EFFECT OF RADIATION ON THE STABILITY OF SILICON NITRIDE AND METAL NITRIDE SEMICONDUCTOR FET DEVICES

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ABSTRACT

This report summarizes the supplementary experimental work that was performed for the study of the "Effect of Radiation on the Stability of Silicon Nitride and Metal Nitride Semiconductor FET Devices." This is divided into three main work areas:

1. The preparation of the silicon nitride films and the evaluation of those films as a capacitor dielectric.

2. Etch rate enhancement of the Si_3N_4 films due to irradiation.

3. Radiation test equipment set-up, and evaluation of the test procedure.

FOREWORD

This first quarterly report was prepared by the Solid State Research Center, Hughes Aircraft Company, Newport Beach, California on NASA Contract NAS 8-18003, entitled, "Effect of Radiation on the Stability of Silicon Nitride and Metal-Nitride-Semiconductor Field Effect Devices," for work performed during the period from July 1, 1966 to October 1, 1966.

The studies were performed at the Solid State Research Center at Newport Beach and Malibu, and at the Radiation Laboratory, at Fullerton, under the general direction of Mr. Richard J. Belardi.

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I. INTRODUCTION

The purpose of this program is twofold:

(1) To study the effect of radiation on etch rate enhancement of the silicon nitride films, and

(2) To compare the effect of gamma radiation on the Metal-Oxide-Semiconductor versus the Metal-Nitride-Semiconductor Insulated Gate Field Effect Transistor.

Literature survey shows that enhanced etch rates following proton and electron bombardment has been already achieved in silicon dioxide layers.[(1)] More recently, in an abstract of a paper to be presented soon, T. W. O'Keefe[(2)] mentions the feasibility of electron irradiation in preparing simple planar nitride devices without the use of etch resists. Since radiation has an effect on the silicon nitride properties, which are also affected by the methods and conditions of growth,[(3)] it is important that these properties be understood and brought under control during this initial phase of the program. Moreover, the successful preparation of Metal-Nitride-Semiconductor devices, which is essential to this program, depends largely on the control of these film properties.

A supplemental program was therefore initiated with emphasis on the evaluation of the silicon nitride characteristics. During this interim, the art of growing the nitride film has been gradually reduced in temperature to 600°C from the previous 800°C level, which is a factor in reducing possible effects due to contamination. The film was evaluated in the preparation of planar low leakage junctions and as a capacitor dielectric. Feasibility was demonstrated and the results are presented in Section II.

Simultaneously, with the above study, investigations were carried out in radiation techniques and their evaluation as a means for enhancing etch rates in the silicon nitride. Argon and Krypton ion sources were investigated at different energy levels. An electron source from an electron beam welder was also given an evaluation. Gamma radiation from a copper x-ray tube and a cobalt 60 source were also tried. A discussion of this work appears in Section III of this report.

Under the gamma radiation effects studies, Section IV, the related test equipment was set up and checked. Hughes MOS-FET's were manufactured and the necessary pre-irradiation measurements were taken. Some preliminary irradiation tests were also performed for the purpose of evaluating the radiation test procedures and requirements.

II. SILICON NITRIDE PREPARATION

A. Growth Conditions, Reproducibility and Control

Silicon nitride films are prepared in an r.f. heated environment by the pyrolysis of silane and anhydrous ammonia at temperatures above 575°C (Fig. 1). The product of the reactants is as follows:

$$3\ SiH_4 + 4\ NH_3 \rightarrow Si_3N_4 + 12\ H_2$$

The chemically cleaned, single crystal silicon wafer is introduced into the reaction chamber under dry nitrogen conditions. The chamber is next closed and the system is purged with hydrogen. At approximately 600°C the silane and the ammonia are introduced into the reaction chamber and the silicon nitride growth is monitored. It is possible to visually observe the color change due to the increase in thickness during film growth. Once the desired thickness is achieved, the SiH_4 - NH_3 is turned off and the system is allowed to purge with H_2 during the cool-down cycle. This is followed by a final N_2 purge prior to sample removal.

The nitride film is uniform in thickness and reproducible from run to run, as evidenced by interference colors and voltage breakdown measurements. Initial results show, that the device quality film is susceptible to preheat treatment. It is not known, at this time, if

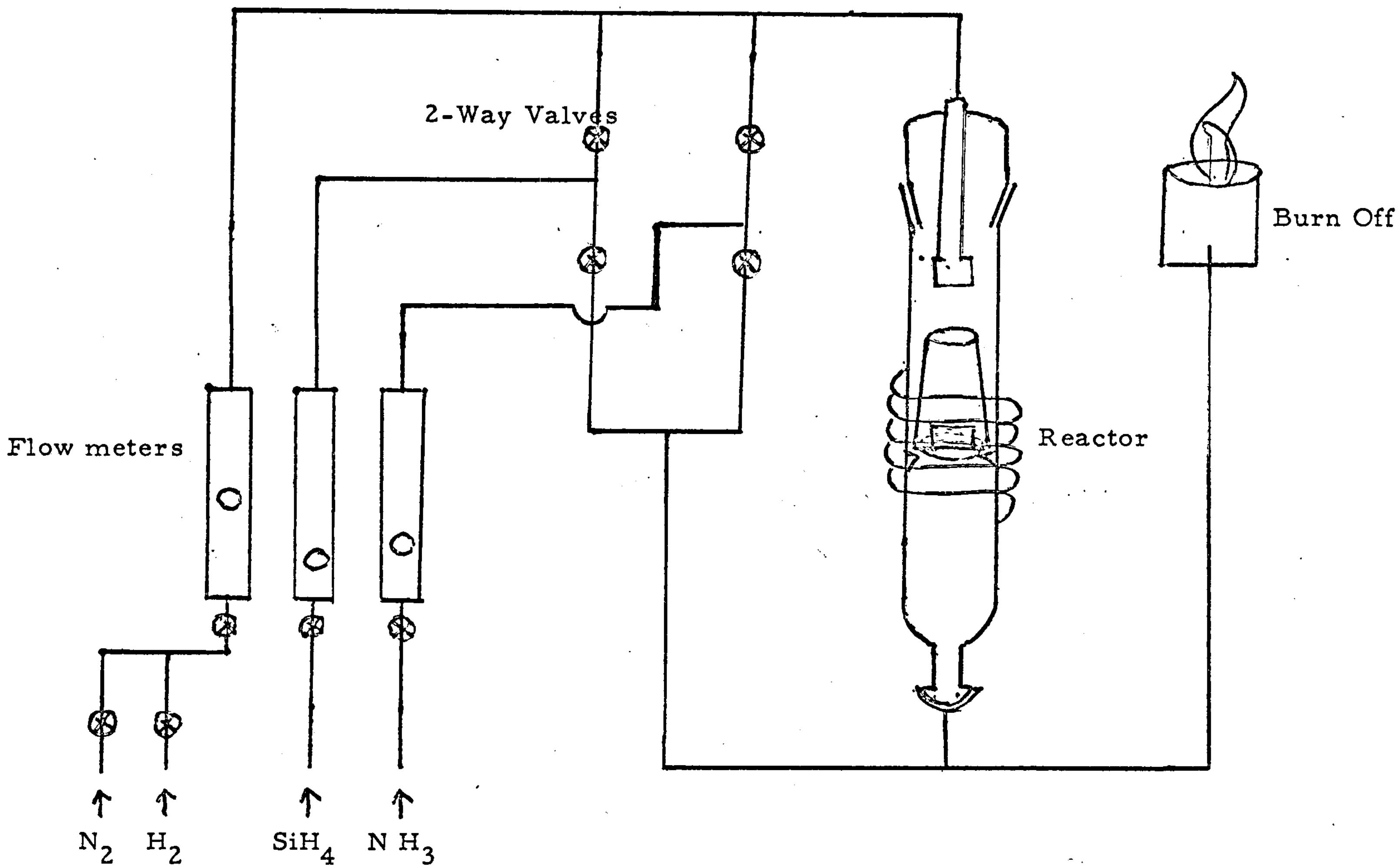


Figure 1. A Schematic for the Pyrolytic Deposition of Silicon Nitride (Si_3N_4)

this is due to contamination problems, or to the hydrogen heat treatment. In absence of this treatment, however, better quality films were obtained.

Growth rate of the nitride film is sensitive to the ammonia-silane ratio and their concentration in the gas stream. At higher temperatures, the growth rate is enhanced several-fold, as illustrated in Fig. 2.

Table I below gives certain growth conditions and film thicknesses:

TABLE I

Reaction Temp. $\pm$ 15°C	Gas Flows SiH_4	NH_3	H_2	Growth Time Minutes	Thickness Å $\pm$
800	31	51	26	15	2000 $\pm$ 150
700	56	51	26	15	1800 $\pm$ 150
600	104	137	2	12	1200 $\pm$ 150
700	104	137	2	12	3.6 microns
800	104	137	2	12	7.0 microns surface shattered

The Si_3N_4 film thicknesses at 700°C and similarly at 800°C demonstrate in each case two extremes of growth rates obtained as a result of varying SiH_4/NH_3 ratio and concentration. In comparison, the reaction at 600°C under optimum growth conditions has become temperature limited.

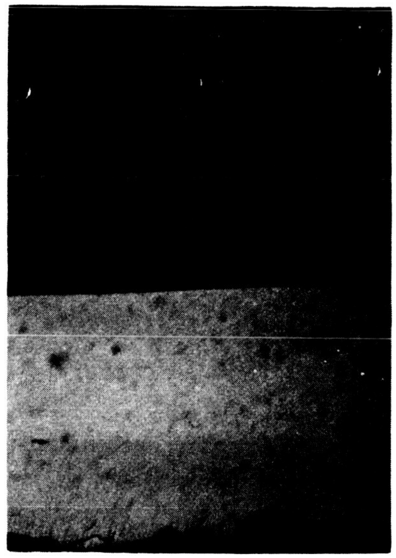


Fig. 2a. Deposit Temperature $600^{o}C$. Thickness 1200 Å

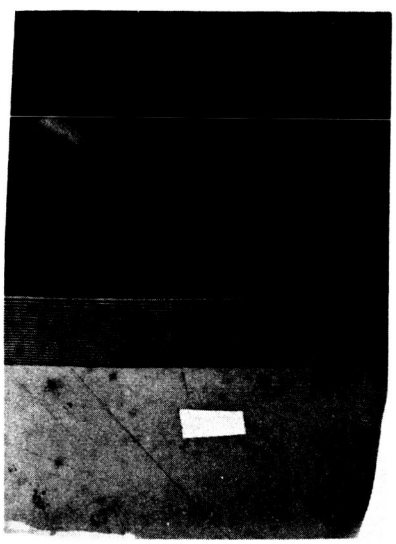


Fig. 2b. Deposit Temperature $700^{o}C$.
Thickness 3.6 microns

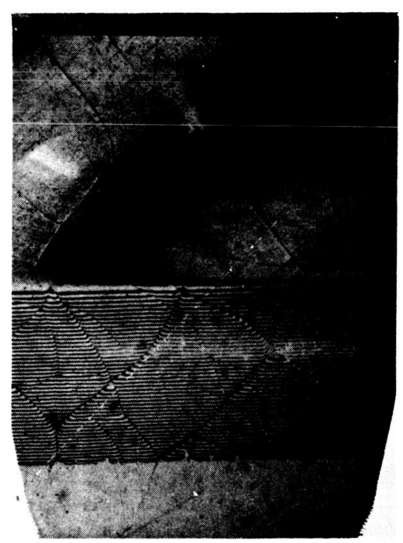


Fig. 2c. Deposit Temperature $800^{o}C$
Thickness 7.0 microns

FIGURE 2. ANGLE SECTION. SILICON NITRIDE GROWTH versus TEMPERATURE

B. Etchants for the Nitride Film

The silicon nitride film is soluble in hydrofluoric acid solutions at room temperature and in phosphoric acid solutions at elevated temperatures. Both of these etchants do not attack silicon.

Table II below gives some approximate values of etch times in HF and phosphoric acid for a 2000 Å pyrolytic Si_3N_4 film.

TABLE II

ACID	TEMP.	TIME
48% HF	25°C	3 - 3-1/2 min.
12% HF	"	9 - 10 min.
4.8%	"	32 - 35 min.
85% H_3PO_4	"	No etch in 15 hours
85% H_3PO_4	102°C	20 - 25 min.
42.5% H_3PO_4	"	40 - 45 min.
21%	"	75 - 85 min.
10.5%	"	130 - 140 min.

It is obvious from the above table that the nitride films are slow to etch, and under the above conditions it would be impossible to mask directly by present known masking techniques. A lifting of the photoresist mask will occur prior to complete Si_3N_4 removal. An indirect solution to this problem is the use of an SiO_2 mask. Fig. 10 demonstrates the feasibility of this technique.

C. Silicon Nitride Films as a Mask for Diffusion

Silicon nitride films make a very good mask for the boron impurity (Fig. 3) which is used in the diffusion of source and drain for a "p" channel IGFET device. The diffusion constant for boron in silicon nitride is definitely smaller than that of SiO_2. This means that nitride films with a fraction of the SiO_2 film thickness can be used effectively for masking against similar diffusions. In addition to boron, the nitride film can also be used to mask against other diffusion impurities.

The silicon nitride film, however, has its problems. At temperatures higher than $1100^{o}C$, and in strong oxidizing atmospheres, conversion of the nitride film to SiO_2 occurs. The use of mild oxidizing atmosphere or preferably non-oxidizing conditions during any extended high temperature heat treatment operation is deemed necessary.

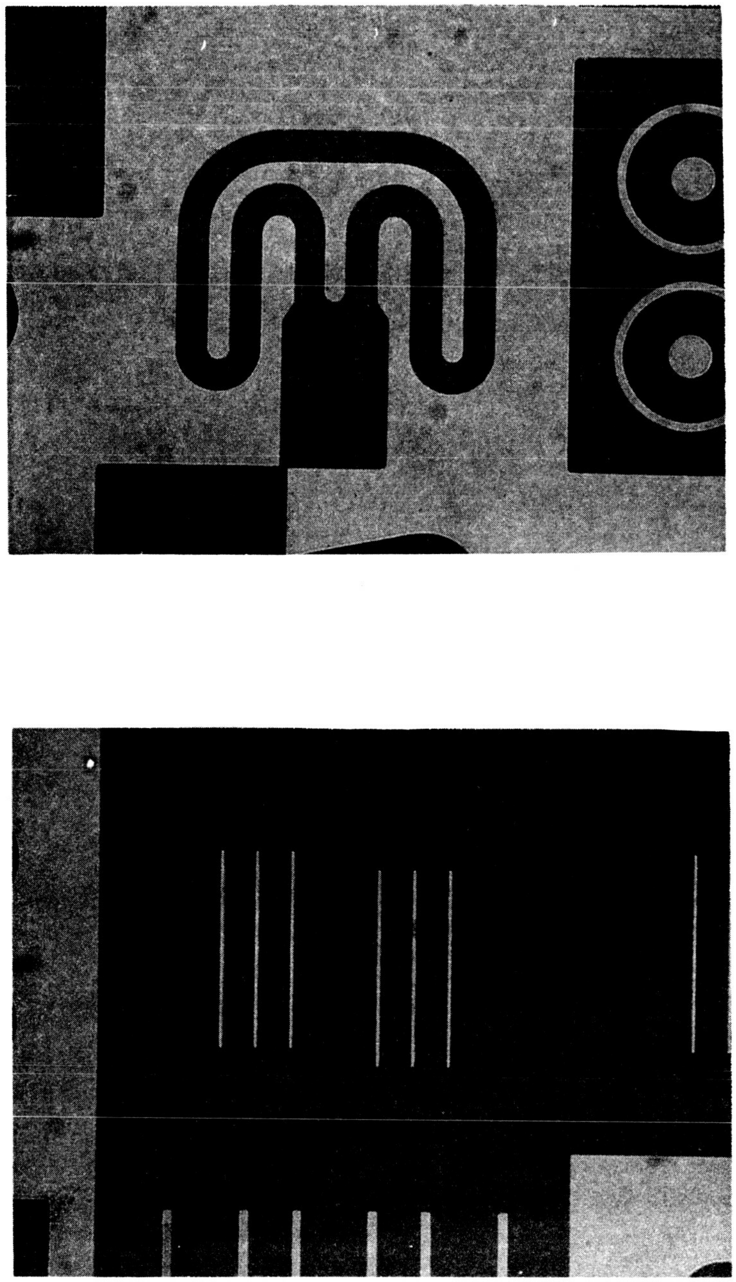


FIGURE 10. Phosphoric Acid Etch Pattern Using Pyrolytic SiO_2 Film as a Mask

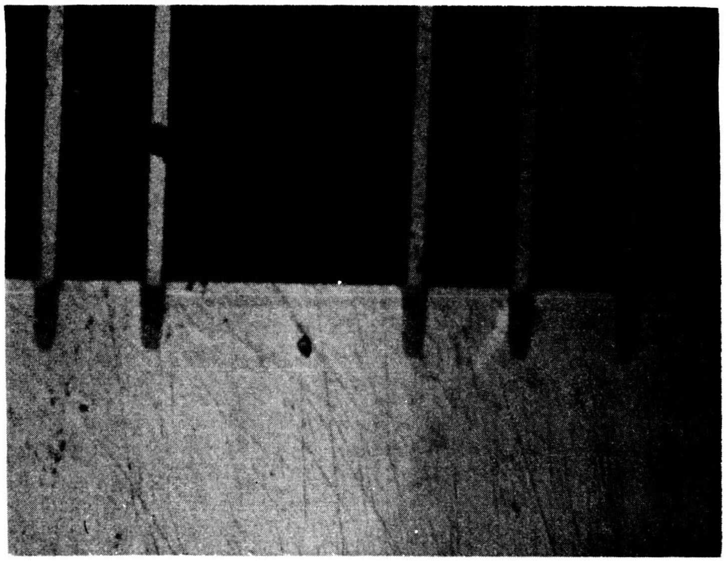


FIGURE 3. Silicon Nitride as a Diffusion Mask

D. Capacitor Measurement

The circuit in Fig. 4 is used to display the differential capacitance of MNS capacitors as a function of applied d.c. voltage. The d.c. bias is supplied by a sawtooth generator. A small r.f. signal is superimposed on the d.c., and the resulting r.f. current through the 1 K Ω resistor is proportional to the capacitance of the device being tested. This is displayed vertically on the scope, while the d.c. voltage is displayed horizontally. A sharp drop in capacitance occurs at the threshold voltage, allowing measurement of the threshold voltage with an accuracy of $\pm$ 1 volt. By shifting the voltage range over which the d.c. bias is swept, the presence (or absence) of polarization in the nitride can be determined. Other faults in the nitride can sometimes be detected by anomalies in the C-V curve.

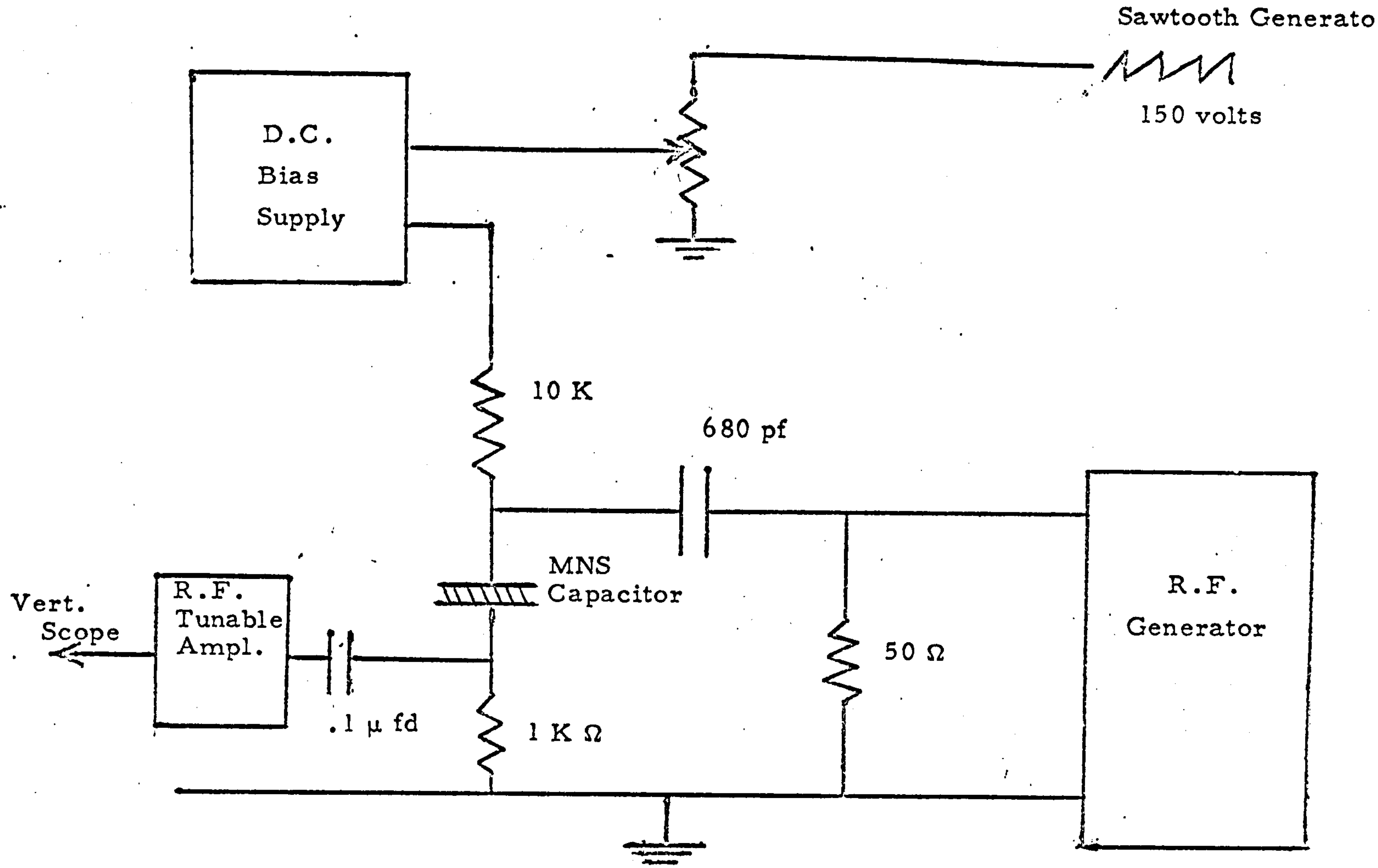


Figure 4. Circuit for Capacitance-Voltage Measurement

The samples prepared for this experiment are for the study of the effect of preheat treatment on the quality of the silicon nitride film. In the one case, the film was prepared in the normal manner, where the SiH_4-NH_3 mixture was introduced as soon as the growth temperature of 800°C is reached. This is referred to here as a no-preheat cycle. In another test, a preheat of one hour at 800°C preceded the introduction of the SiH_4-NH_3 mixture. To maximize any contribution due to preheat treatment, a third experiment was performed with the preheat conditions at 1150°C followed by a return to 800°C for the normal growth of the silicon nitride film. The results for these three conditions are reported below.

Figure 5a shows C-V curves on a sample MNS capacitor which was fabricated using no preheat treatment. In Fig. 5a, the applied voltage is swept from 0 volts (extreme left) to -40 volts (extreme right). Threshold occurs at about -6.5 volts. In Fig. 5b, a +20 volt d.c. bias is inserted into the circuit, so that the voltage sweeps from +20 volts (left) to -20 volts (right). Threshold now occurs at -3.5 volts, indicating polarization in the capacitor. Fig. 5c shows the I-V loop in more detail, with no discernible ionizing traps except perhaps just below threshold. The test, involving a preheat treatment of 1 hr. at 800°C showed similar results to no preheat conditions.

Fig. 6 shows the same kind of C-V displays on a sample which was preheated at 1150°C. A much greater degree of polarization is evident, along with the presence of some traps.

C-V DISPLAY OF THE MNS CAPACITOR

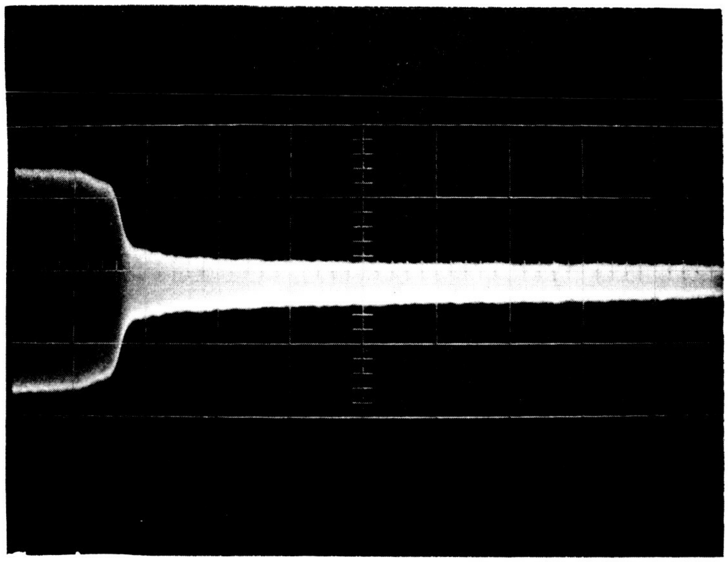


Figure 5a. Voltage Sweep 0 to -40 volts

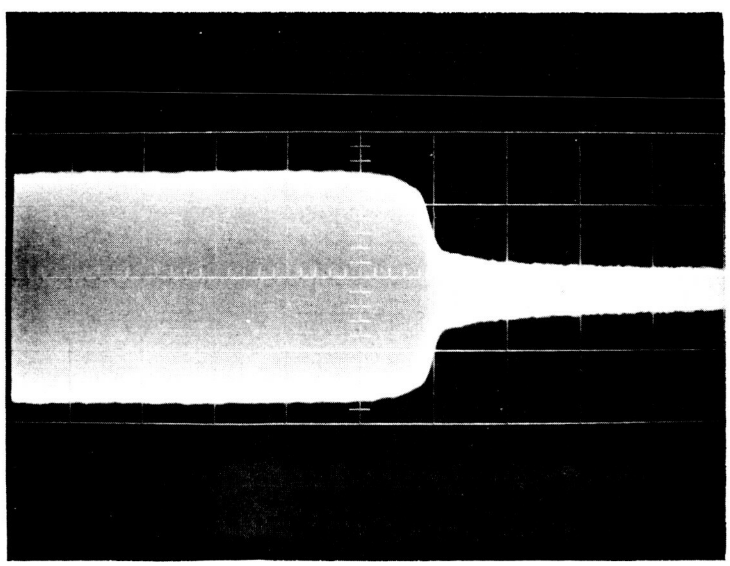


Figure 5b. Voltage Sweep +20 volts to -20 volts at -20 volts.

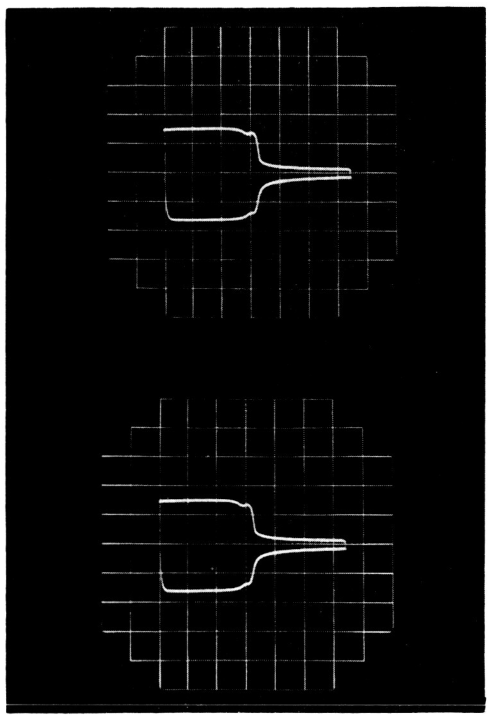


Figure 5c. IV Loop at Room Temperature.

$$i = C\frac{dv}{dt}$$

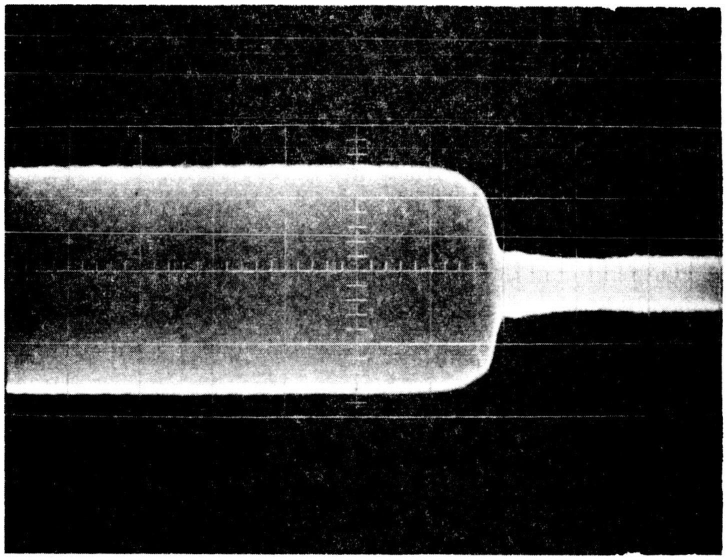


Figure 6a. Voltage Sweep 0 to -40 volts

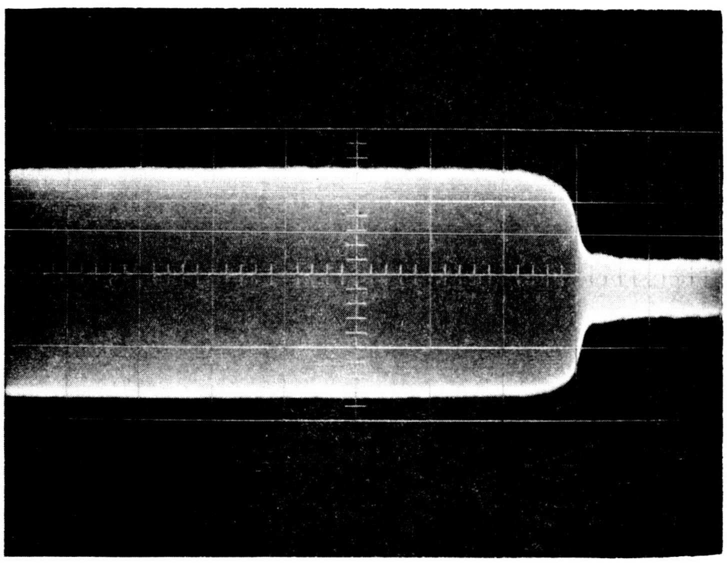


Figure 6b. Voltage Sweep +20 V to -20 V
at -20 V

FIGURE 6. C-V DISPLAY OF THE MNS CAPACITOR WITH H_2 PREHEAT.

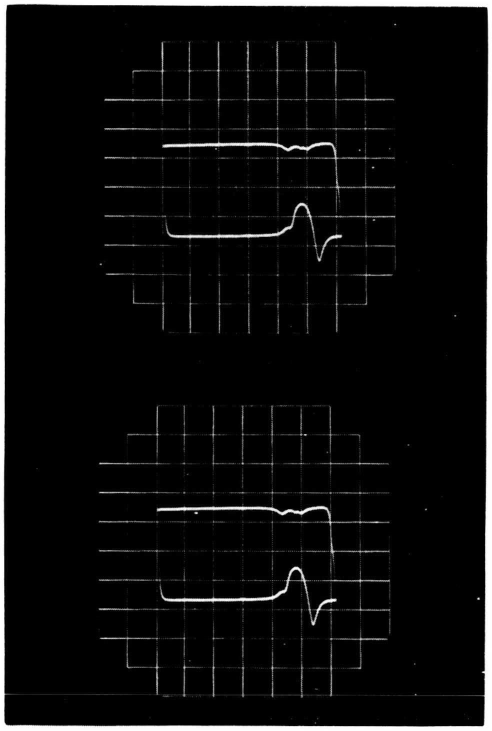


Figure 6c. IV Loop at Room Temperature $i = C\frac{dv}{dt}$

The technique just described was useful for a quick comparison of several samples, all of which exhibited considerable polarization. However, the sweeping d.c. bias suppresses much of the polarization. Only after applying a static bias for a considerable time can the maximum polarization be seen. Application of -30 volts for 1 minute to the sample shown in Fig. 5 shifts V_T to -23.5 volts.

III. ENHANCEMENT OF ETCH RATE BY IRRADIATION (Dr. H. L. Garvin)*

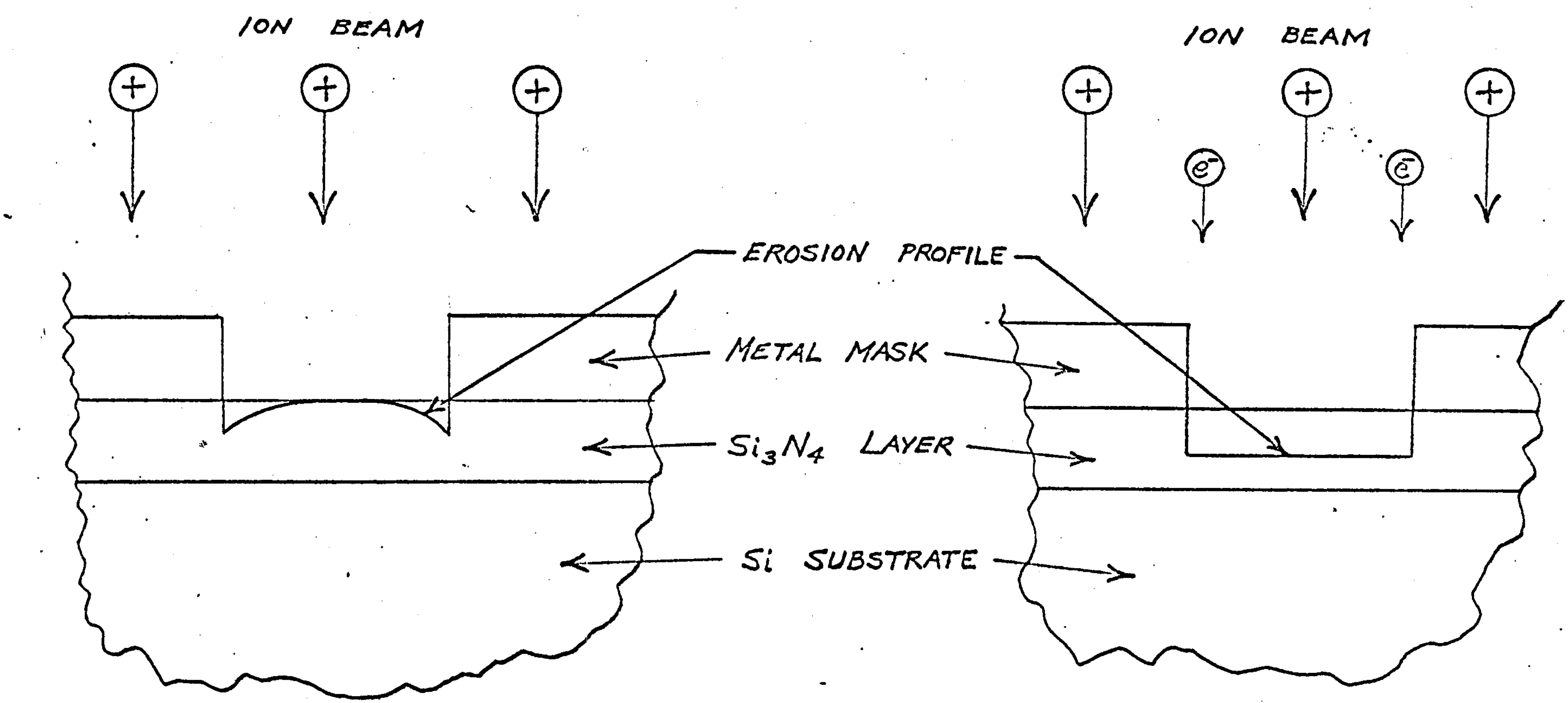
A. Ion and Electron Source Exposure

In order to investigate the possibility of selectively enhancing the chemical etch rate of silicon nitride passivating layers in micro-electronic components, beams of electrons, argon ions, and krypton ions were used to bombard the nitride layers. The layers of Si_3N_4 (typically 1500 to 2000 Å thick) were pyrolytically grown on single crystal silicon substrate wafers. For electron bombardment the wafers were placed in the vacuum chamber of the 3 kW electron beam welder and passes were made over the wafer with a very low-power beam. If electron beam currents of 0.5 mA at 40 keV were used, the thermal shock was sufficient to shatter the wafer. When the beam was de-focussed to approximately 0.25 mA at 40 keV, the wafer did not shatter and two passes were made across the sample. These samples were tested for selective chemical etching and the results showed no apparent etch enhancement when exposed to hydrofluoric acid etchants.

The ion beams of argon and krypton were produced in an electron bombardment ion source, the design of which was developed by means of analog and digital computer studies. The ions were accelerated to between 3 and 7 keV and focussed to give normal incidence to the nitride surface. Ion beam currents were adjusted

*This work has been performed at the Hughes Research Laboratories, at Malibu to a steady level in the range of 30 to 100 $\mu A/cm^2$ at the target. Preliminary experiments indicated that the silicon nitride could be completely removed by sputtering from the silicon substrate and thus chemical etching would not be required at all. In order to determine if etching enhancement occurred due to the ion bombardment, several of the samples were bombarded only long enough to remove approximately half of the nitride layer. Subsequent chemical etching removed the remaining part of the nitride layer in less time than required to remove the entire film, but the time required suggests that the ion bombardment accomplished a partial removal and no noticeable enhancement of etch rate was observed for the remaining part of the layer.

Due to the high electrical resistivity of the silicon nitride films, the uniformity of ion beam erosion was found to be strongly influenced by the accumulation of positive surface charge injected by the positive bombarding ions. Interferometer measurements were made of the erosion depths made by bombardment with 5 keV argon ions through 0.040 in. diameter holes in a metallic mask. Without beam neutralization the erosion pattern is as shown in Fig. 11(a), whereas the addition of neutralizing electrons to the beam and to the nitride surface resulted in uniform erosion as shown in Fig. 11(b). The extent to which surface charging takes place is dependent upon the secondary electron yield and the thickness of the material. In order to insure uniformity of the samples and to retain the fine details of



(a) UNNEUTRALIZED ION BEAM

(b) NEUTRALIZED ION BEAM

Fig. 11. Comparison of erosion profiles due to neutralized and unneutralized ion beams.

micromachining, the ion bombardments for this program have been made in the presence of low energy (typically 90 V) neutralizing electrons.

Having established the feasibility of controlled removal of silicon nitride from silicon substrates it then became necessary to produce metal-nitride-semiconductor (MNS) devices which could be compared to MOS devices. Although work is in progress to develop finely focussed ion beams for maskless production of MNS devices, the most significant comparisons can be made at this time by using the existing designs and masks which are used to produce MOS devices and apply them directly to produce MNS devices. Thus, by photo techniques contact masks were applied directly to the surface of the nitride and the windows in this mask defined the areas from which the nitride was removed by the ion beam. Although a portion of the mask is also removed by the ion beam, its thickness (typically 0.5 to 1.5 μ) is sufficient to remain during the complete removal of the nitride from the window areas. Metallic masks of chromium and aluminum were used first (Fig. 8) but later samples showed that SiO_2 (Fig. 9) and even 1 μ-thick layers of Kodak KTFR resist (Fig. 7) itself would serve as an adequate mask. During ion bombardment of the photoresist material a physical change takes place which may harden the material to withstand the ion bombardment and definitely makes the material less susceptible to normal chemical removal techniques.

The quality of image produced in the nitride with each of these masks is as good as the image in the mask and no evidence of undercutting is observed. Test device patterns have been ion beam micromachined into Si_3N_4 layers on silicon substrates and also into nitride layers which have been grown over diffusion-doped source and drain regions of the substrate. Studies are now in progress to determine the effects of the ion bombardment in these doped regions.

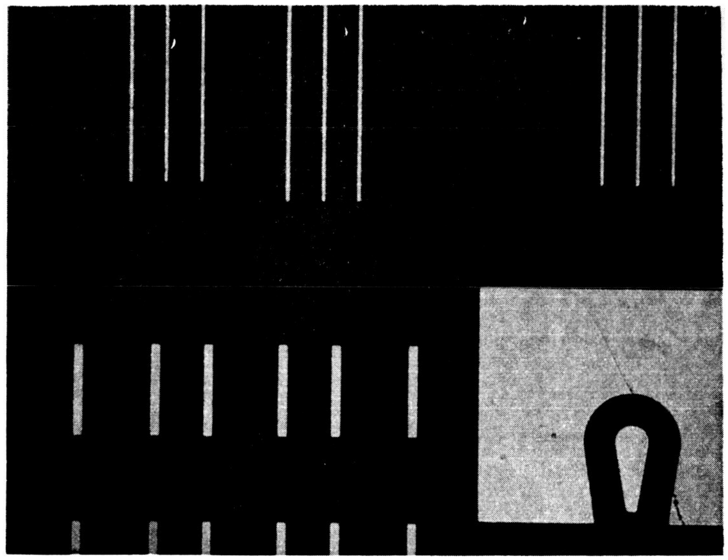


Figure 7. Argon Beam Exposure Pattern Using Kodak KTFR Film as a Mask

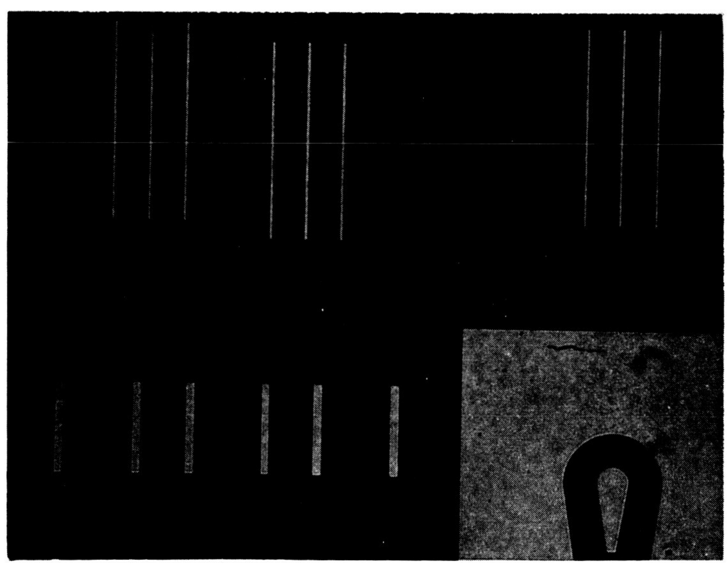


Figure 8. Argon Beam Exposure Using Evaporated Aluminum Film as a Mask

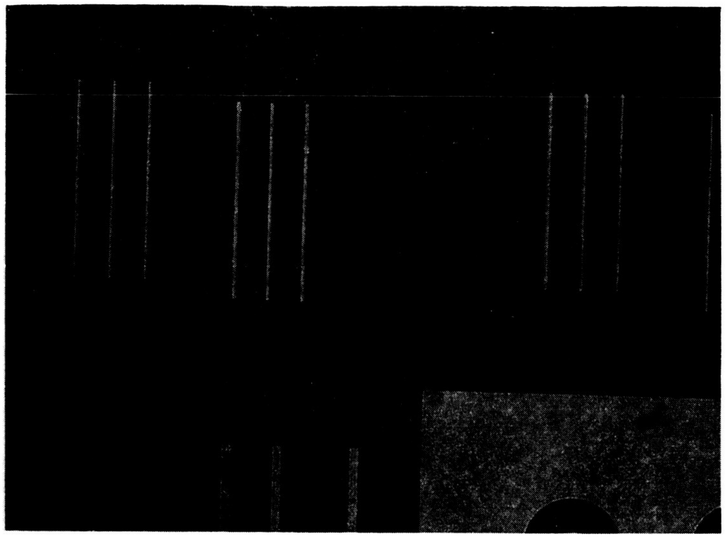


Figure 9. Argon Beam Exposure Pattern Using Pyrolytic SiO_2 Film as a Mask

B. Gamma Ray Exposure

Two sources of gamma rays were employed in the exposure of silicon nitride to gamma radiation, for the purpose of possible etch rate enhancement, namely, the Norelco X-ray diffraction unit and the cobalt 60 source chamber.

The X-ray source has a maximum input rating of 45 KV at 15 ma, and uses a water-cooled copper tube located at a distance of one cm from the sample. A silicon nitride and a SiO_2 coated silicon wafer were simultaneously exposed to maximum intensity for two hours. Upon repeatedly exposing the samples to 12% HF etch, no indication of preferential etching was noticed. The x-ray test was repeated for a 16-hour exposure time at maximum intensity. No preferential etching was noticed.

In another experiment, a Si_3N_4 coated silicon wafer was irradiated with a cobalt 60 source at 1.2×10^6 rads for a one hour duration. Evidence of etch rate enhancement was not noticeable. All above tests were performed on approximately 2000 Å Si_3N_4 films, and 3000 Å SiO_2 films.

IV. GAMMA RADIATION EFFECTS STUDIES (R. W. Marshall)*

A. Introduction

The effects of gamma radiation on the parameter stability of insulated-gate field-effect transistors (IGFETS) has been studied extensively by several investigators including Hughes during the past two years.[4, 5] These studies have shown that the gate turn-on threshold voltage, V_T, is the IGFET parameter which is most sensitive to gamma and other types of ionizing radiation. The change in the gate turn-on threshold voltage has been found to be proportional to the total radiation dose and to be dependent upon the gate bias applied during irradiation. Significant changes in V_T, i.e. ≥ 0.1 volt, have been measured for gamma doses of 1×10^3 rads (Si). Changes in transconductance and mobility do not occur until gamma doses on the order of 1×10^6 rads (Si) have been accumulated.

It is believed that the damage mechanism involved in the change in V_T is similar to the oxide ion migration problem which causes V_T to change when MOS transistors are operated at elevated temperatures ($80^\circ C$ - $100^\circ C$) with the gate reverse biased. Gamma radiation injects charges into the silicon dioxide dielectric and into the interface between the oxide and the silicon channel by ionizing the device materials. When these mobile ions are trapped in the insulator or at the interface, a static charge is built up which changes the magnitude of V_T.

*This work has been performed at Hughes Aircraft Company, Radiation Laboratories, Fullerton.

Recent IGFET studies[6] have shown that the gate turn-on threshold voltage of devices which use silicon nitride as the gate-channel insulator is more stable than that of MOS FETS in an ionizing radiation environment. For this reason p-channel enhancement MOS FETS and p-channel enhancement MNS FETS will be fabricated from identical masks and the parameter stability of the two device types will be compared in a gamma radiation environment.

B. Radiation Test Program

As previously stated, the MOS transistor parameter which is most sensitive to gamma radiation is the gate-threshold voltage, V_T. The mechanism by which radiation causes V_T to change is in part associated with surface effects. The measurements which determine the comparative parameter stability between MNS and MOS IGFETS will, therefore, include the parameter measurements which give an indication of gate-threshold voltage and surface changes. These parameters are:

1. Gate-threshold voltage V_T
2. Drain-source leakage current, I_{DSS}
3. Gate-source leakage current, I_{GSS}
4. Drain-source breakdown voltage, BV_{DSS}
5. Gate-source breakdown voltage, BV_{GS}

The gate-source breakdown voltage test, BV_{GS}, is often destructive due to the creation of hot spots in the thin dielectric (1000 Å) when high fields are present. For this reason, BV_{GS} will be measured for one typical device after each radiation dose interval. If the test proves to be destructive, a total of eight devices will be destroyed during the test series. All other measurements will, therefore, be made before the BV_{GS} test is made in order to obtain a maximum amount of information.

In addition, measurement of the V_D - I_D characteristics of the devices will provide information concerning the following device parameters:

1. Transconductance, g_m
2. Gate-threshold voltage, V_T
3. Effective mobility, μ_{eff}

The MOS and MNS structures will be simultaneously and uniformly irradiated with Co^{60} or other appropriate gamma ray sources (e.g., 10 MeV electron Bremsstrahlung from the Hughes Research Linac). Measurements of device parameters will be made before and after irradiation of 4 dosages. Dosages to devices will be determined with conventional integral and time resolved dosimetry such as fluorads and silicon p-i-n diodes.

The four dosages will be 10^2, 10^3, 10^4, and 10^6 rads (Si). These levels were selected based on knowledge of radiation dose sensitivity of commercial MOS devices. Specifically, significant surface effects begin near 1000 rads (Si) and bulk effects begin to mask surface effects near 10^6 rads (Si). These dose levels, therefore, allow determination of parameters changes in the MOS and MNS IGFETS.

C. <u>Progress to Date</u>

Commercially available p-channel enhancement MOS FETS were purchased from Texas Instruments and Fairchild (FI100). In addition, Hughes p-channel MOS FETS were manufactured from the MNS masks. Detailed electrical measurements were performed on these devices in order to provide the required pre-irradiation characteristics.

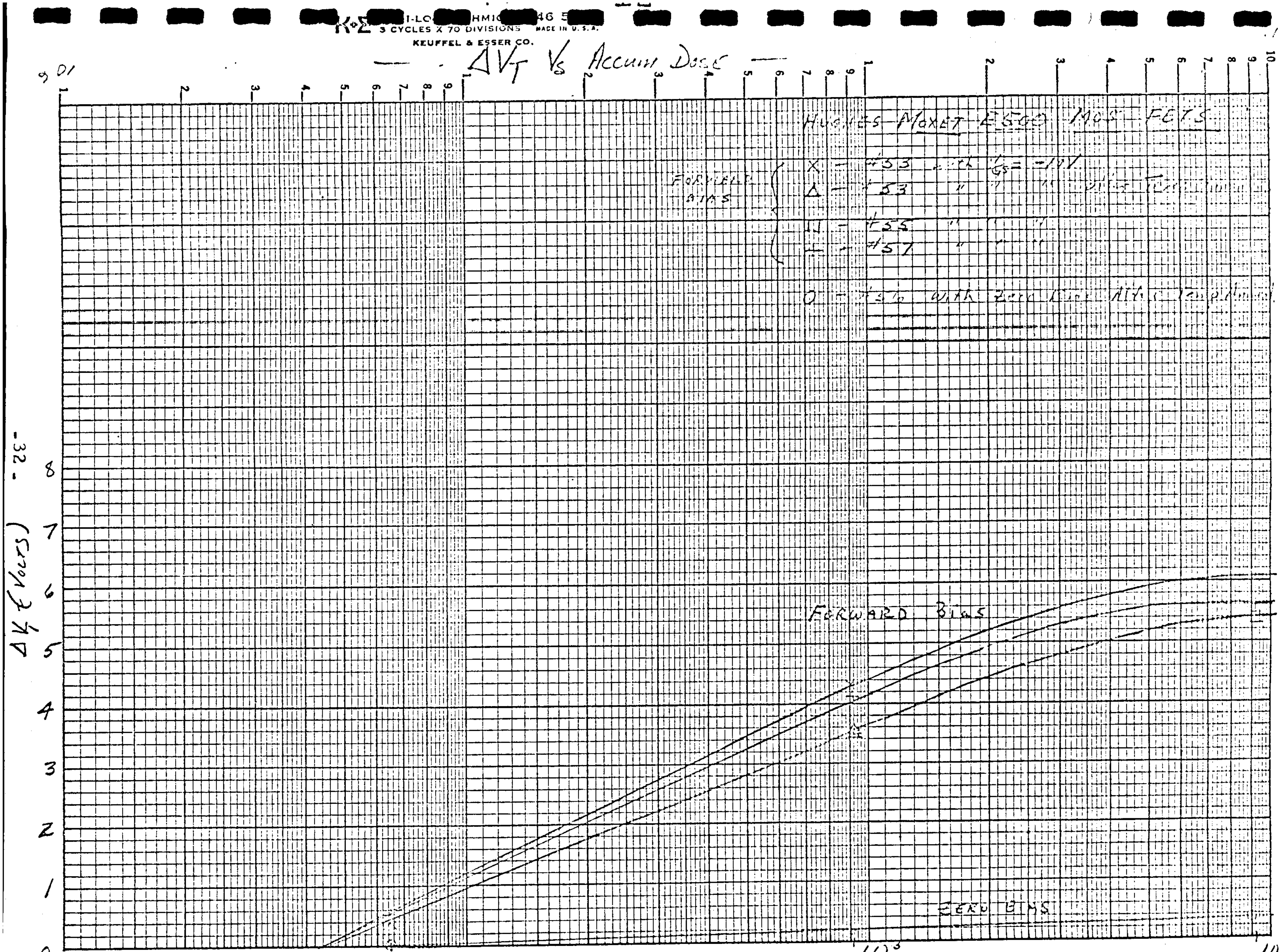
Ten of the Hughes devices were irradiated at doses up to 10^6 rads (Si) in order to check out the radiation test fixtures and evaluate the test procedures. The preliminary irradiation of the Hughes MOS devices resulted in the following conclusions:

1. The gate turn-on threshold voltage, V_T, is more sensitive to gamma radiation when forward biased. (Reference Figure 12).

2. The radiation dose threshold level is 1×10^4 rads (Si) when biased at $V_{GS} = -10$ volts (forward biased).

3. No other significant parameter changes were noted at doses up to 1×10^6 rads (Si).

4. The threshold voltage, V_T, can be annealed to its pre-irradiation value by shorting all external leads together and heating to $300^{\circ}C$ for ten minutes.



ΔV_T Vs Accum Dose
Hughes Moxet E500 MOS FETS
X – #53 with V_GS = -11V
Δ – #53
□ – #55
+ – #57
Forward Bias
O – #5? with Zero Bias
Forward Bias
Zero Bias
ΔV_T (Volts)
Accumulated Dose in Rads (si)

FIGURE 12.

V. CONCLUSIONS AND PLANS FOR NEXT QUARTER

The study of certain silicon nitride film properties as a prerequisite for the successful manufacture of insulated gate devices was considered valuable and essential. The ability to selectively etch the film, use it as a mask during diffusion and as a dielectric in the preparation of metal-nitride-semiconductor capacitors was demonstrated. The polarization effects observed during these capacitor measurements appear to be sensitive to the conditions of the nitride film growth and it is not known at this time if this is a property of the film itself.

Etch rate enhancement of Si_3N_4 films by electron, gamma, and ion irradiation was evaluated. The electron radiation source exposure under the conditions of this experiment gave no noticeable evidence of increased etch rate in the silicon nitride. Similarly, exposure to a maximum dose of gamma radiation from a Co 60 source and from an x-ray copper tube gave no significant results of etch rate enhancement. Ion sources, however, using argon and krypton proved very successful in partially or fully removing the nitride film without resort to chemical etching. Underlying junctions in the bombarded region appear to retain their low leakage properties.

Selective masking of the nitride film during ion beam exposure was evaluated thru the use of three different masks, namely, aluminum, Kodak photoresist (KTFR), and pyrolytic oxide. The aluminum mask gave a higher incidence of electrical shorts. The photoresist mask was hardened during exposure and presented difficulties in removal. The oxide did not present any problems and therefore, will be used in future selective nitride masking by ion irradiation. All films, however, gave good resolution.

The test equipment for evaluation of radiation effect studies has been set up and pre-irradiation measurements on Hughes MOS Field Effect devices were taken in order to check out the radiation test equipment and procedure.

MOS devices from Texas Instrument and Fairchild Camera and Instrument Company have been purchased for future comparison with Hughes fabricated MNS Field Effect Transistor under radiation effects.

Plans for the next quarter will include:

1. Continuation of etch rate enhancement studies by irradiation.
2. Pre-treatment of the silicon surface prior to the nitride film deposit and the effects of silicon nitride growth conditions on polarization behavior.
3. Initial fabrication of metal-nitride semiconductor field-effect transistors for the purpose of radiation effects study.

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