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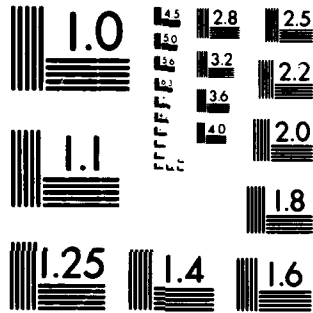
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**Report DAAG 29-77-C-0027
Phase 3**

SPUTTER DEPOSITION OF DIELECTRIC THIN FILMS

**FINAL REPORT
FEBRUARY 1980**

**Prepared by
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ABSTRACT

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1. INTRODUCTION

This effort on the sputtering of thin dielectric films is a logical outgrowth of the work during 1977 - 1978 on evaporated dielectrics.[†] A major reason for this work has been to improve the resolution of the Itek PROM through the development of new single layer and multilayer dielectric coatings. As was demonstrated in the Phase I report coatings having higher dielectric constants than the parylene dielectric layer presently used will increase the resolution markedly. Further, it was shown that an inorganic dielectric layer exhibited ten times better signal-to-noise in the Fourier plane. Rather than repeat a duplicate description of that work here, it is suggested that the reader refer to the Phase I report.[†]

During that study it was found that most of the attractive dielectrics, SiO_2 , TiO_2 , Ta_2O_5 , Y_2O_3 , etc. disassociated sufficiently during evaporation that it was impossible to reconstitute them in a stoichiometric fashion during deposition. Even with extremely slow evaporation rates (1-2 Å/sec) and with maximum oxygen pressure in the chamber (1×10^{-4} Torr), reoxidation was incomplete at substrate temperatures as high as 300°C . Further while materials such as TiO_2 and Ta_2O_5 could be improved by post-baking in air or oxygen, this process generally had no effect on the other materials and if $\text{Bi}_{12}\text{SiO}_{20}$ (BiSOx) was used as a substrate, the substrate was damaged by the high temperatures required. The final blow was the discovery that a SiO_2 layer covering either a TiO_2 or Ta_2O_5 layer completely inhibited the post deposition oxidation of the lower layer. This prevents

[†]Dielectric and Optical Properties of Thin Films, Contract No. DAAG29-77-C-0027 1978.

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the post-baking of multilayer films. For these reasons it was determined that evaporated coatings were not suitable as high optical quality dielectrics in applications requiring thick layers and high electric fields.

Hence the effort was redirected toward a study of sputtered films during the second phase of the program. In addition it was recognized that Itek lacked the capability of performing some of the more abstruse, but nonetheless necessary, measurements on these films. Therefore, a joint program was developed in which Dr. Armand Tanguay of the University of Southern California would provide the needed support. Unfortunately his finding was not approved until Itek was nine-months into the second phase of its program, hence the collaboration to date has been necessarily sketchy. This will be resolved as both programs are on line in Phase III, but as a result, the major results to be reported here concern deposition details, with only that characterization which could conveniently be carried out at Itek.

2. NEW EQUIPMENT AND TECHNIQUES

2.1 SPUTTERING EQUIPMENT

Itek has operated three types of sputtering equipment. The first is a large, but very old, ion beam system. This unit may be used with dielectrics but it has two drawbacks; first it is one of the first of its kind and hence has very poor reliability. In fact, in only one instance during this program was it possible to complete a deposition without at least one breakdown. (The most common failure is burning out of the neutralizing grid.) Second, the substrates run very hot, often exceeding 350°C, and no independent temperature control is possible. Hence work on this machine was quickly terminated in favor of more desirable alternatives.

The second unit is the standard MRC dc diode sputtering system using 5 inch targets. This unit has been in use for some time in the deposition of indium tin oxide for transparent conductive coatings for Itek PROMs. However, it is not readily convertible to rf operation as is required for sputtering of dielectrics. Also, while the substrate surface, either glass or BiSOx, rises uncontrollably to approximately 125°C during the desposition, simply because the substrate is in the ionic path and hence is subject to the energy of the plasma discharge. Further, control of the composition and thickness of films prepared with this system has always been extremely erratic.

The unit which was chosen for this program is the Sloan Sputtergun, a magnetron rf system which confines the plasma

discharge between an anode disc and the target ring which surrounds it. The substrate support lies beneath the ring, completely outside the region of the plasma. This unit was completely untried, having been purchased within a month of the start of the program, and it was in fact operated for the first time there. As a result, a good deal of learning was necessary. The basic form of the system is shown in Figure 2-1.

The sputtergun head was installed on the 4 inch Veeco vacuum station which had been used previously with the dc system. Further the dc supply was wired into the second target in the head so that indium tin oxide (ITO) could be deposited. The purpose of the ITO was to make conductively coated substrates on which the dielectric films could be deposited for dielectric testing and also to form top electrodes in the rare cases where a symmetrical sandwich (ITO-dielectric-ITO) was desired. These coating were also used during the latter stages of the program to fabricate test PROMs.

It was quickly discovered that the extreme weight of the sputtergun head (approximately 100 pounds) was completely beyond the capability of the existing lift whose hydraulic piston was required to support both the dead weight and the cantilevered torque (50 ft. lbs) of the head. For this reason a new lift was assembled (Figure 2-2). In this system the torque is taken up by the 2 inch square steel tube which encloses a 1.5 inch diameter steel rod. The rod provides the major support, but the combination allows the entire head to be rotated away for cleaning, sample installation, and repair. The actual lift mechanism is an air operated piston located between a base plate bolted to the bottom of the square tube and the support arm of the sputtering head. The head rides up and down the square tube on three steel rollers which are bolted to the support arm structure.

The air operated piston is somewhat rougher in operation than a standard water (or oil) hydraulic piston due to the bounce resulting from the compressible gas. The choice was

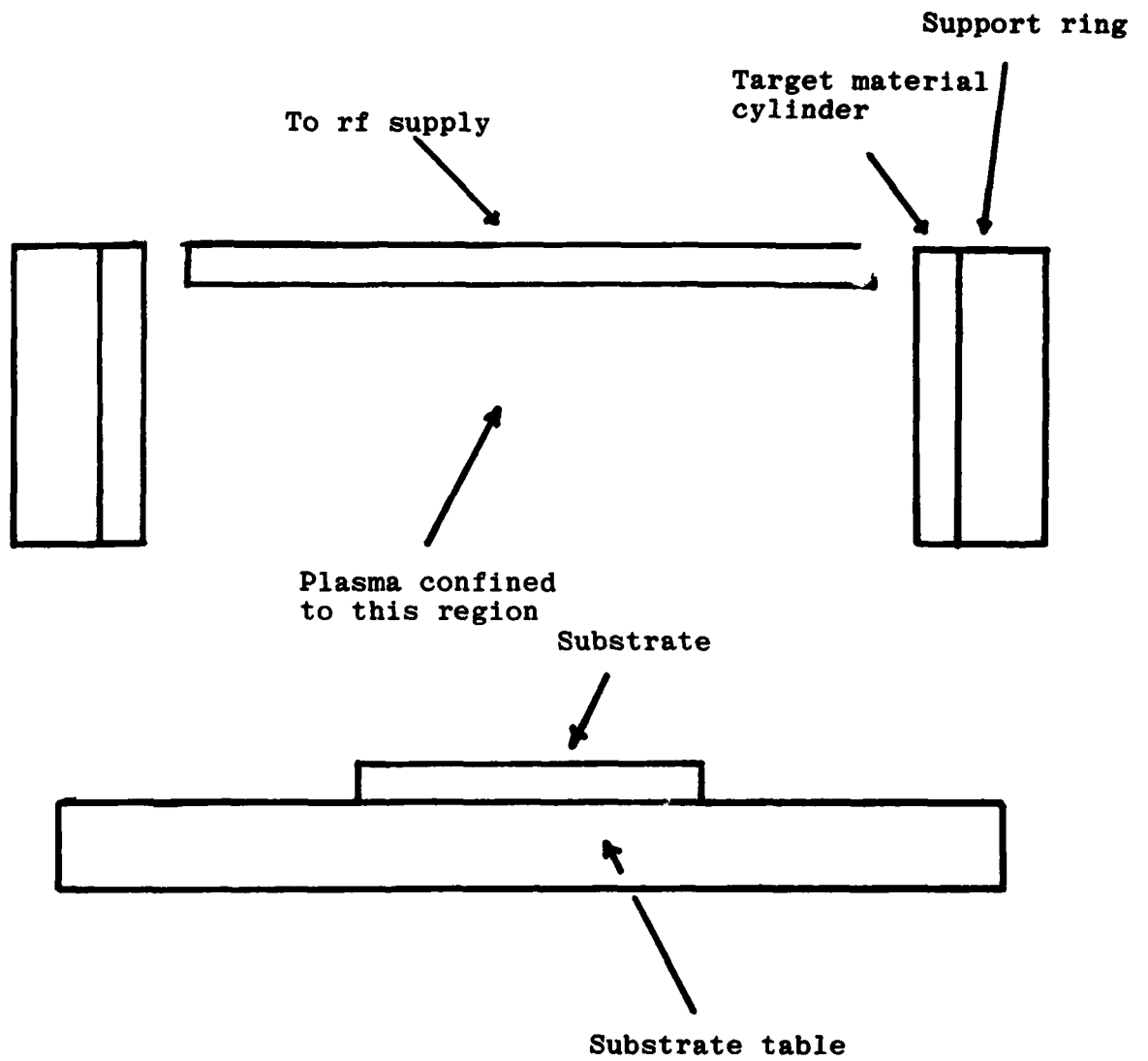


Figure 2-1 - Sloan magnetron cylindrical sputtering target geometry

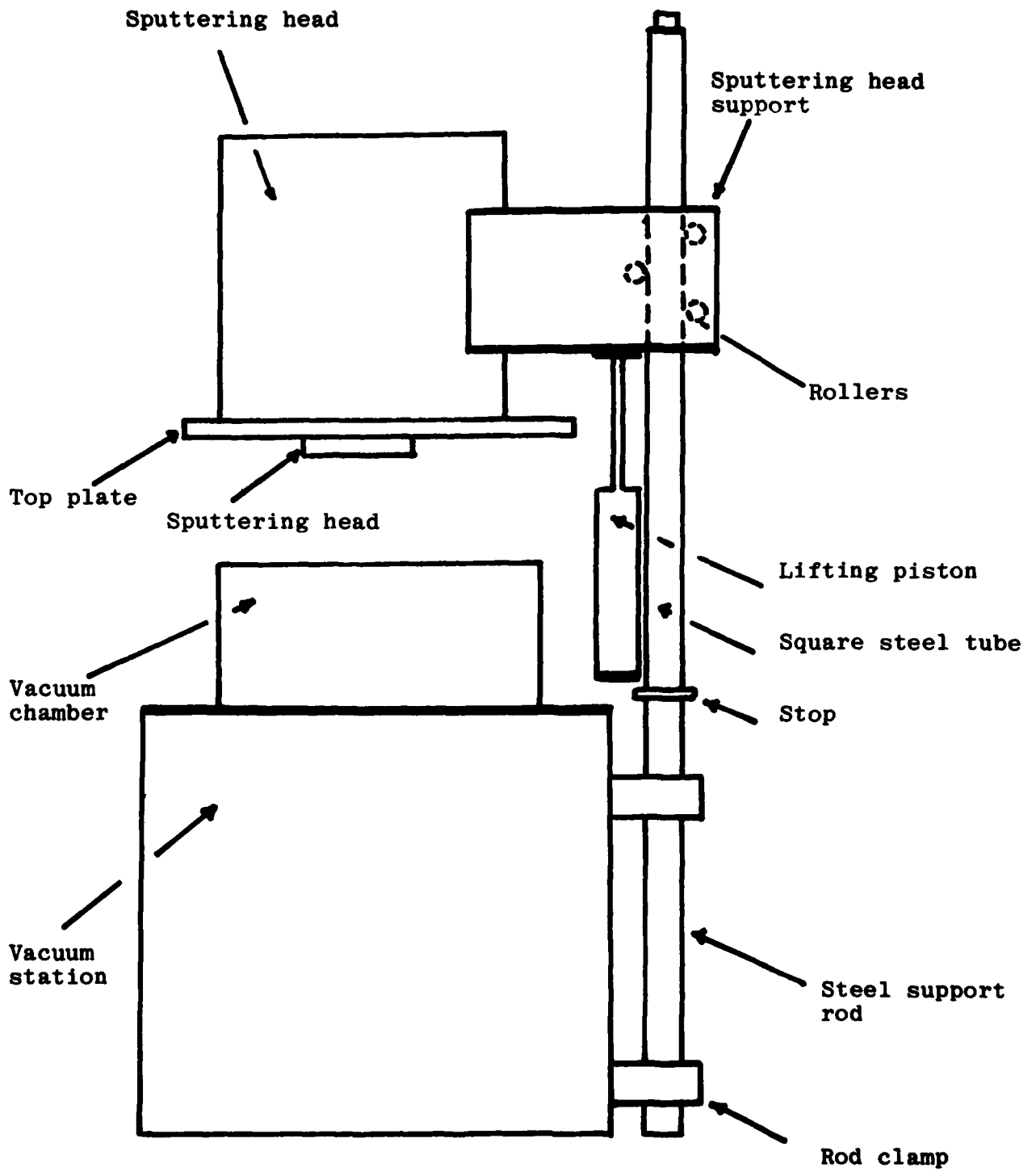


Figure 2-2 - Diagram of lift arrangement for sputtering head

made based on the small area available between the vertical tube and the wall of the deposition chamber. A piston with a sufficiently small diameter to fit in the available space requires approximately 125 psi to lift the weight of the head. Available water pressure was only 80 psi and given the choice between a booster pump and an air bottle it was decided that the slight roughness encountered when operating the lift with air pressure was acceptable

2.2 TARGETS

There are two approaches to the selection of targets for sputtering. The first is to use targets composed of the oxide to be sputtered and to operate under conditions where the small amount of oxygen lost due to evaporative disassociation is replaced by the oxygen in the sputtering atmosphere. The second is to use a metallic target and to sputter slowly in a heavily oxygenated atmosphere so that the metal oxidizes during deposition. There are advantages and disadvantages to each approach. Oxide targets are much harder to make, being sections cut from drawn tubes or sintered ceramics. Some of the more abstruse materials cannot be fabricated in that form at all due to the high firing temperatures required for preparation. Further, due to the processing required these targets are less pure than the metallic targets. 5N pure SiO_2 and 4N pure Ta_2O_5 are available, whereas Si and Ta are available in 6N purity. On the other hand, our experience with evaporated films supports a thesis that high levels of oxidation (or reoxidation) can be very difficult to achieve. Further the high percentages of oxygen required if metal targets are used produces large amounts of heat near the substrate. Hence, the advantage of the "cold" magnetron sputtering system is lost. It was decided that during this phase of the program, oxide targets would be used exclusively since reoxidation was believed to be a major problem.

3. EXPERIMENTAL RESULTS AND CONCLUSIONS

3.1 SPUTTERED INDIUM TIN OXIDE

The first task undertaken was the development of an ITO coating capability using the magnetron. This was necessary since the original MRC system had been stripped to make way for the magnetron head, and an ITO coating capability is basic to the preparation of samples for electrical testing. Initial runs were made using the same voltage settings as had been used with the MRC system (2.3 kv dc). Gas pressures were 4.8×10^{-3} torr Ar and 2×10^{-4} Torr O_2 . Both the argon and oxygen were ultra high purity gases (5 N+) and the argon was further treated by passage through a MRC gas purification filter. The source/substrate distance (all measurements are referred to the bottom of the source ring) was 2 inches.

Several features were immediately apparent. First, the coatings were blue grey indicating the presence of small quantities of InO. This was corrected on later runs by adjusting the gas pressures to 4×10^{-3} Torr Ar and 1×10^{-3} Torr O_2 . Also, the deposition rate was extremely slow, 25 A/minute, approximately one-fourth of that observed with the MRC head. Further investigation disclosed that the plasma discharge covered only the top third of the target ring. It was originally believed that this was due to a problem in the system, but discussions with the manufacturer yielded the information that this is normal in dc operation. The only disadvantage is that it causes rapid erosion of the top half of the target ring sharply shortening target lifetime. At the manufacturer's suggestion we investigated rf

sputtering of the ITO. The extremely small amount of work in this area indicated we could expect a further factor of two reduction in deposition rate, but no change was noted when a rf input power of 300 watts was used. Presumably this is a result of involving the entire target surface in the sputtering process. The extraction rate per unit area is lower, but the target area involved is two to three times larger than with dc operation.

In an effort to increase the deposition rate an adjustable height substrate table was assembled so that the source-substrate distance could be adjusted. This consisted of a rack and pinion arrangement mounted on the feedthrough collar and adjusted by means of an unused rotary feedthrough. The rigid water lines to the substrate table were replaced by standard flexible vacuum tight water lines. Reducing the source-substrate distance to 1-inch with the arrangement increased the deposition rate sharply, but the substrate surface temperature also increased rapidly. At a 2 - 3 inch spacing the substrate surface remained at 50 - 70°C when the table was cooled with normal tap water (10 - 15°C), but at 1 inch temperatures above 150°C were recorded. As the resultant temperature gradient (140°C through the substrate) was considered excessive it was decided to accept the comparatively slow deposition rate obtained at a 2 inch spacing.

The other effect noted on these coatings was a change in electrical conductivity. The coatings from the MRC system typically have a resistivity of 10^{-2} ohm-cm while those from the magnetron have a resistivity consistently 5 times greater. The reason for this is uncertain. There is a slight difference in target composition: the MRC target is 9 percent tin oxide while the magnetron target is 7 percent tin oxide, but similar composition changes in evaporated coatings showed an insignificant resistivity change of 5 - 15 percent. However, due to the different deposition process involved here the film composition

may have a much larger variation. No attempt was made to determine the exact composition of the sputtered film. As with all other ITO films prepared at Itek, whether by sputtering or evaporation, a vacuum post bake was required to stabilize the conductivity. The sheet resistance of unbaked films increases 10 - 50 times within a few days of preparation, but if the samples are baked between 100 and 300°C for at least 1 hour in the moderate vacuum produced by a mechanical pump (10^{-2} Torr), the resistance stabilizes within a factor of two.

3.2 SiO₂ COATINGS ON GLASS AND ITO

At the completion of the ITO work, an effort was begun on SiO₂ deposition. Coatings were made at various source/substrate spacings and power levels using a gas mixture of 4.8×10^{-3} Torr argon and 2×10^{-4} Torr oxygen. The usable power was found to be rather sharply limited since at rf power levels above 700 watts the target can crack. As a result an operating power level of 500 watts was used for all subsequent depositions. In this range the deposition rate ranged from 170 Å/min. at a source/substrate spacing of 1 inch to 45 Å/min. at 4 inches. The rate is very nearly proportional to the inverse of the distance. There is also a strong variation of coating thickness as a function of axial distance from the center of the target. At 1 inch the coating was extremely nonuniform and even at 2 inches a 5 to 10 percent thickness variation was observed over a 1 inch diameter. This was very disappointing since the target geometry had indicated that coatings would probably be uniform over a 2 inch diameter. The implication of this result is that any attempt to deposit highly uniform coatings using this system will require planetary fixturing or at least substrate rotation of some sort.

The initial SiO₂ coatings made in this work all exhibited a faint red-brown tinge typical of the presence of SiO, again indicating that the sputtered material was dissociating and not reoxidizing completely. As a result the gas mixture was changed

to 4×10^{-3} Torr argon and 1×10^{-3} Torr oxygen. On the manufacturer's recommendation the total gas pressure was held at 5×10^{-3} torr throughout the study. With this mixture and a source/substrate spacing of 2 inches a 2 μm thick coating could be deposited in 4 hours with a substrate surface temperature only slightly higher than 100°C . These coatings appeared to be clear, as did coatings deposited at other spacings. It must be recognized that the term "clear" has a limited connotation. The Cary spectrophotometer has a transmission resolution of approximately 1 percent. Samples of SiO_2 on ITO coated slides were unreadable due to the periodic variation of transmission with wavelength caused by interference in the films. However, this could be eliminated by the use of bare SiO_2 substrates having the same refractive index. One can make transmission measurements on these samples, correct for refractive index and show that absorption must be less than 1 percent. However, for a 2 μm thick coating, 1 percent absorption corresponds to an absorption coefficient of 50.

The only other measurement attempted was dielectric breakdown. This was made using the test equipment described in the Phase I report. The improvement over the evaporated SiO_2 films described in Phase I was quite dramatic. Whereas, the breakdown fields of the evaporated coatings was never greater than 1×10^6 V/cm and was frequently as low as 1×10^5 V/cm, the breakdown field of the clear sputtered coatings was consistently above 5×10^6 V/cm. Thus, the typical 2 μm coatings would support 1000 V. This is extremely significant for coatings of potential interest for the Itek PROM. A recent theoretical calculation[†] indicates that the limiting value for the dielectric strength of SiO_2 should be in the range of $7-8 \times 10^6$ V/cm which compares favorably with the results obtained in this study.

Detailed C-V and I-V measurements were not attempted here. Samples were sent to USC for further investigation, but as mentioned earlier this data is not yet available.

[†]D.K. Ferry, J. Appl. Phys 50, 1422 (1979).

3.3 SiO₂ COATINGS ON Bi₁₂SiO₂₀ (BiSOx)

Having achieved good dielectric properties with SiO₂ the effort now turned to the problem of deposition on BiSOx. Initial coatings were made using the same parameters as with the ITO and SiO₂ substrates. It was found that while one surface of the crystal could be coated readily, attempts to coat the second surface invariably resulted in crazing of the first coating. Since the crazing did no damage to the crystal surface it was suggested that the poor adhesion was due to a layer of absorbed water on the crystal surface. (The SiO₂ coating fabricated during Phase I which adhered was deposited at 350°C and the crystal was exposed to the plasma. Both these effects tend to clean the surface, in fact they produced pitting.) Hence, the water cooled substrate table was replaced with a substrate heater which used the heating element from a diffusion pump and a variac as the thermal source. Substrate temperatures of 250°C were obtained but the power could not be applied during the evaporation since the heating coils caused the rf to short to the heater. Hence, coatings were made by heating the substrate to 250°C to drive off surface contaminants, then cooling to approximately 175°C and disconnecting the heater power before starting to sputter. Without the water cooled table the rf plasma provided sufficient energy to maintain the substrate temperature at 175°C during the run. Adhesion was improved somewhat but first side crazing during the second side coating process continued.

The solution was discovered as the result of an aborted run in which the heater coil failed before sputtering was started. In that case the first surface coating remained intact. It was now clear that the thermal gradient through the BiSOx crystal was the problem. When the first side was coated the first surface becomes convex due to thermal expansion of the hotter first (top) surface. On cooling the crystal relaxes to flat, but the coating now has a built in strain level. The crystal can be heated isothermally without damage to the coating, but as soon as a

gradient is applied so that the second surface is hotter, the first surface becomes concave and the additional stress cracks the first surface coating. This problem was corrected by placing the crystal on an alumina plate supported by an alumina tube. All heaters, water cooled substrate tables, etc. were eliminated. The alumina ceramic is not optimum since a better thermal insulator would be preferable but this proved adequate for the time. Using this arrangement coatings were prepared which showed only slight crazing near the edges of the BiSOx wafer. When the source/substrate distance was increased to 3 inches, the coatings on the flat surfaces were uncrazed although insignificant chipping of the coating on the ground, beveled edges remained. A semiquantitative scotch tape adhesion test indicated that the bonding of the second surface was still almost double that of the first surface.

3.4 SPUTTERED Ta_2O_5 ON GLASS AND ITO

Only a small amount of work was performed on sputtered Ta_2O_5 . Based on the experience with SiO_2 the initial coating operation was carried out at 1×10^{-3} Torr of oxygen. A 3 inch source/substrate spacing was used as was the insulating ceramic disc. The rf power was 300 watts. The resulting coating was transparent and unlike the evaporated coatings required no post baking. However, the deposition rate was extremely slow (less than 20 A/minute). In order to obtain a reasonable deposition rate the source/substrate spacing was reduced to 2 inches and the power increased to 700 watts. The result was a transparent coating deposited at 80 A/minute which adhered well to a glass substrate but which partially delaminated from the BiSOx substrate. The delamination is presumably caused by the severe heating of the substrate surfaces due to the high rf power level and short distance used. The dielectric strength of the coating was somewhat low, $\approx 10^6$ V/cm. It is not known at this time whether there is any deleterious effect due to the high (100 ppm) impurity level in the target or whether oxidation may be incomplete. The breakdown test gave qualitative indication of abnormally high conductivity.

3.5 SiO₂ PROM TEST

At the end of the technical effort it was decided to proceed with the fabrication and testing of a SiO₂ coated PROM (Pockels Readout Optical Modulator) even though the coating characterization was incomplete. The PROM consists of a single crystal wafer of bismuth silicon oxide coated on each side with a dielectric layer and a transparent conducting electrode. The PROM is a real-time optical storage device that can be illuminated with blue light from an image and will then store the image as a two dimensional charge distribution. The image can then be read out in coherent red light by means of the Pockels effect in the crystal. A complete description of the device can be found elsewhere.[†]

The device was fabricated entirely in the sputtering system, a significant improvement over normal fabrication procedures which require three systems: vapor deposition (parylene), electron beam evaporation (SiO₂ passivation) and sputtering (ITO electrodes). The entire process required only four pumpdowns and installation of a substrate translator would reduce this to two. In any case "operator present" time was less than half of that normally required. The BiSOx crystal was coated completely on one side with 2 μm of SiO₂ followed by 0.1 μm of ITO, then was turned over and the coating repeated. Deposition conditions duplicated those developed earlier as shown in Table 3-1.

As with the earlier test specimens there was some edge chipping but no surface crazing. The additional ITO coating had no apparent effect on adhesion. The device was packaged in the normal fashion and placed in the standard PROM test set-up. Image contrast was observed to be uniformly low at all voltages. The image was, however, sharp indicating that there was no lateral conduction. However, apparently, the conductivity of the SiO₂ coatings was high enough that some voltage remained on the BiSOx crystal after exposure. No breakdown, sparking or visible damage to the coatings was observed. The contrast was simply low.

[†]D.S. Oliver et al., Appl. Phys. Lett. 17, 416 (1970).

Table 3-1 - Conditions for SiO₂ PROM Dielectric Fabrication

	SiO ₂	ITO
Argon pressure	4x10 ⁻³ Torr	4x10 ⁻³ Torr
Oxygen pressure	1x10 ⁻³ Torr	1x10 ⁻³ Torr
Source/substrate spacing	3-inch	3-inch
Deposition rate	80 A/min	25 A/min
Deposition time	4-hours	40-minutes
rf power	500 watts	300 watts
Reflected power	<5 watts	<5 watts

Without the detailed data from the C-V and I-V studies to be conducted by USC it is impossible to determine the exact source of the conductivity. One can speculate that it is a stoichiometric problem and perhaps the SiO_2 is incompletely oxidized or perhaps impurity conduction is the problem but without careful measurements it is difficult to determine exactly how to proceed. It is anticipated that this data will be available shortly as the USC program gets into full operation.

3.6 Ta_2O_5 COATING ON BiSOx

Due to time constraints only one attempt was made to coat Ta_2O_5 on BiSOx . A poor optical quality test wafer was placed in the system during the final test run on ITO described in Section 3.4. This coating partially crazed, presumably due to the high temperature developed by the 700 W rf sputtering level. It is significant that the portion that crazed was that section closest to the center of the target and hence hottest. Since the result of the breakdown test of the film on ITO showed a low breakdown field, no attempt was made to fabricate a PROM from this wafer since the background birefringence would have made it extremely difficult to analyze the test target exposure data.

4. SUGGESTIONS FOR FUTURE WORK

This work has shown that there is promise in the sputtered thin films for high dielectric strength coatings if the remaining conductivity problem can be solved. Further, Dr. Tanguay at USC is now nearly fully operational on his part of the program so the cooperative effort originally envisioned should begin to bear fruit. The work to be pursued is as follows:

1. Complete work on SiO_2 and Ta_2O_5 .
2. Investigate Y_2O_3 as an alternate material with a dielectric constant midway between SiO_2 and Ta_2O_5 .
3. Depending on the results of the USC conductivity investigation determine whether to study deposition methods using pure metal targets.
4. Modify the sputtering system to; (a) permit gas introduction directly in the target area to achieve improved mixing and (b) to accommodate a thin film optical monitor.
5. Study sputtering techniques for depositing $\text{SiO}_2/\text{Ta}_2\text{O}_5$ or $\text{SiO}_2/\text{Y}_2\text{O}_3$ multilayers on BiSOx .