JOURNAL OF MATERIALS SCIENCE 7 (1972) 257-264

Sputtered Zinc Sulphide Films on Silicon

T. G. R. RAWLINS, R. J. WOODWARD
Signals Research and Development Establishment, Christchurch, Hampshire, UK

The design of a triode rf sputtering system for the deposit of high purity films of zinc sulphide is outlined. It is shown that a variety of dopants may be incorporated in the film over wide concentration ranges by means of this technique, and it is also shown that rare earth cathodoluminescent films may be formed. When deposited under suitable conditions a moderate degree of crystallographic orientation of the films has been achieved.

1. Introduction

Although vapour transport processes have been used in the preparation of single crystal films of II-VI semiconductor compounds on group IV semiconductors [1-2], much of the more current work has involved essentially physical deposition methods. Most physical methods have used vacuum evaporation [3-5], however a few have used sputtering, in some cases reactive [6-8] and in other instances by sputtering the compound in an inert atmosphere [9-11].

In the early stages of work on the preparation of Si-ZnS heterojunctions, it was felt likely that the stoichiometry of evaporated films would vary significantly. It is the experience of the authors (see also [12] and [13]) that provided the vapour pressures of the constituents of a compound are not grossly dissimilar significant changes in stoichiometry do not occur in sputtered films; those that do can usually be allowed for by changes in target composition.

Various instances of the preparation of high purity compounds and alloys by means of sputtering have been cited in the literature, amonst them for example superconducting alloys [14] and of more immediate relevance the work on Cadmium semiconductor compounds [9-11] which has demonstrated the feasibility of controlling stoichiometry and conductivity whilst maintaining purity. In addition however there seems to be an excellent prospect of adding dopant atoms to the deposited films both by incorporation in the target and by addition to the sputtering gas, this latter possibility is not necessarily by reactive sputtering only as has been shown by Winters and Kay [15]. With these factors in mind therefore ZnS layers were deposited on silicon by sputtering from pure and doped targets of the compound, rather than by reactive sputtering in argon and hydrogen sulphide in which stoichiometry is perhaps less easily controlled.

The objective of the work described in this paper was to produce high purity films of ZnS both doped and undoped, on silicon. This work formed part of a programme directed towards the development of a solid state image converter for infra-red to visible conversion by means of a heterojunction between group IV and group II-VI semiconductors. Though the implication of an efficient heterojunction is that the structure should be epitaxial, less emphasis was placed on this aspect here for epitaxy had already been achieved by an evaporation technique [5].

2. Experimental Procedure

2.1. System Considerations

It is not the purpose of this paper to give detailed discussion of the mechanisms of sputtering or the techniques used to sputter semi-insulators such as ZnS, these are adequately described in the literature e.g. the review articles [12] and [13]. It suffices to say that when one wants to sputter material from a target of very high resistivity, it is necessary to prevent the accumulation of charge on the surface of the target material. Build up of such charge will raise the surface to such a potential that ions are prevented from striking the target and removing material. The particular technique used in this work has been that of radio frequency sputtering.

In general rf sputtering systems may take two forms either diode or triode, in the former, where the rf voltage is applied between target (cathode) and substrate (anode) not only is the sputtering current controlled by the rf voltage but also the plasma discharge is initiated and controlled by the same source. In a triode system however the plasma discharge is initiated and controlled quite separately from the rf voltage which is only applied to the target material. The diode system inevitably suffers from lack of flexibility for the density of ions available for bombarding the target cannot be controlled separately, and the sputtering rate is usually rather low for a given input power compared with the triode system. It is normally possible to operate all power supplies in a triode system at voltages very much lower than the diode system, this assists very considerably in reducing the amount of spurious sputtering (and hence contamination of the deposit) due to a background of high energy ions. The main disadvantage of a triode system is supposed to be the possible contamination of deposited films by material evaporated and/or sputtered from the filament (which supplies the electrons used to create the plasma). Undoubtedly there is a possibility of such contamination, but careful design of the system should reduce this to a negligible level as shown by mass spectrographic analysis of the deposited films (see below). The system actually used in this work was of the triode form.

It is evident that there are two principal aspects of the experimental system which affect the purity of the deposit namely the starting material (the target and substrate), and the environment in which the deposit has been carried out, an attempt was made to optimise both of these aspects.

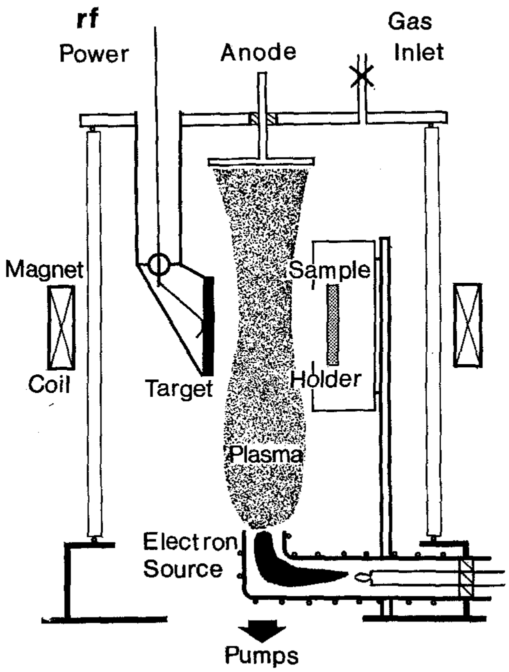

Figure 1 Schematic drawing of the sputtering chamber.

2.2. System Environment

The basic vacuum system used was a high pumping speed oil diffusion pump system incorporating thermoelectrically cooled chevron baffles, figs. 1 and 2 show the experimental system in schematic outline (for clarity the sample heater supply is omitted). The general approach to the design of the system has been to reduce the number of objects and the surface area in the active volume to the absolute minimum and to ensure the greatest possible degree of cleanliness. To achieve these aims all parts in the system were made of low sputtering yield material, stainless steel was used rather than aluminium as a more suitable material to tolerate the expected high temperatures. In fact it was not possible to make the electron source out of stainless steel, and copper with a thick chromium plating was used; the filament was made out of tungsten wire. The principal vacuum enclosure was a pyrex glass cylinder. In addition to the normal gauging a mass spectrometer was incorporated, this was not run during sputtering for it was felt that the necessary differential pumping would introduce sampling errors which would ensure that there was little relation to the spectrum in the vacuum chamber. Quite apart from the cleanliness of the surfaces in the system it is clear that the purity of the sputtering gas is an important factor in securing a high purity deposit. In order to try and retain a reasonably high degree of purity and cleanliness throughout, steps were taken to remove harmful impurities from the argon. This was standard argon of 99.99% purity, the primary impurities being oxygen, hydrogen, some nitrogen and water. With this in mind the gas was first passed through a platinum catalyst unit to combine the hydrogen and oxygen to form water. Subsequently it was passed through a molecular sieve to remove all the water vapour. Finally it was passed through a Millipore 1.2 μm filter, to remove any possible molecular sieve particles, via a shut-off valve into the vacuum chamber.

Of prime concern in relation to deposit purity is the manner in which the sample substrate was held. This was mounted in a re-entrant fashion into the stainless steel box so that the substrate surface could only "see" the target and cold portions of the sample holder box, there was no possibility of a direct line-of-sight view of the electron source. The sample was radiantly heated from the rear by a tungsten filament, temperature was not monitored but approximately calibrated from power input under given sputtering conditions.

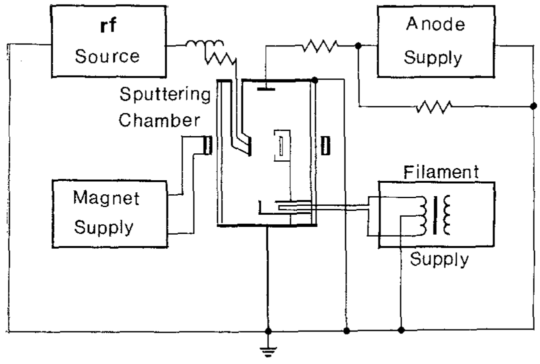

Figure 2 Block diagram of the circuitry of the sputtering system.

In operation it was found that an anode potential of 40 to 50 V DC was quite adequate to give a saturated plasma current of ~ 3A. This was sufficiently low to reduce the possibility of spurious sputtering due to the incidence of high energy ions on other parts of the system. In addition the electron source filament was run at a low AC voltage (with a grounded centre tap) to reduce the possibility of sputtering from it, and the filament temperature was restricted to ~ 1900 K to reduce evaporation. It was found that a magnetic flux density of ~ 25 mT was sufficient to localise the plasma column, this served both to increase the density of ions available for sputtering and to ensure that most of the ions (the visible portion of the plasma) were constrained away from everything in the system but target and sample holder; these were kept separated by about 60 mm at the outer edges of the plasma column. The ultimate pressure with the system hot (not at sputtering pressures) was found to be ~ 1.0 mNm^{-2} (8×10^{-6} torr).

The target assembly comprised essentially of a rigid air-spaced (low capacitance) coaxial cable terminating in a glass-to-metal seal, the other side of which was the stainless steel cage assembly holding the target which was contacted by a phosphor bronze spring. The rf source used to supply the target was a transmitter having an output power of ~ 100 watts with final amplifier voltage of ~ 700 V. The frequency used was 3.75 MHz, it was found convenient to keep it to this low value to reduce resistive effects, and also to ensure that the length of the system ground lead was $\ll \lambda/4$. Admittance measurements on the target assembly, showed that it was intrinsically capacitive as was expected with an extremely small value of conductance, indicating the large effective resistance of the target. The output impedance of the transmitter was variable, ranging from 50 to 600 Ω, but even this range is insufficient to match such disparate loads efficiently. However when the extreme non-linear nature of the plasma is taken into account [16, 17] it seems pointless to try and match the load exactly. In the light of these considerations it was found most suitable to use simply a variable series inductance to match the target (by adding an equal and opposite reactance to that of the target assembly), and in addition to ensure that the transmitter was as close as possible to the target assembly to reduce radiative and other losses to an absolute minimum.

2.3. Target and Substrate

Targets for rf sputtering, particularly of compound semiconductors of this type, usually present something of a problem. Although it has been found possible to sputter from powder, it is generally more satisfactory to sputter from a rigid disc with the highest possible density. Sputtering has been done in this work from sintered discs containing various impurities and from hot pressed discs with and without deliberate impurities. One of the most useful forms of hot pressed target was nominally pure ZnS in the form of 2 in. diameter Irtran—2 discs, with a 1 in. diameter evaporated Cr-Au contact on the rear. No analysis of these discs was undertaken, mass spectrographic studies of deposited films showed that they were of a purity comparable to that of evaporated films [5]. As the density of these discs is ~ 99.7% of bulk ZnS no problem was found in evaporating continuous metal contact films on the back surface. Other hot pressed discs were obtained commercially, these were doped with manganese, manganese and copper, and terbium in the form of $MnCl_2$, $CuSO_4$, TbF_3, and Tb_2O_3 and undoped but were generally only about 80% of bulk density and thus presented some contact problems.

Additional targets were produced in these laboratories from Optran grade ZnS powder which was sintered with the dopant, manganese and copper, or terbium, in the form $MnCl_2$, $CuSO_4$, Tb_2O_3, and $TbNO_3$, and then ground up and resintered to improve homogeneity; these targets were generally about 70% of bulk density and thus also presented contact problems.

The silicon substrates used were either (111) or (100) oriented, chemically/mechanically polished, and comprised both N- and P-type high and low resistivities. Prior to deposit, the substrates were cleaned and lightly etched to ensure that there was a minimum of work damage and surface contamination together with the best possible surface flatness. The procedure used is outlined in appendix A.

3. Results

3.1. Vacuum Environment

In operation (not under sputtering conditions) the mass spectrum of the system was typical of that expected for an air leak, this is perhaps to be expected when considering the large number of hot seals. On occasion traces of peaks due to organics were observed. At no time either before or after a sputtering run was any trace of ZnS or its usual cracking products observed (in decreasing magnitude mass No: 64, 66, 68, 67 with traces of 65 and 70), it is clear that elemental S could not be recognised easily due to coincidence with the O_2^+ peak. Prior to the beginning of a run the system was normally baked to desorb impurities before back filling with argon, it was found that running the system with a relatively high throughput of argon had a beneficial effect on the mass spectrum observed immediately after the run, recently this observation has been independently confirmed and put on a more quantitative footing [18].

3.2. Deposited Layers

Initial setting up runs were done using a glass target. The main factor influencing deposition rate for a given target-sample spacing was anode current although this was not exhaustively examined, it was observed that a reduction of ~ 20% in current reduced the rate by slightly less than half.

When the most dense Irtran-2 targets were used rates up to about 1.2 μm/h were obtained, although it was found advisable to reduce this somewhat (0.5 μm/h) to avoid overheating the target. When the substrate was heated the deposit rates fell with increasing substrate temperature. When less dense targets were used rates were somewhat less and appeared to fall as the density was reduced. Some sixty or so films were deposited both doped and undoped, ranging in thickness from 10 nm to 2 μm. The principal factors affecting thickness/rate of deposit were anode current and the contact to the target (affected by target density), rf matching and target-sample separation were of less importance, the quality of the deposit was most dependent on the density and homogeneity of the target and cleanliness of system and substrate. In general the deposits were extremely uniform in thickness over the 20 mm diameter area of the layer, as shown by the colour of white light interference effects for the thinner layers, and were very clean and smooth in appearance.

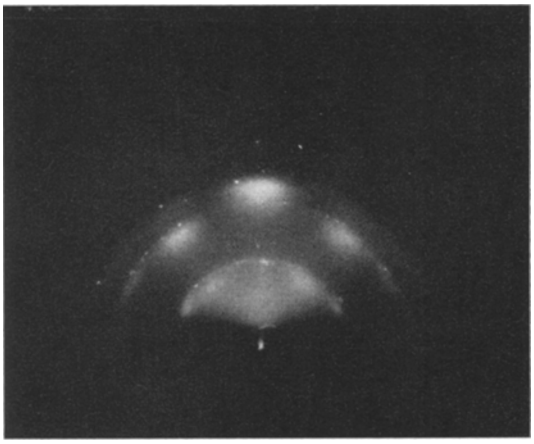

Figure 3 Glancing angle electron diffraction pattern from ZnS layer sputtered on (100) silicon, substrate temperature ~ 200° C, rate 0.15 μm/h.

As was to be expected deposits formed at room temperature (no direct substrate heating) were amorphous. As the substrate temperature is increased there is a noticeable improvement in ordering, fig. 3 shows the glancing angle electron diffraction pattern from an undoped layer deposited on (100) silicon at an estimated substrate temperature of 200° C. In this particular instance the deposit rate was reduced to 0.15 μm/h. As may be seen the deposit is moderately highly oriented but not yet epitaxial. Glancing angle X-ray diffraction was used to examine some of the thicker doped layers and a similar degree of ordering was observed. Unfortunately time did not permit a complete examination of the range of deposit conditions needed to produce epitaxy. There does seem little doubt however, that over the range of conditions examined, the higher the deposit rate – the less the perfection of the deposit. Also for a given deposit rate, the higher the substrate temperature – the better the perfection of the deposit (consistent with vapour pressure considerations [19] allowing any deposit at all at a particular temperature). Examination of deposited layers in the scanning electron microscope shows that the surfaces are extremely smooth; even at the highest magnifications a slight surface roughness is only just perceptible. In general no etching of the silicon is observed either by SEM or optical microscopy, this is in contrast to the layers produced by evaporation [5] in which etching was observed, due presumably to the dissociation of ZnS and subsequent formation of volatile sulphides of silicon. The clear implication is that the sputtered layer is deposited in the form of ZnS and not by combination of zinc and sulphur on the substrate.

Infra-red reflection spectra of the samples were run, and within the accuracy of the measurement, the refractive index of the deposits was the same as bulk ZnS.

3.3. Purity and Luminescence of Layers

The impurity content of the deposited layers was investigated by means of a special mass spectrographic analysis technique, using an MS7 mass spectrometer, by the Fulmer Research Institute. The sensitivity of this technique is somewhat limited on these films because of the high resistivity of the sample as well as the small sample size. Nevertheless the sensitivity seems to be such as to indicate impurity content to levels of some tens of ppm. The most useful presentation of results for undoped films seems to be in a comparison with films deposited by UHV evaporation [5]. Considering initially films produced using the Irtran target it is evident that these are very similar to those produced by sublimation in UHV except that typically there is ~ 30 ppm Al in the sputtered film compared with < 10 ppm in the evaporated film (this is probably due to the Al foil which had to be placed around the cage assembly to prevent the plasma shorting out the rf). There is also 30 ppm Na compared with 10 ppm for the evaporated films – due perhaps to ion bombardment of the glass walls releasing sodium ions, and an apparently higher value for Sr, < 30 ppm compared with < 10 ppm, the source of which is obscure. Otherwise impurities are all less than or equal in level to those in evaporated films. No unusual excess of tungsten (from the filament) was observed in sputtered films, the overall level being < 30 ppm. It is clear from MS7 analysis that layers produced from other nominally undoped commercially fabricated targets are very much less pure, having impurity contents up to 0.1 %. No luminescence was observed from any undoped films. Terbium doped targets produced in these laboratories were analysed by MS7, and in one instance also by emission spectrograph, in this latter case the mass spectrometer showed 2000 ppm of Tb, and emission results gave 1.25 %, the reason for the discrepancy is not clear though it may be associated with target inhomogeneity. Other targets indicated Tb concentrations ranging from 3.2 ppm to 13000 ppm, manganese-copper doped targets showed concentrations ranging from 320 ppm to 60000 ppm manganese combined with 100 to 1300 ppm copper. Commercially prepared targets were not analysed. Table I shows analyses of typical films from some of these targets; it is evident firstly, that the dopant has carried across into the layer, and secondly that in general the actual amount is close to that in the target. There are obvious discrepancies when the layer is very thin (due to the inaccuracy of the analysis with very small volumes), and in the copper concentration determined for Mn-Cu doped layers, it is not clear why this should be so.

TABLE I Layer Impurity Concentrations.

Target	Film thickness	% Mn	% Cu	% Tb
	0.04 μm	—	—	0.065
SRDE	0.1	—	—	1.1
0.5% Tb	0.3	—	—	0.4
	0.6	—	—	0.5
SRDE	0.3	0.5	1.2	—
0.6% Mn	0.6	0.4	0.05	—
0.01% Cu	2.0	0.4	0.33	—

Though it is clear from mass spectrographic analysis of the deposited layers that dopant ions have been successfully introduced, luminescence results show that not all of these ions are in such an environment that they will give luminescent emission. The films were examined by three techniques, cathodoluminescence (at low and high voltages), electroluminescence, and by photoluminescence though this latter technique gave completely negative results. Electroluminescence measurements were complicated by the fact that all layers (both doped and undoped) were of extremely high resistivity, it should be noted however that the dopants introduced were not intended to increase conductivity but to produce luminescent centres. Electroluminescence was observed in only two layers, one, the last sample in table I and the other, the 0.6 μm Tb-doped sample in table I. Although emission was observed it was not possible to characterise it for it was too broad band. It should be noted that though both of these samples contained large concentrations of dopant ions, neither showed any cathodoluminescence. Infact only Tb doped layers showed any cathodoluminescence and only those produced from targets which had been doped originally with Tb in the form of Tb_2O_3 and then only at 20 kV excitation, the emission spectrum of a Tb doped layer is shown in fig. 4 with the corresponding target spectrum and assignment of levels to emission lines. In general with one possible exception (because it was not analysed – this was the only luminescent film produced from commercially prepared targets) this cathodoluminescence was only observed from layers produced by sputtering from very lightly doped targets $\sim$ 3 ppm Tb. No emission was observed from films doped with manganese or manganese-copper despite analysis showing that dopant concentrations from 3 ppm to about 0.5% were obtained in the layers.

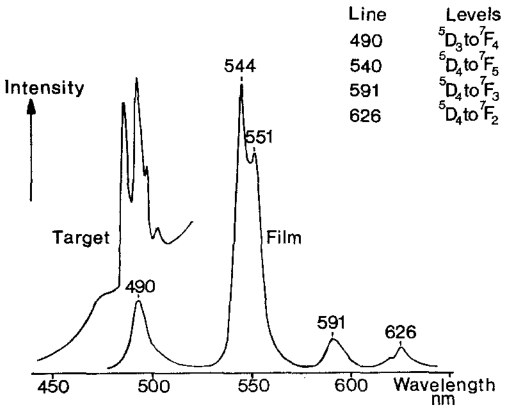

Figure 4 Cathodoluminescence spectrum of Tb doped film at 77 K with corresponding target spectrum and level assignment. (Spectra not to the same scale).

3.4. Electrical Properties of Layer-Substrate

Although no exhaustive examination was made of the electrical properties of the hererojunction formed, contacts were put on some of them (primarily undoped) and I-V characteristics examined for layers on N-type substrates only. Sputtered contacts of both Au and Pt were used on the ZnS and sputtered Au on the silicon. Only slight rectification was observed and usually the characteristics were symmetrical, though a somewhat higher conductivity was obtained with ZnS positive (forward direction), the characteristic being more or less identical to those of single crystal films. They were linear in both directions up to about 0.4 V, suggesting either a 0.4 V barrier for the forward direction or space charge limited injection of electrons from the N-type silicon. The I-V characteristics were not light sensitive. For a more complete study of the electrical properties of these and other Si-ZnS heterojunctions, see [20].

4. Discussion

Results obtained from the sixty or so doped and undoped films that have been deposited, indicate that provided adequate precautions in the design of the apparatus are taken, it is possible to rf sputter ZnS in the compound form with a reasonable degree of purity. Mass spectrographic analysis of the undoped films suggests that the factor which is often cited as the disadvantage of the triode approach – contamination of the deposits by material from the filament, does not necessarily occur. If this is the case triode sputtering presents a more flexible approach than diode sputtering, for separate control of plasma current is an important means of controlling deposit rate. Furthermore it is clear that various dopants may be introduced into the deposit over a range of concentrations whilst not altering the intrinsic purity of the deposit. In terms of purity it appears that the deliberate use of relatively high flow rates of sputtering gas to flush impurities away from the deposition region is a practical alternative to sputtering in an environment which is more akin to that in a UHV system. It is clear however, [15] that a certain amount of gas may be incorporated into the deposited film though no analysis of argon content has been made in this work.

The luminescence results obtained show that though appropriate dopant ions may be introduced into the deposit they are not always in the correct site for luminescence, though it seems likely that this may depend on the actual form of the dopant in the target (which previous sputtering experience indicates is the probable form in which it is carried across to the layer). It is also possible that the structure of the layer may affect occupation of the correct sites. The fact that layers with a high concentration of Tb do not show cathodoluminescence may be a result of concentration quenching. The spectra obtained from these layers are those which are to be expected for the particular dopant.

Although complete epitaxy has not been achieved, it seems extremely likely that a more complete examination of deposit conditions would give a suitable epitaxial process. The structural results obtained so far fall into the general pattern for sputtering epitaxy established by previous authors for both compounds [9] and elemental semiconductors [21, 22], namely that for a given substrate temperature an increase in deposit rate (rate of growth of the deposit) tends to reduce crystal perfection of the layers; also for a given deposit rate an increase in substrate temperature improves the perfection of the deposit (though these factors are also affected by other variables such as ion current density). Thus it may be anticipated that an increase in substrate temperature or decrease in deposit rate – or both, would result in a closer approach to a single crystal epitaxial deposit. It may well also be that an improvement in substrate cleanliness e.g. by reverse sputtering would help to produce single crystal layers, for in the sputtering case reported here no deliberate attempt was made to remove the oxide [5] by sublimation, and in general no etching effects were observed which would otherwise remove it. In one instance only some etching has been observed, the fact that this did occur rather suggests that in that particular case free sulphur was present in the target which resulted in the formation of volatile sulphides.

5. Conclusion

The feasibility of using rf sputtering for producing pure films of ZnS has been shown using a specially designed clean triode sputtering system. In addition it has been demonstrated that a variety of dopant ions may be introduced into sputtered layers, over a wide range of concentrations, in a controlled and reproducible manner using this sputtering system. Films exhibiting rare earth emission under cathodo-excitation have been prepared using this technique. It has also been shown that without particular effort with regard to structural optimisation, films with a moderate degree of orientation can be produced in this system.

6. Acknowledgements

We would like to thank Mr W. G. Dixson BEM for polishing various substrates, Dr W. A. Shand for preparation of targets and other assistance, Dr A. F. J. Cox and Dr J. M. Williams for cathodo- and electroluminescence measurements, and Mr W. S. Nicol for electrical measurements.

References

1. H. J. HOVEL and A. G. MILNES, *Int. J. Electronics* **25** (1968) 201. *J. Electrochem. Soc.* **116** (1969) 843.
2. I. BERTOTI, M. FARKAS-JAHNKE, E. LENDVAY, and T. NEMETH, *J. Mater. Sci.* **4** (1969) 699.
3. J. T. CALOW, P. J. DEASLEY, S. J. T. OWEN, and P. W. WEBB, *Phys. Stat. Sol.* **28** (1968) 295.
4. P. L. JONES, C. N. W. LITTING, D. E. MASON, and V. A. WILLIAMS, *Brit. J. Appl. Phys.* (*J. Phys. D.*) Ser **2** **1** (1968) 283.
5. T. G. R. RAWLINS, *J. Mater. Sci.* **5** (1970) 881.
6. T. K. LAKSHMANAN and J. M. MITCHELL, *Proc. Amer. Vac. Soc. 10th Symp.* (1963) 335.
7. G. BATAILLER, P. BUGNET, J. DESFORGES, and S. DURAND, Compt. Rend. **264** Ser B (1967) 320.
8. J. POMPEI, 2nd Symposium on the deposition of Thin Films by sputtering, Rochester New York, June 6-7, 1967.
9. G. A. ROZGONYI and W. J. POLITO, *Appl. Phys. Letts.* **8** (1966) 220, *J. Vac. Sci. Tech.* **6** (1969) 115.
10. G. BRINCOURT and S. MARTINUZZI, *Le Vide* **131** (1967) 253.
11. M. LICHTENSTEIGER, I. LAGNADO, and H. C. GATOS, *Appl. Phys. Letts.* **15** (1969) 4, 18.
12. L. I. MAISSEL, in "Physics of Thin Films, Advances in Research and Development" **3** Ed. G. Hass and R. E. Thun (Academic Press, London, 1966) 61.
13. J. L. VOSSEN and J. J. O'NEILL, JNR. *RCA Review* **29** (1968) 149.
14. J. M. SEEMAN, *Trans AVS Vacuum Symposium* New York (1965) 269.
15. H. F. WINTERS and E. KAY, *J. Appl. Phys.* **38** (1967) 3928.
16. H. S. BUTLER and G. S. KINO, *Phys. Fluids* **6** (1963) 1346.
17. M. P. BACHYNSKI, *RCA Review* **28** (1967) 111.
18. G. A. SHIRN and W. L. PATTERSON, *J. Vac. Sci. Tech.* **7** (1970) 453.
19. P. GOLDFINGER and M. JEUNEHOMME, *Trans. Faraday Soc.* **59** (1963) 2851.
20. W. S. NICOL, to be published.
21. E. KRIKORIAN and R. J. SNEED, *J. Appl. Phys.* **37** (1966) 3665.

22. C. K. LAYTON and K. B. CROSS, *Thin Solid Films* **1** (1967) 169.

Received 16 August and accepted 8 October 1971.

Appendix

SUBSTRATE CLEANING PROCEDURE

1 Dip in dilute nitric acid
2 Rinse in deionised water
3 Ultra-sonic rinse in hot deionised water-detergent solution
4 Two stage ultra-sonic rinse in deionised water
5 Ultra-sonic rinse in electronic grade isopropanol
6 Two stage cleaning in vapour bath of electronic grade isopropanol
7 A 30 to 60 sec etch with etchant agitated in either:

(a) 5:3:3 parts by volume of hydrofluoric acid, nitric acid, acetic acid at 20°C
OR
(b) If the substrate can be heated prior to deposition in the vacuum chamber to a sufficient extent that an iodine layer can be driven off, 1:5:4.4 parts by volume of hydrofluoric acid, nitric acid, acetic acid, the whole then saturated with iodine and used at 40°C.

8 The etch is then quenched by flooding with deionised water and the etch thoroughly rinsed away
9 The substrate may then be placed straight in the vacuum system (wet) if the water can be pumped away, or is rinsed in boiling electronic grade methanol and then placed in the vacuum system.