precipitates are pyramids bounded by $\langle 111 \rangle$ surfaces.

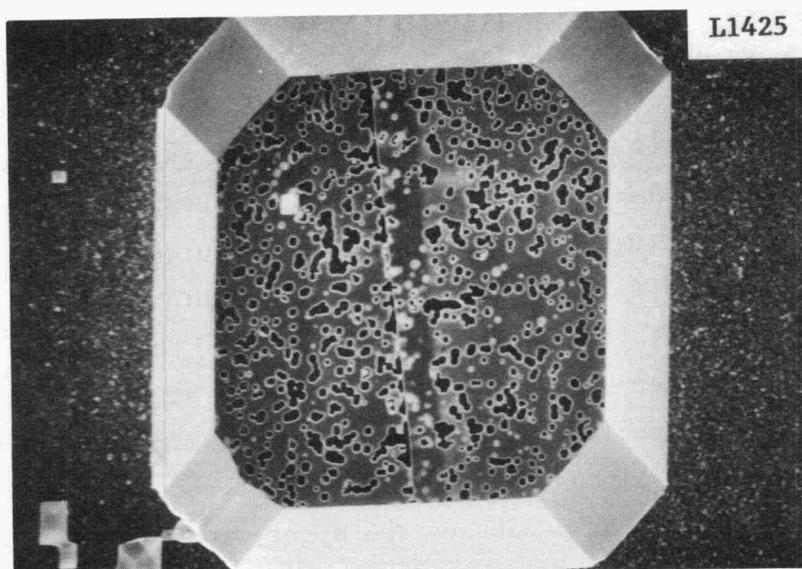


Fig. 10. Thin, perforated window in sample 3-2, attributed to inadequate boron doping.

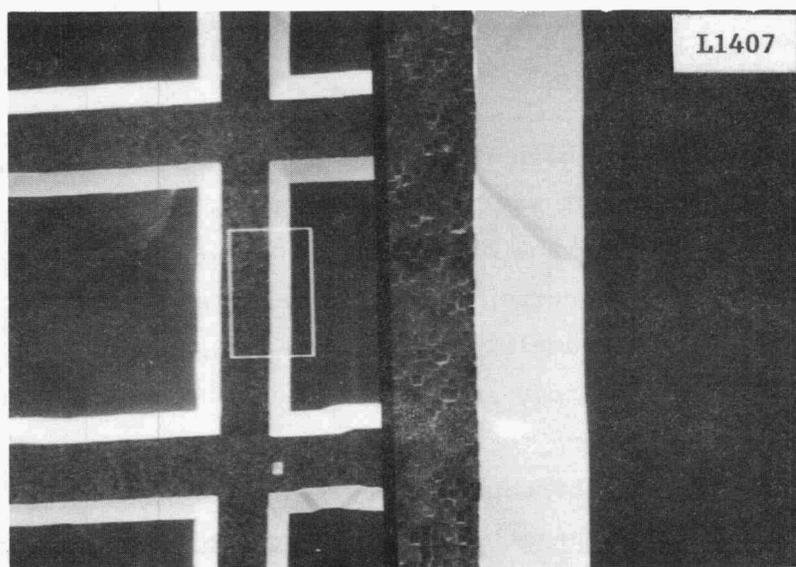


Fig. 11. Sample 7-1, showing square windows with smooth bottoms and well-defined corners. This wafer was implanted on the polished side, so the (as-received) etched side of the wafer is up. Compare the surface texture on the supporting rib (inset) to the textures in Fig. 8.

Measured directly with a hand-held alpha detector, the source gave $\sim 3690 \pm 40$ counts/min.* When the thick part of the wafer was placed over the source, no alphas were detected, as one would expect. Then, because the windows were smaller than the source, one window was punched out and a reading was taken through that opening. The measured activity, 2495 ± 46 counts/min, accounts for screening and collimation effects and thus serves as the appropriate baseline for determining the absorption by one of the thin windows. When one of the intact windows was placed over the source, the measured activity was 1174 ± 2 counts/min. demonstrating that about one-half of the alphas were able to pass through the silicon while retaining enough energy to penetrate the Mylar windows of the conventional α -detector used to make the measurement.

This result is especially encouraging given that there is some attenuation of the α -particles by the Mylar windows. These losses would be eliminated in practice by depositing the scintillator directly on the silicon, as shown in Fig. 3, and eliminating the Mylar completely.

FLUID-SAMPLE CELL FOR SAXS

For this application, both the absorption and scattering of X-rays should be low. A convenient way to measure sample absorption in the ORNL SAXS camera is to determine the attenuation of a strong scatterer. A strong scatterer made from carbon filled polyethylene is put into the beam on the source side of the sample position. The total scattering into the detector is measured. The test sample is then put in place so as to attenuate this scattering and the total scattered intensity is measured again. After appropriate corrections the ratio of intensities indicated about 89% transmission by the silicon window, which compares well with the 94% transmission of Mylar tape measured in the same way.

The scattering of X-rays by the silicon window was then measured using a 5-m camera length to cover the range of angles (Q -values) from 0.01 to 0.1 \AA^{-1} . Because the scattering is isotropic, the two-dimensional data can be plotted as a circular average and converted to actual scattering cross section vs scattering angle. As shown in Fig. 12, for most angles of interest scattering by the silicon window is significantly lower than that

*Average of three 1-min readings for each case.

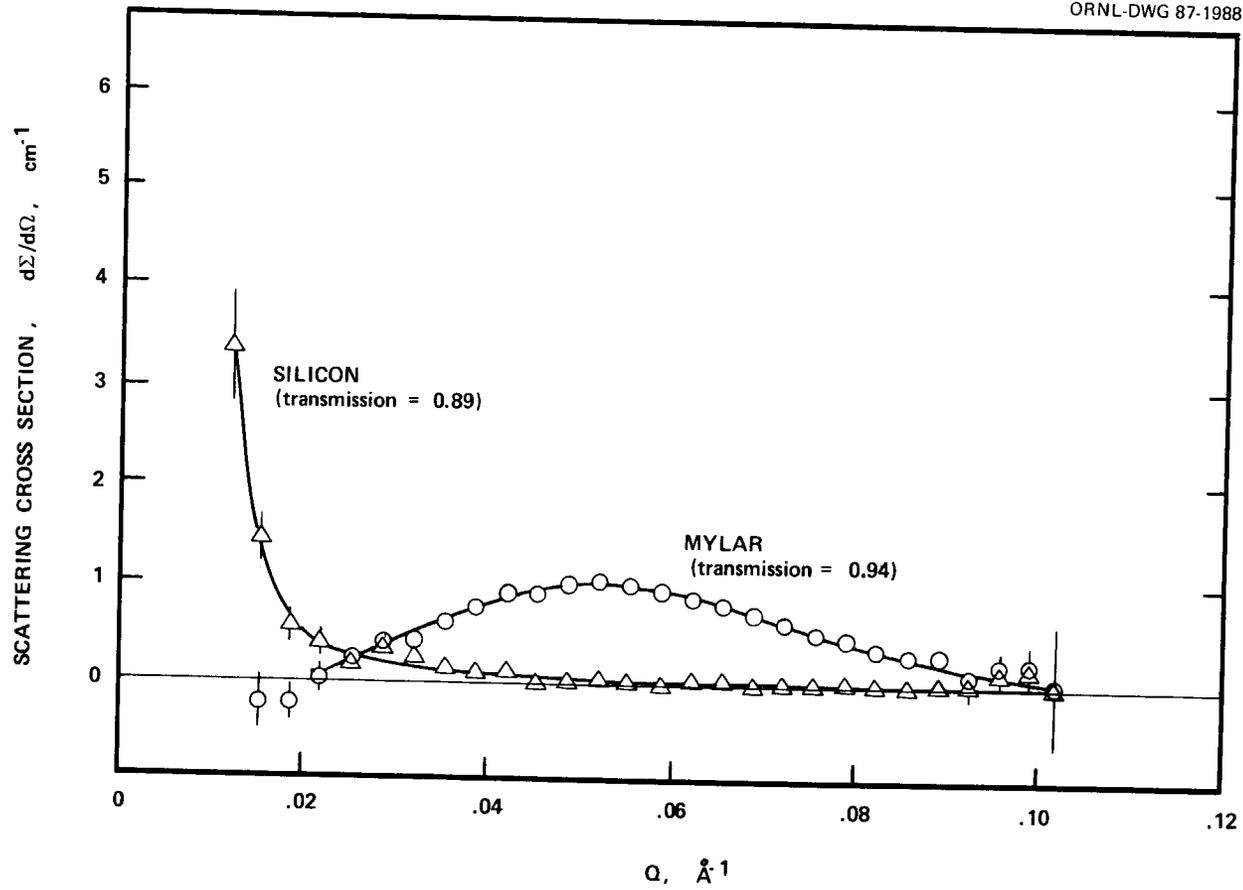


Fig. 12. Scattering cross sections of single-crystal silicon and Mylar tape as a function of scattering angle.

interest scattering by the silicon window is significantly lower than that of the Mylar. (The strong scattering at very low angles is probably from the SiO_2 layer, which was still present on the wafer. This layer can be removed by a brief etch in buffered hydrofluoric acid.)

Because of the superior strength and chemical resistance of single-crystal silicon, we conclude that the conceptual liquid sample holder shown in Fig. 5 is feasible and could make possible new types of X-ray scattering experiments involving liquids, slurries, emulsions, etc.

SUGGESTIONS FOR FUTURE WORK

1. A prototype alpha detector should be built using a full-sized silicon wafer and the photomask shown in Fig. 6b. Adhesives should be investigated to determine the best combination of strength, optical transparency, and index of refraction.

2. Alternate scintillator materials should be investigated with the goal of making a detector that is also sensitive to β - and γ -radiation.

3. A single window like that proposed for the SAXS camera could be coated with scintillator material and attached to the end of a fiber-optic cable. Properly designed, this probe would be miniature, waterproof, and rugged and could be used for remote monitoring of radiation levels in storage tanks, pipes, and other areas.

4. A complete SAXS sample cell should be fabricated and subjected to pressure tests to determine the minimum safe window thickness. The mechanical properties of the gold-silicon solder must be analyzed to determine the adequacy of the soldered joint; a more secure joint configuration could be designed if needed.

CONCLUSIONS

1. Thin windows that are mechanically strong and yet transparent to many kinds of radiation can be fabricated by anisotropic chemical etching of single-crystal silicon.

2. A rugged α -radiation detector has been designed to use etched silicon in place of aluminized Mylar. The new design should be substantially more resistant to environmental contamination and mechanical damage.

3. A sample chamber has been designed for studying liquids by small-angle X-ray scattering. It benefits from the strength and chemical resistance of single-crystal silicon as well as the ability to form strong, airtight joints by soldering to low-expansion alloys.

ACKNOWLEDGMENTS

S. Spooner measured the X-ray absorption and scattering, and M. M. Chiles measured the α -transmission. The report was prepared for publication by A. R. McDonald.

REFERENCES

1. K. E. Peterson, "Silicon as a Mechanical Material," *Proc. IEEE* **70**(5), 420-57 (1982).
2. J. M. Borky and K. D. Wise, "Integrated Signal Conditioning for Silicon Pressure Sensors," *IEEE Trans. Electron. Devices* **ED-26**, 1906 (1979).
3. L. M. Roylance and J. B. Angell, "A Batch-Fabricated Silicon Accelerometer," *IEEE Trans. Electron. Devices* **ED-26**, 1911 (1979).
4. K. E. Peterson, "Micromechanical Membrane Switches on Silicon," *IBM J. Res. Dev.* **23**, 376 (1979).
5. S. C. Terry, J. H. Jerman, and J. B. Angell, "A Gas Chromatograph Air Analyzer Fabricated on a Silicon Wafer," *IEEE Trans. Electron. Devices* **ED-26**, 1880 (1979).
6. J. C. Greenwood, "Ethylene Diamine-Catechol-Water Mixture Shows Preferential Etching of p-n Junction," *J. Electrochem. Soc.* **116**(9), 1325-6 (1969).
7. A. Bohg, "Ethylene Diamine-Pyrocatechol-Water Mixture Shows Etching Anomaly in Boron-Doped Silicon," *J. Electrochem. Soc.* **118**(2), 401-2 (1971).
8. R. W. Hendricks, "The ORNL 10-Meter Small-Angle X-ray Scattering Camera," *J. Appl. Crystallogr.* **11**, 15-30 (1978).

INTERNAL DISTRIBUTION

- | | | | |
|--------|-------------------------------|--------|-----------------------------|
| 1-2. | Central Research Library | 48. | A. J. Moorhead |
| 3. | Document Reference Section | 49. | N. H. Packan |
| 4-5. | Laboratory Records Department | 50. | A. C. Schaffhauser |
| 6. | Laboratory Records, ORNL RC | 51. | V. K. Sikka |
| 7. | ORNL Patent Section | 52. | G. M. Slaughter |
| 8-12. | M. L. Bauer | 53. | C. J. Sparks |
| 13. | H. R. Brashear | 54. | S. Spooner |
| 14. | W. L. Bryan | 55-57. | P. T. Thornton |
| 15. | M. M. Chiles | 58. | P. F. Tortorelli |
| 16. | D. F. Craig | 59. | J. R. Weir |
| 17. | J. H. DeVan | 60-64. | R. F. Wood |
| 18. | C. K. Dubose | 65. | H. D. Brody (Consultant) |
| 19. | B. G. Eads | 66. | G. Y. Chin (Consultant) |
| 20. | J. I. Federer | 67. | F. F. Lang (Consultant) |
| 21-25. | P. H. Fleming | 68. | W. D. Nix (Consultant) |
| 26. | T. J. Huxford | 69. | D. P. Pope (Consultant) |
| 27-46. | R. J. Lauf | 70. | E. R. Thompson (Consultant) |
| 47. | S. A. McElhaney | | |

EXTERNAL DISTRIBUTION

71. DOE, OAK RIDGE OPERATIONS OFFICE, P.O. Box E, Oak Ridge, TN 37831
Assistant Manager for Energy Research and Development
- 72-176. DOE, TECHNICAL INFORMATION CENTER, P.O. Box 62, Oak Ridge,
TN 37831

For distribution as shown in DOE/TIC-4500, Distribution
Category UC-37 (Instruments).

1

2

3

4