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THE USE OF METALORGANICS IN THE PREPARATION OF SEMICONDUCTO MATERIALS: GROWTH ON INSULATING SUBSTRATES

H. M. MANASEVIT

Autonetics Division of North American Rockwell Corporation
3370 Miraloma Avenue, Anaheim, California 92803, U.S.A.

Semiconductor films of II–VI and III–V compounds and alloys have been produced on semiconductors and insulators by decomposing appropriate Group II and Group III metalorganic compounds in the presence of Group V hydrides and Group VI hydrides or alkyl compounds. Group II metalorganics are also found ~~to be~~ suitable dopant sources for the III–V compounds. The halide-free growth process is compatible with existing CVD systems for the formation of elemental semiconductors.

1. Introduction

Recently, a process has been developed which appears to overcome the temperature restrictions, flow requirements etc. of CVD[1–3]) for the epitaxial growth of compound semiconductors. This process requires only one controlled hot temperature zone for the *in situ* formation and growth of the semiconductor compound on the heated substrate and occurs in an atmosphere probably free of etching species.

In this process semiconductor films of II–VI and III–V compounds and alloys are produced by decomposing appropriate Group II and Group III metalorganic compounds in the presence of the appropriate Group V and Group VI hydrides or alkyls. In addition, the Group II metalorganics and Group VI hydrides are sources for the introduction of dopant impurities.

Earlier published reports in which the metalorganic–hydride process for compound semiconductor formation was described were made by Didchenko et al. in 1960[4]) and Harrison and Tompkins in 1962[5]). In both cases the experiments were performed under relatively low pressure conditions in closed systems. Didchenko reported the preparation of InP by first reacting trimethylindium (TMI) and PH_3 at −125 °C and then decomposing the reaction product at about 275–300 °C into a dark gray powder identified as InP.

Harrison and Tompkins[5]) described experiments leading to the production of high-resistivity crystalline films of InSb at ~160 °C from TMI and SbH_3. They also believed they had formed GaAs from trimethylgallium (TMG) and AsH_3 by mixing them at room temperature in 1:1 molecular proportions in the vapor state for 18 hours and then heating the mixture to about 200 °C. The film they obtained displayed high resistivity, but unfortunately was not characterized further so as to be definitely identified as GaAs.

In 1968, Manasevit and coworkers[6,7]) described the use of metalorganics and hydrides in CVD for the preparation of thin films of single-crystal semiconductor compounds and alloys. They used triethylgallium (TEG) or trimethylgallium (TMG) and Group V hydrides in producing GaAs, GaP, $GaAs_{1-x}P_x$, and $GaAs_{1-x}Sb_x$ on semiconductors and insulators such as sapphire (Al_2O_3), spinel ($MgAl_2O_4$), and beryllia (BeO).

Later, Thomas [8]) decomposed TEG and triethylphosphine (TEP) and grew layers of GaP on S at 485 °C. Rai-Choudhury confirmed Manasevit et al. by using the TMG–AsH_3 process to grow GaAs on G As and Al_2O_3[9]), and found the electrical properties of the films comparable on both substrates. The early stages of growth[10]) and properties[11]) of GaAs on Al_2O_3 were studied. Manasevit et al.[13]) also studied the metal-organic–hydride process for the formation of epitaxial III–V aluminum compounds. AlAs and $Ga_{1-x}A$ As were prepared on GaAs and (0001) Al_2O_3 using trimethylaluminum (TMA) as the source of Al and T G as the Ga source.

In 1970, Minden[13]) reported some optical properties of AlAs films grown on GaAs by the TMA–A H_3 process. Also, Manasevit et al. expanded their studies to include the II–VI compounds[14]) and demonstrated

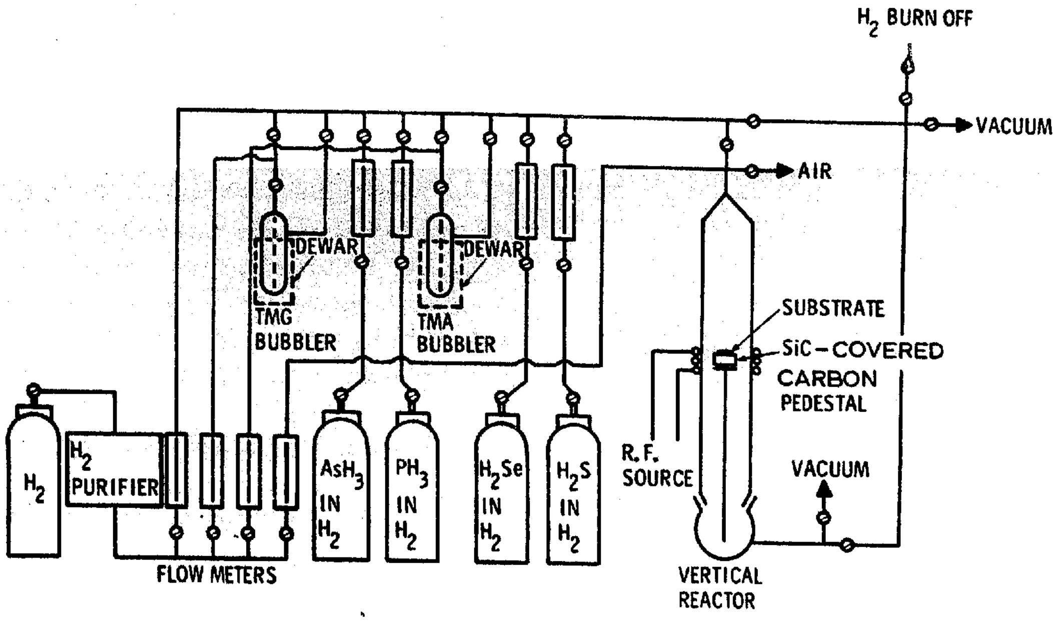


Fig. 1. Schematic of deposition apparatus.

the feasibility of using metalorganics in the preparation of the sulfides, selenides, and tellurides of Zn and Cd, on insulating substrates. The same Group II metalorganics were also found to be effective dopants in the preparation of p-type GaAs in these halide-free systems[15]). Lindeke et al.[16]) used gallium diethylchloride and AsH_3 to prepare homoepitaxial layers of GaAs. It was not ascertained if the process could be used to grow epitaxial films on insulating substrates, but it seemed to provide epitaxial films at much lower temper tures than other reported halide processes.

The studies in our laboratories have since been expar led to include single-crystal films of the nitrides of Al id Ga on Al_2O_3 and other substrates[17]).

e results of the studies of compound semiconductoi rowth using CVD processes involving metalorganics e reviewed in the following sections.

2. perimental

: rather simple type of apparatus used in these stu s is shown schematically in fig. 1. It is typical of th used in studying the growth of elemental semico ictors. It consists principally of a single vertical 60 n O.D. quartz tube 38 cm long containing a SiC-co ed carbon pedestal which can be inductively heated d rotated; stainless-steel bubblers for containing the iquid metalorganics and SbH_3; appropriate flow meters for monitoring the carrier gas, Group V hydride(s), and dopant flows; a H_2 burn-off area; and a manifold made from ¼-inch stainless-steel tubing. Provisions are made for bypassing the quartz reactor and keeping the reactants separate until the gases are equilibrated and ready to be mixed for film formation.

The metalorganics are introduced into the reactor by bubbling a carrier gas through them and are mixed with the appropriate hydrides – such as AsH_3, PH_3, AsH_3–PH_3, AsH_3-SbH_3, NH_3, H_2S, and H_2Se – controlled independently by simple flow meter adjustments.

The structural nature of the deposits is determined by X-ray diffraction, reflection electron diffraction (RED), and/or electron microscopy techniques. The presence and relative compositions of As, P, and Sb in the films and the compositions of the alloys are determined using an electron microprobe.

The electrical properties of the films are evaluated from measurements of the Hall effect made on specially shaped samples etched in the films by standard photolithographic techniques. The carrier concentrations are deduced from the Hall coefficient R_H according to $n = 1/eR_H$, and the Hall mobility is determined from the product $\mu_H = \sigma R_H$, where σ is the electrical conductivity.

3. Studies of GaAs epitaxy on insulators

Both TEG (b.p. = 143 °C at 760 mm Hg) and TMG (b.p. = 55.6 °C at 760 mm Hg) were successfully used in the preparation of GaAs. Because of its greater volatility, most studies were made using TMG. Growth rates of GaAs of up to 1.5–2 μm/min were easily achieved using TMG. Epitaxial growth has been achieved at 650–750 °C on a number of insulating substrates of various orientations. These are compiled in table 1[18]).

TABLE 1

Orientation relationships between GaAs and single crystal oxides

Substrate	Parallel relationschips
(0001) Al_2O_3	(111) GaAs $\parallel$ (0001) Al_2O_3
$(11\bar{2}3)$ Al_2O_3	(111) GaAs $\parallel$ $(11\bar{2}3)$ Al_2O_3
$(11\bar{2}5)$ Al_2O_3	(111) GaAs $\parallel$ $(11\bar{2}5)$ Al_2O_3
$(11\bar{2}6)$ Al_2O_3	(111) GaAs $\parallel$ $(11\bar{2}6)$ Al_2O_3
$(01\bar{1}2)$ Al_2O_3	(111) GaAs $\parallel$ $(01\bar{1}2)$ Al_2O_3
(100) $MgAl_2O_4$	(100) GaAs $\parallel$ (100) $MgAl_2O_4$
(110) $MgAl_2O_4$	(100) GaAs $\parallel$ (110) $MgAl_2O_4$
(111) $MgAl_2O_4$	(111) GaAs $\parallel$ (111) $MgAl_2O_4$
$(10\bar{1}0)$ BeO	(100) GaAs $\parallel$ $(10\bar{1}0)$ BeO
$(10\bar{1}1)$ BeO	(111) GaAs $\parallel$ $(10\bar{1}1)$ BeO
(0001) BeO	(111) GaAs $\parallel$ (0001) BeO
(100) ThO_2	(100) GaAs $\parallel$ (100) ThO_2

A rather unexpected result was the observed growth of (100) GaAs on both Czochralski (110) $MgAl_2O_4$ and (100) $MgAl_2O_4$[7]). Epitaxy on (100) $MgAl_2O_4$ was found to be very surface-sensitive and not always reproducible; on (110) $MgAl_2O_4$ epitaxy was reproducible, but the electrical properties of the films were sometimes inconsistent. Further work is needed to explain the unusual crystallography. High quality reproducible films were produced on (111) $MgAl_2O_4$.

GaAs films on (0001) Al_2O_3 were found to possess a much lower degree of residual stress than that found for Si on Al_2O_3 or $MgAl_2O_4$. This is not unexpected since the mean linear thermal expansion coefficients for GaAs (6.4×10^{-6}/deg C)[19]) and Al_2O_3 ($\perp c$-axis, 7.7×10^{-6}/deg C), between 20–500 °C)[20]) are not too far apart at room temperature. Essentially no bowing could be observed for a 35 μm GaAs film grown on a 10 mil Al_2O_3 substrate. However, a 1 μm film did curl a 5 μm Al_2O_3 platelet concave downward, indicating that the residual stress in the GaAs is compressive.

3.1. PROPERTIES OF UNDOPED GaAs FILMS ON INSULATING SUBSTRATES

Studies on thick films deposited by the TMG–AsH_3 process have shown that excellent quality GaAs can be grown on (0001) Al_2O_3, (111) stoichiometric $MgAl_2O_4$[11]) and (110) stoichiometric $MgAl_2O_4$. The quality of the material, however, is found to be a function of film thickness, improving as the film is grown thicker. Since the electrical properties of the films change with thickness, the measured electrical parameters are necessarily averages over the thickness of the film. In general, the outermost layers of a film will have properties superior to the average value, and the layers nearer the interface will have inferior values.

Thick nominally-undoped films have been found to be n-type with net (average) carrier concentrations ranging from $\sim 10^{15}$ to 10^{17} cm^{-3} (refs. 9, 11), depending on the purity of the materials used to grow the films and the AsH_3–TMG concentration ratio used. Average mobilities greater than 5000 cm^2/V-sec have been obtained[11]).

Angle lapping and staining of n-type films have revealed the presence of a layer of high resistivity near the substrate surface[11]). It was found to be independent of film thickness, and therefore probably not related to diffusion of impurities from the substrate. The thickness of the layer was dependent on the carrier concentration of the donors in the film.

Once the film has converted to n-type, the carrier concentration rises rapidly to the thick-film value. The mobility tends to continue to improve as the film becomes thicker, usually "saturating" at a maximum value by the time the film has grown to $\sim$20 μm.

3.2. P-TYPE DOPING STUDIES USING DIMETHYLCADMIUM AND DIETHYLZINC

An all metalorganic-hydride process has been used at Autonetics to produce p-type GaAs films on Al_2O_3 and $MgAl_2O_4$[15]). Dimethylcadmium (DMCd) and diethylzinc (DEZ) have been combined with TMG–AsH_3 to produce films with net acceptor carrier concentrations up to about 5×10^{17} cm^{-3} (Cd-doped) and 8×10^{19} cm^{-3} (Zn-doped). The observed doping levels are found to be exponential functions of growth temperature when other parameters remain fixed. Nearly bulk electrical properties have been achieved in Zn-doped films as thin as 1 μm and in thick (>10 μm) Cd-doped films.

3. ELECTRON MICROSCOPY AND REFLECTION ELECTRON DIFFRACTION (RED) STUDIES

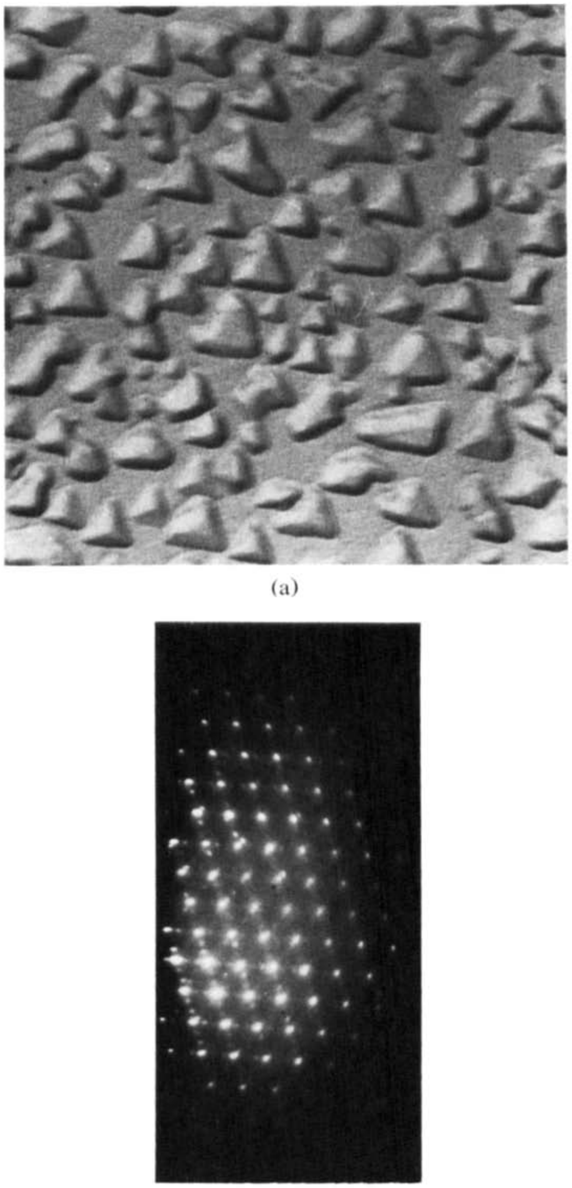
Studies of the early stages of growth of GaAs, under conditions which produce epitaxial films, indicate the formation of many discrete nuclei which coalesce to form large islands and eventually produce complete surface coverage. Annealing at or near the growth temperature has shown that the nuclei are highly mobile and move in such a fashion as to promote the growth of larger islands, as seen in fig. 2. Several of the crystallites in the figure appear to have merged, and most possess the triangular exterior shape associated with the growth of planes with trigonal symmetry. The RED pattern for this film indicates the presence of some twin structure in the GaAs. In addition, stacking faults could be inferred from the spots of irrational indices clustered closely around the indexed diffraction spots. Ordered structure has been confirmed in a deposit a fraction of a micron thick (~3000 Å) by RED patterns, although twinning is quite common in such thin layers. As film thickness increases, the quality of the growth improves, with electron microscope replicas showing a much smoother surface appearance and the RED patterns exhibiting Kikuchi lines and little evidence of twin structure in the upper layers.

3.4. GaAs ON GaAs

The Autonetics group has also performed a number of epitaxial growth experiments on (111) Cr-doped GaAs, but an extensive study has not yet been made. However, thick films (~35 μm) with measured mobilities exceeding 6500 cm^2/V-sec at room temperature at carrier concentrations of $\sim 5 \times 10^{15}$ cm^{-3} have been produced on GaAs and used to prepare working Gunn-effect devices[21]).

Thickness measurements of thin-film GaAs growth made simultaneously on (110) and (100) GaAs substrates indicated similar growth rates. This is different from what has been reported in halide systems[22]). This seems to indicate that the presence of halides in transport growth processes influences the growth rates on different orientations of the substrate.

i-Choudhury[9]) used the metalorganic–hydride method to deposit GaAs on (100) GaAs as well as on (0001) Al_2O_3. The low mobilities and high carrier concentrations found in films grown on both substrate materials are likely due to impurities in the reactants.


(a)

(b)

Fig. 2. GaAs/(0001)Al_2O_3 produced by reaction of residual TMG with AsH_3 for 6 min in an AsH_3–H_2 atmosphere. (a) Surface structure; (b) (110) RED pattern.

The absence of published film thickness data precludes the suggestion of any other reasonable explanations.

The studies of Lindeke et al.[16]) were limited to GaAs substrates, using gallium diethylchloride and AsH_3 to prepare GaAs. Room temperature mobilities of ~5000 cm^2/V-sec at carrier concentrations of 4.2×10^{15} cm^{-3} were obtained in films grown on (100) GaAs at deposition temperatures of 575 °C. It was not reported if the process would provide epitaxial films on insulating oxide substrates.

4. Other heteroepitaxial III-V compound semiconductor studies

The metalorganic-hydride growth process has been extended to the preparation of other epitaxial III–V compound semiconductors. Metalorganics were also found to be effective replacements for the hydrides in some growth systems. Table 2 indicates those III–V compounds that have been reported to date.

By reacting PH_3 with TMG, we were able to fo n GaP. However, it was apparent from the studies t' at controlling the growth of single-crystal films of GaP n insulators would be somewhat more difficult than ie control of GaAs film growth. Whereas GaAs films c ly 0.3 μm thick are single crystal on Al_2O_3, GaP film of the same thickness exhibited only highly prefe ed orientation on (0001) Al_2O_3 although single-cry al growth occurred on GaAs. However, the films on Al)$_3$

TABLE 2

Epitaxial III–V compound semiconductors formed from metalorganics and hydrides

Compound	Substrate	Reactants	Growth temperature (°C)	Ref.
GaAs	Ge, GaAs, Al_2O_3, $MgAl_2O_4$	TMG–AsH_3 TEG–AsH_3	650–750	6, 7, 9
GaP	GaAs, Al_2O_3	TMG–PH_3	700–725	7
	Si	TMG–TEP	485	8
$GaAs_{1-x}P_x$ ($x = 0.1$–0.6)	GaAs, Al_2O_3, $MgAl_2O_4$	TMG–AsH_3–PH_3	700–725	7
$GaAs_{1-x}Sb_x$ ($x = 0.1$–0.3)	GaAs, Al_2O_3	TMG–AsH_3–SbH_3 TMG–AsH_3–TMSb	725	7
AlAs	GaAs, Al_2O_3	TMA–AsH_3	700	12, 13
$Ga_{1-x}Al_xAs$ ($x = 0.2$–0.9)	GaAs, Al_2O_3	TMG–TMA–AsH_3	700	12
AlN	Si, Al_2O_3, α-SiC	TMA–NH_3	1250	17
GaN	Al_2O_3, α-SiC	TMG–NH_3	925–975	17

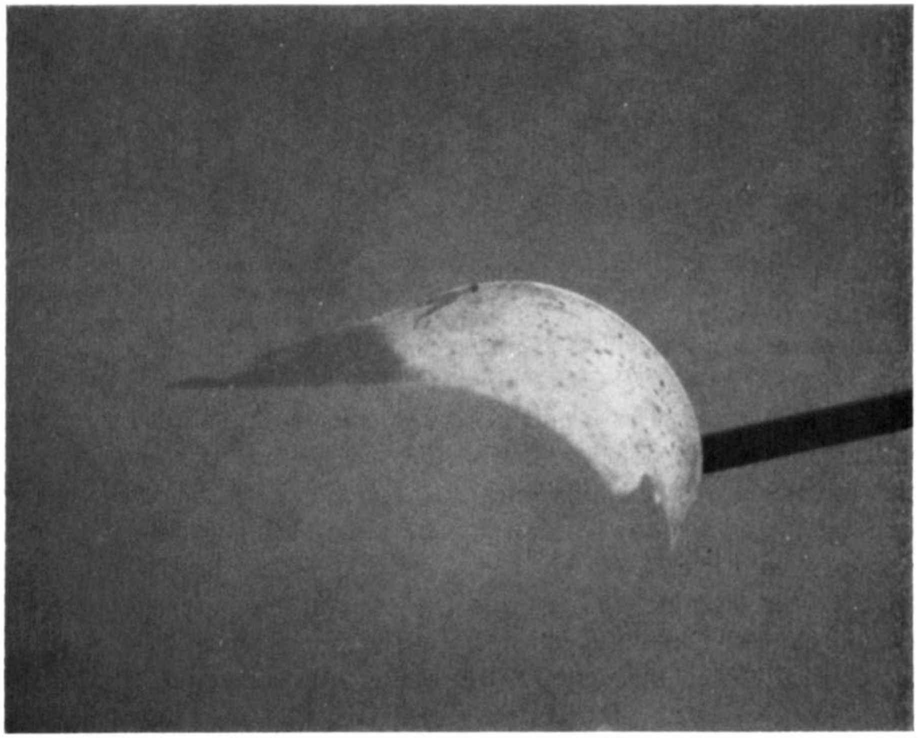


Fig. 3. Bending of a thin sapphire platelet caused by residual stress in the GaP film.

nproved in quality with thicker growth; the surfaces ere identified as single-crystal (111) orientation by ED.

As was observed for GaAs on a thin Al_2O_3 platelet, aP growth caused curling of the platelet in a concave ownward direction. This indicated the residual stress the GaP film to be compressive. This effect is shown fig. 3.

Additions of PH_3 to the AsH_3–TMG mixtures normally used for growing GaAs films led to the formation of single crystal $GaAs_{1-x}P_x$ films. In the preliminary studies, single crystal films with the composition $GaAs_{0.9}P_{0.1}$ and $GaAs_{0.4}P_{0.6}$ were grown on (0001) Al_2O_3 and (111) $MgAl_2O_4$.

Thomas[8]) produced single-crystal growth of GaP on Si by the thermal decomposition of a gas-phase mixture of TEG and TEP at 485 °C. Extremely slow growth rates (~2000 Å/hr) were used to produce 2700–4500 Å thick films. Unfortunately it was not reported if the success of the process is growth rate, substrate, and/or temperature limited.

Manasevit and Simpson[7]) found that adding SbH_3 to AsH_3–TMG mixtures yields $GaAs_{1-x}Sb_x$ alloys. Films with approximate compositions in the range $GaAs_{0.9}Sb_{0.1}$ to $GaAs_{0.7}Sb_{0.3}$ were obtained by arbitrarily changing the AsH_3 flow and/or H_2 flow through the liquid SbH_3. Better growth, as determined by RED, was obtained on (100) GaAs than on (0001) Al_2O_3 at the higher concentration of Sb. Epitaxial alloy films were also achieved by using TMSb in place of SbH_3.

The metalorganic–hydride process was also demonstrated to be feasible for the production of single-crystal films of AlAs and the mixed III–V Al compounds, materials of interest for opto-electronic devices[12]). By pyrolyzing TMA and AsH_3 in a H_2 atmosphere at about 700 °C, single-crystal films of AlAs have been ned on GaAs[12,13]) and on single-crystal insulating trates[12]). A 1–2 μm thick yellow layer of (111) AlAs ned on (0001) Al_2O_3 was found to be unstable in air. inden[13]) has recently reported on some of the cal properties of AlAs films prepared by the TMA– process on GaAs substrates. The optical absorp- edge measured on a polycrystalline layer on quartz cated a direct bandgap of 3.5 eV. The indirect gap 2.8 eV; and the high-frequency dielectric constant determined to be 15.2. The surfaces of the AlAs films grown on GaAs were also reported to be unstable.

We attempted to produce AlP by the pyrolysis of TMA in H_2 at 700 °C in an atmosphere containing excess P formed by decomposing PH_3. The resulting film (~1 μm thick) was not stable in the laboratory atmosphere, even during the minute it took to transfer the wafer to the electron microscope chamber. Reid et al.[23]) also observed reaction at the AlP surface on exposure to moist air for films formed at 900 °C. Some of the unexpected optical effects observed by Richman for AlP films[24]) might also be related to this instability.

Alloys of $Ga_{1-x}Al_xAs$ have also been prepared in the Autonetics laboratories by mixing different quantities of TMA and TMG and reacting these with AsH_3 at the heated pedestal. Simple flowmeter adjustments controlled the TMA and TMG ratios. Alloys over the composition range $Ga_{0.1}Al_{0.9}As$ to $Ga_{0.8}Al_{0.2}As$, as analyzed with the electron microprobe, have been prepared on Al_2O_3 and GaAs substrates.

Nitrides of Al and Ga have also been produced in our laboratories by the metalorganic–hydride process. Pyrolysis of TMA and NH_3 in H_2 formed high-resistivity single-crystal insulating films of AlN on Si, α-SiC

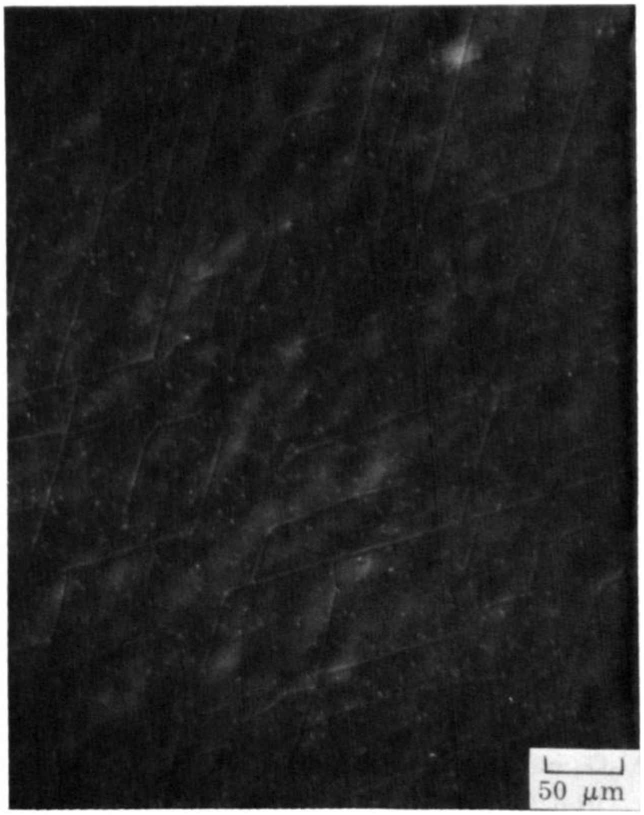


Fig. 4. AlN growth features on (111) Si.

and Al_2O_3 at temperatures of about 1250 °C[17]). Crazing which was observed in 1 μm thick films grown on (111) Si (fig. 4), did not occur on $(01\bar{1}2)$ Al_2O_3. Single-crystal films of GaN have been produced from NH_3 and TMG on α-SiC and Al_2O_3 at temperatures of about 925–975 °C. The only GaN film on Al_2O_3 that was measured was n-type, with a carrier concentration above 10^{19} cm^{-3}, and a mobility ~60 cm^2/V-sec[17]).

5. Heteroepitaxial II-VI compound semiconductor films

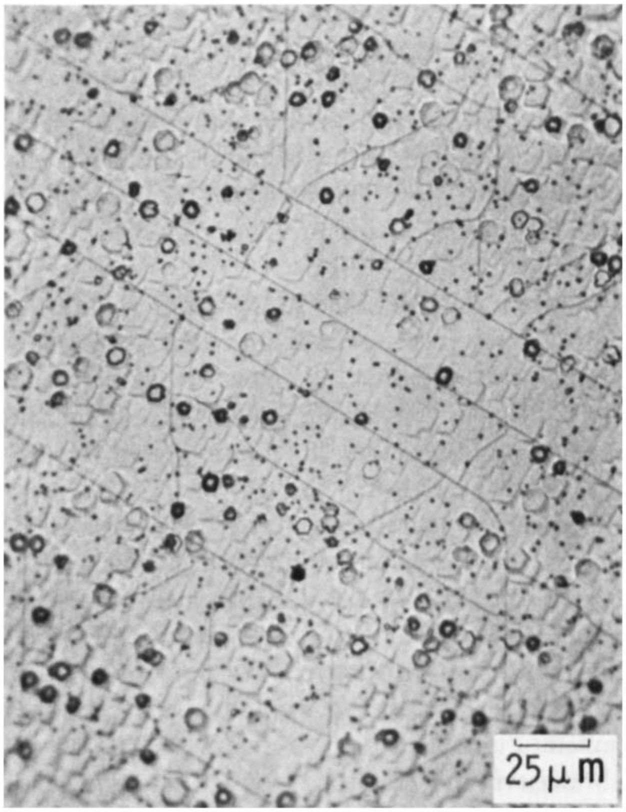
II–VI compounds have also been grown on insulating substrates using metalorganics and hydrides[14]). The II–VI compounds which have been produced by this method are summarized in table 3. We have produced these films by reacting DEZ or DMCd with H_2Se, H_2S or dimethyltellurium (DMT) using essentially the same apparatus previously described for the formation of GaAs and other III–V compounds, but in some experiments a vertical quartz reactor containing a side arm and a center tube extending to about 1–2 inches from the pedestal was used. The hydrides are usually introduced into the reactor through the side arm prior to admitting the metalorganic in its carrier gas via the center tube. The product of their mixing is then decomposed at the heated pedestal to produce the II–VI compound. ZnSe growth on a number of orientations of Al_2O_3, BeO, and $MgAl_2O_4$ supplied the same heteroepitaxial relationships as those found for the growth of GaAs on these substrate planes (see table 1). This, therefore, suggests a similar growth mechanism. Goodman[25]) also obtained (111) ZnSe growth on $(01\bar{1}2)$ Al_2O_3 but by an evaporation technique.

TABLE 3

Compound II–VI semiconductors formed from metalorganics and hydrides

Compound	Substrate	Reactants	Growth temperature (°C)	Ref.
ZnSe	Al_2O_3, $MgAl_2O_4$, BeO	DEZ–H_2Se	725–750	14
ZnS	Al_2O_3, $MgAl_2O_4$, BeO	DEZ–H_2S	750	14
ZnTe	Al_2O_3	DEZ–DMT	500	14
CdSe	Al_2O_3	DMCd–H_2Se	600	14
CdS	Al_2O_3	DMCd–H_2S	475	14
CdTe	Al_2O_3, $MgAl_2O_4$, BeO	DMCd–DMT	500	14

The nature of the surface of ZnS growth on Al_2O_3, $MgAl_2O_4$, and BeO is recorded in fig. 5. Some crazing can be observed in the films grown on (0001) Al_2O_3 and (111) $MgAl_2O_4$. The films were grown at about 750 °C from DEZ and H_2S.



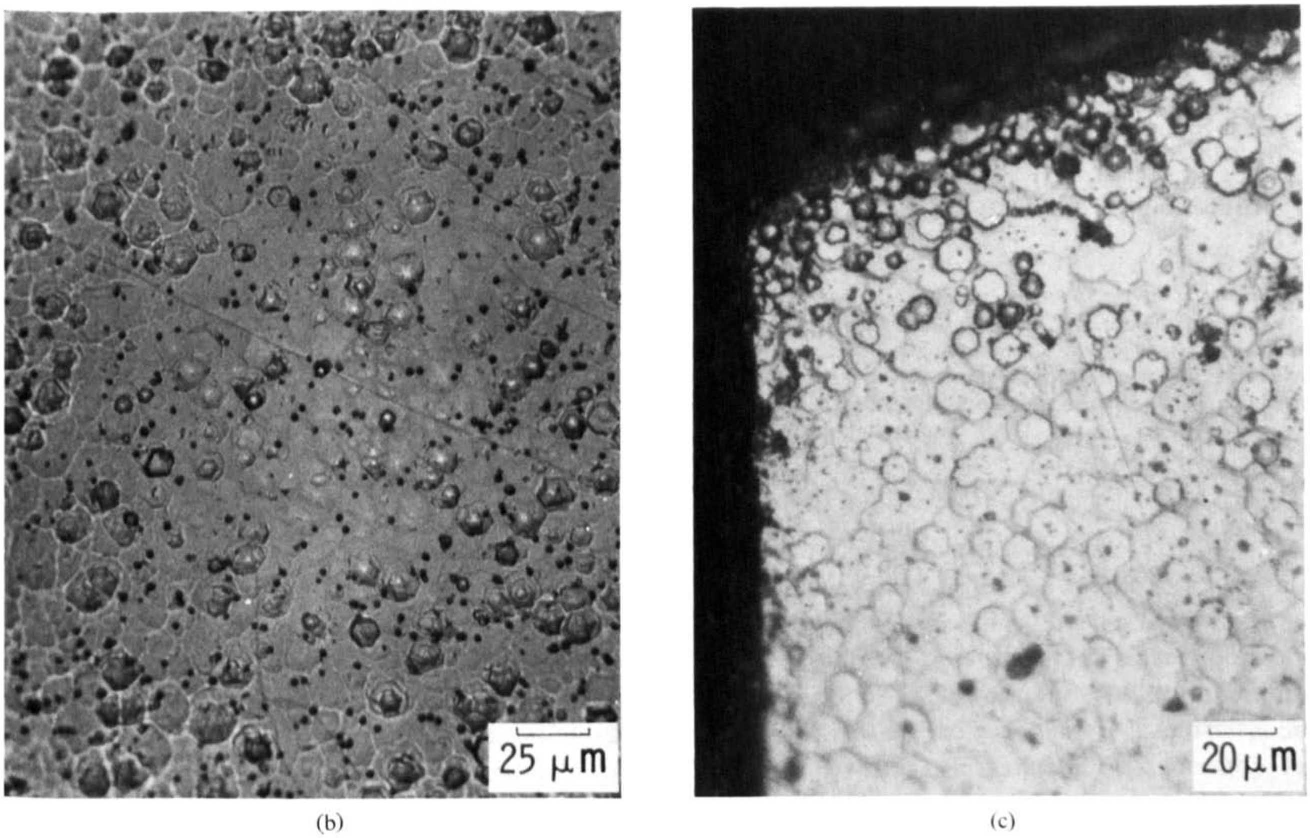
(a)

Single-crystal films of (111) ZnTe were also produced on Al_2O_3. They were made by reacting DEZ with DMT at about 500 °C. The films possessed a deep orange color and varied in reflectivity with change in substrate orientation.

Replacing the DEZ with DMCd led to the formation of the corresponding Cd-group VI semiconducting compounds. CdS was obtained on (0001) Al_2O_3 by reacting DMCd with H_2S at 475 °C; the growth of CdSe on (0001) Al_2O_3 from DMCd and H_2Se was achieved at about 600 °C; and single-crystal film of CdTe were produced at about 500 °C from DMCd and DMT on Al_2O_3, $MgAl_2O_4$, and BeO.

6. Conclusions

The results to date using metalorganics in the preparation and doping of compound semiconductors are encouraging. The technique obviates the use of multiple temperature zones for the CVD process, simplifies



(b) (c)

Fig. 5. ZnS growth on (a) (0001) Al_2O_3; (b) (111) $MgAl_2O_4$; (c) $(10\bar{1}1)$ BeO.

the procedure of compound growth considerably, minimizes autodoping which might be caused by the attack of the substrate by transport agents used in other CVD processes, and makes compound semiconductor film growth compatible with the growth of elemental semiconductors. One can expect this new CVD technique to be very instrumental in making different compound semiconductor films more available for device studies.

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