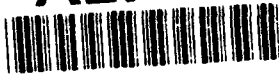


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3. ABSTRACT (Maximum 200 words)

The overall goals are to develop the fundamental chemistry of group III and group V compounds in several key areas. The underlying rationale is that we may be able to ultimately improve on the preparation and properties of important materials used in the semiconductor industries. The research comprises a collaboration between a synthetic inorganic chemist and a chemical engineer. The three main areas are (1) single source precursors for OMCVD of III/V semiconductors, (2) alternative precursors for OMCVD studies, group III or V hydrides, and (3) the organometallic chemistry of bismuth. The first area will employ compounds of the general type $[R_2M(\mu-R'_2E)]_2$ ($M=Al, Ga, In$; $E=P, As$; $R, R'=alkyl, aryl, etc.$) and the second will employ similar compounds but with R or R' replaced by the hydride ligand. We will explore the relationships between precursor structures, mechanisms of decomposition and thin film formation, and the morphological and electrical properties of the grown films. In the third area we will explore the organometallic chemistry of bismuth and compounds which may serve as precursors to important materials such as InBiSb.

14. SUBJECT TERMS OMCVD, precursors, thin film formation, organometallic.			15. NUMBER OF PAGES
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Sincerely,

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Dr. John G. Ekerdt DAAL03-89-K-0164
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1 MAY 1993

FINAL REPORT - ARO PROPOSAL NUMBER 26748-CH - 7/31/93

**Single Source Precursors for the OMCVD of III/V
Compound Semiconductors**

FINAL REPORT

Richard A. Jones and John G. Ekerdt

1 January 1993 - 31 May 1993

**U. S. ARMY RESEARCH OFFICE
26748-CH
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A. STATEMENT OF PROBLEM STUDIED

**Single Source Precursors for the OMCVD
of III/V Compound Semiconductors**

The overall goals are to develop the fundamental chemistry of group III and group V compounds in several key areas. The underlying rationale is that we may be able to ultimately improve on the preparation and properties of important materials used in the semiconductor industries. The research comprises a collaboration between a synthetic inorganic chemist and a chemical engineer. The three main areas are (1) single source precursors for OMCVD of III/V semiconductors, (2) alternative precursors for OMCVD studies, group III or V hydrides, and (3) the organometallic chemistry of bismuth. The first area will employ compounds of the general type $[R_2M(\mu-R'_2E)]_2$ ($M=Al, Ga, In$; $E=P, As$; $R, R'=alkyl, aryl, etc.$) and the second will employ similar compounds but with R or R' replaced by the hydride ligand. We will explore the relationships between precursor structures, mechanisms of decomposition and thin film formation, and the morphological and electrical properties of the grown films. In the third area we will explore the organometallic chemistry of bismuth and compounds which may serve as precursors to important materials such as InBiSb.

B. SUMMARY OF IMPORTANT RESULTS

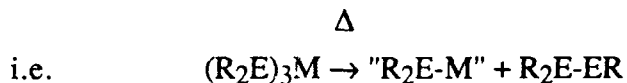
The technical details of the research results are more fully documented in the publication and technical reports previously filed during the period of this research. Our initial experiments focused on the decomposition pathways of the dinuclear GaAs precursors $[\text{Me}_2\text{Ga}(\mu\text{-}t\text{-Bu}_2\text{As})]_2$ (**1**), $[\text{Et}_2\text{Ga}(\mu\text{-}t\text{-Bu}_2\text{As})]_2$ (**2**), $[\text{Me}_2\text{Ga}(\mu\text{-}i\text{-Pr}_2\text{As})]_3$ (**3**), and $[\text{Me}_2\text{Ga}(\mu\text{-Me}_2\text{As})]_3$ (**4**).

Two reactor systems have been used. The initial pyrolysis experiments were conducted in a low pressure system ($<10^{-4}$ Torr) in order to reproduce the conditions under which GaAs films are deposited. The pyrolysis system consists of a quartz tube through which the precursors flow. The temperature of the pyrolysis zone is increased linearly with time. Reactions are monitored by feeding the reactor effluent directly into an HP 5970B Mass EI mass spectrometer which was modified to accommodate the large gas load. The results indicate that for the dimeric precursors, **1** and **2**, the 1:1 Ga:As stoichiometry present in the precursor remains intact until the molecule decomposes to form GaAs on a heated surface. Trimeric precursors, **3** and **4**, appear, however, to lose their stoichiometry by reacting to form diarsines, Me_4As_2 for example from **4**, at temperatures as low as 150°C . One possible explanation for diarsine formation from trimers and not dimers could be associated with the possibility of forming a stable product from the trimer, such as $[\text{R}_2\text{Ga}]_3\text{As}$. A related compound, $[(\text{thf})\text{Br}_2\text{Ga}]_3\text{As}$, has been reported in the literature [R.L. Wells, S. Shafieezad, A. T. McPhail and C. G. Pitt, J. Chem. Soc., Chem. Commun., 1823 (1987)] and forms the basis for our explanation. A trigallium-arsine compound cannot form from a single dimer and this may explain the lack of diarsine formation from the dimers. The pyrolysis results indicate the *t*-Bu ligands begin to be eliminated from the As atoms at 325°C and reaction is complete by 450°C . The *i*-Pr ligands are slightly more thermally stable with reaction beginning at 375°C and with complete elimination occurring by 500°C . Methyl ligands appear to be eliminated from the Ga atoms between 400 and 500°C . Ethyl ligands are more thermally labile with elimination occurring from 325 to 450°C . The other reactor system, which was constructed during the period of the grant, is designed for chemical beam epitaxy. The system features a load lock/preparation chamber and a main deposition chamber each of which are pumped by turbomolecular pumps. A mass spectrometer has been fitted to the deposition chamber in order to insure that conditions are kept constant throughout a

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deposition and to guarantee repeatability between runs. Using the new system epitaxial growth of GaAs has finally been achieved on (100) GaAs substrates using compounds **1** and **2**. The key to achieving epitaxy appears to lie in operating the reactor at pressures no greater than 10^{-5} Torr and at temperatures of 400°C and above. At higher pressures there is apparently still enough gas phase reaction and nucleation for the films to become polycrystalline. Photoluminescence spectra of films grown from **1** at 500°C indicate the presence of both silicon and carbon as impurities. ICP-MS analysis of the starting material shows high concentrations of Si which probably arise from stopcock grease used in the synthesis. Steps are being taken to eliminate this source of contamination. The carbon, however, probably originates from the methyl ligands of the precursor.

Since the binary (homoleptic) compounds of general formula $(R_2E)_3M$ ($M = Al, Ga, In$; $E = P, As$; $R = t-Bu, i-Pr$) contain no M-carbon bonds we considered them to be possible precursors for the ME materials despite the apparently unfavorable 1:3, M:E ratio. They have proven to be surprisingly versatile as OMCVD precursors. We believe that this may be due to the fact that they contain no metal-carbon bonds and the mechanistic pathway for decomposition involves the expulsion of the small stable molecule R_2E-ER_2 .



The R_2E-M fragment then decomposes to ME. Thus $(t-Bu_2As)_3Ga$ gives epitaxial layers of GaAs at 525°C, 5×10^{-5} Torr pressure. The low temperature (77 K) photoluminescent spectra indicate that the material is n-type and has $1500 \text{ cm}^2/\text{V}\cdot\text{sec}$. mobility.

Several other classes of compounds have been evaluated for film growth. These include a base free diphosphadigallate $[Ar'PGa-t-Bu]_2$ $Ar'=(2,4,6-t-Bu_3-C_6H_2)$; aluminum based compounds which feature PH_2 or AsH_2 units $[t-Bu_2Al(EH_2)]_3$ ($E=P, As$); $\mu-t-BuPH$ and $\mu-t-BuAsH$ bridged dimers $[t-Bu_2Ga(\mu-t-BuPH)]_2$ and $[t-Bu_2Ga(\mu-t-BuAsH)]_2$ as well as cubanes of the type $[\phi_3SiEM-t-Bu]_4$ ($M=Al, Ga$).

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Other studies have continued to focus on various aspects of the decomposition pathways for compounds featuring group 13/15 elements. In particular the cyclic trimeric hydroxy, amido, phosphido and arsenido derivatives of aluminum and gallium of general formula $[\text{t-Bu}_2\text{Al}(\mu\text{-X})_3]$ ($\text{X} = \text{EH}_2$; $\text{E} = \text{N, P, As}$; $\text{X} = \text{OH, OD}$) have been isolated and fully characterized. The use of these compounds as single source precursors to the appropriate solid state semiconductor material is currently under investigation.

Other studies have focused on precursors to gallium nitride (GaN). This is particularly difficult to grow thin films of partly because of the volatility of nitrogen. We have isolated and characterized both primary amide and amine complexes of both gallium and indium and structurally characterized the complexes $[\text{Me}_2\text{Ga}(\mu\text{-NH-t-Bu})_2]$, $\text{Me}_3\text{Ga.NH}_2\text{-t-Bu}$ and $\text{Me}_3\text{In.NH-t-Bu}$. Again, these compounds as well as the alkylamidogallates $\text{Li}(\text{THF})_2(\mu\text{-NH-t-Bu})_2\text{GaCy}_2$ and $\text{MgCl}(\text{THF})_2(\mu\text{-3,5-Me}_2\text{Pz})_2\text{GaCy}_2$ may prove to be viable single source precursors. Of particular interest is the X-ray structure of $\text{Cy}_2\text{GaBrNH}_2\text{Ph}$ which contains a close $\text{Ga}\cdots\text{H-N}$ (agostic) interaction. This may well represent a model of the way in which alkane is eliminated from the reaction of a trialkyl gallium species with an amine to give either a gallium amide

$$\text{R}_3\text{Ga} + \text{H}_2\text{NR} \rightarrow \text{R}_2\text{Ga-NHR}$$

or finally gallium nitride itself.

Finally, examples of a novel class of monomeric precursor which features M-H bonds have been isolated and characterized. Examples include $\text{H}_2\text{AlP}(\text{Mesityl})_2\bullet\text{NMe}_3$ and $\text{H}_2\text{AlAs}(\text{Mesityl})_2\bullet\text{NMe}_3$.

C. LIST OF MANUSCRIPTS PUBLISHED UNDER ARO SPONSORSHIP

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- *25. "Alkyl Amido Gallate Complexes of the Group I and II Elements," D. A. Atwood, A. H. Cowley and R. A. Jones, *Organometallics*. **1993**, 12, 236-238.
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- 1. "Synthesis and Structural Characterization of a Homoleptic Bismuth Aryl Thiolate" D. A. Atwood, A. H. Cowley, R. D. Hernandez, R. A. Jones, L. L. Rand, S. G. Bott and J. L. Atwood, *Inorg. Chem.* Accepted.

SUBMITTED

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- *5. "Dimeric Gallium and Indium Dialkylphosphido Complexes with Unusual Group 13/15 Stoichiometries," D. A. Atwood, V. O. Atwood, A. H. Cowley, H. R. Gobran, and R. A. Jones, *Organometallics*. Submitted.
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D. LIST OF ALL PARTICIPATING SCIENTIFIC PERSONNEL
SHOWING ADVANCED DEGREES EARNED
WHILE EMPLOYED ON THE PROJECT

Graduate Students Supported:

David A. Atwood
Paul R. Harris
Michael Jackson
Vikas Lakheta
Miguel Mardones
James E. Miller

Ph.D.'s Awarded:

**David A. Atwood - "A Synthetic and Structural Study of Heavier Group 13
Complexes of Nitrogen"**

Paul R. Harris - "Synthesis, Characterization & Application of Novel III/V and III/VI Organometallic Compounds"

James E. Miller - "Chemical Beam Epitaxy of GaAs Using Novel Single-Source Precursors"

John W. Nail - "Single Source Precursors for the III/V Semiconducting Materials: From Design to Deposition"

Ronald J. Powell - "A Study of the Chelating Ability of Tertiary Diphosphine Ligands at a Nickel (II) Center"

Lora L. Rand - "Synthesis, Characterization and Reactivity of Sterically Hindered Sulfido-, Selenido- and Tellurido- Transition Metal and Main Group Complexes"

Mark D. Soucek - "Stabilization of Metal Clusters with 1,2-Phenylene Linked Diphosphines"

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