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September 10, 1992

Ms. Angela Potter
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Sugject: NOOO14-89-J-1849

Dear Ms. Potter:

Enclosed is a copy of the Final Report on the above-referenced grant.

Sincerely,

Aaron Wold
Professor of Chemistry
and Engineering (Research)

AW:mp
xc: R. DeMarco/ONR (3 copies)
Director, Naval Research Lab (6 copies)
Defense Tech. Information Center (12 copies)
C. Krause, Brown U. (1 copy)

FINAL REPORT: P.I.: Aaron Wold, Department of Chemistry, Brown University

Summary of work done under contract NOOO 14-89-J-1849 (R&T Code 413u001) during contract period May 1, 1989 to May 31, 1992.

(See summaries from End-of-Year Reports dated September, 1989, May, 1990, May 1991, May 1992 - partially reproduced below).

From 1989 End of Year Report

The work on the synthesis of metal oxide and sulfide thin films was started. This program centered around the development of new methods for the preparation and characterization of oxide and sulfide thin films, as well as the investigation of materials which transmit in the far infrared. An effort was also started on the preparation of diamond films because of its potential as a material for infrared transmission. All of the materials prepared were characterized as to their electronic and optical properties, stability toward decomposition and hardness. Several special transparent gold furnaces were designed and built so the entire growth process could be seen.

During this year we used the novel spray pyrolysis technique developed in this lab to prepare metal oxide and metal sulfide thin films. An aqueous solution of the metal was sprayed onto a substrate and by proper choice of the complex of metal ion, sufficient volatilization took place so that the resulting oxide deposited as a uniform homogeneous film. The decomposition took place in air or oxygen. By this method CuO, ZnO and Al_2O_3 films were grown. Conversion of ZnO to ZnS took place by heating the ZnO films in H_2S.

From 1990 End of Year Report

It was shown that the substitution of iron, cobalt and nickel for zinc significantly increased the hardness of both ZnS and ZnSe. Furthermore the thermal stability of the II-VI compounds was not reduced by the substitution for zinc by these transition metals.

An inexpensive tungsten hot filament reactor was designed, built (at a cost of under $5,000) and produced diamond films on a variety of substrates (Si, Cu, Au, Ni) comparable in quality to those formed with more expensive reactors.

From 1991 End of Year Report

Work on the growth of diamond thin films continued and films were produced on silicon, copper and gold substrates. It was observed that whereas diamond can be readily nucleated on all three substrates, the nucleation density is greatly enhanced by prescratching with 0.25 micron diamond paste. Deposited diamond films adhered to the silicon substrates but could be readily removed from the gold and copper substrates.

Also during this period, work continued on attempts to improve ZnS by the addition of transition metals which has been reported to harden ZnS and improve thermal stability. Hardness measurements made on ZnS containing iron showed that initially the addition of iron caused an increase in hardness.

However, larger substitutions of iron did not further affect the hardness. Thermal stability measurements showed that there was very little effect on the thermal stability as iron was substituted for zinc in ZnS.

Finally, our work on the growth and characterization of La_2CuO_4 single phase thin films from a source solution with a La:Cu ratio of 2:1. Alcoholic solutions of lanthanum copper acetylacetonates were nebulized and pyrolized in order to produce the films.

From 1992 End of Year Report

Single crystals of members of the system $(ZnS)_{1-x}(CuGaS_2)_x$ (x = 0, 0.053, 0.103, 1.00) were grown by chemical vapor transport using iodine as the transport agent. The IR transmission range of $(ZnS)_{0.947}(CuGaS_2)_{0.053}$ and $(ZnS)_{0.897}(CuGaS_2)_{0.103}$ is narrower than that observed for pure ZnS. However these materials still showed good transmission in the long wavelength IR range. The addition of small amounts of $CuGaS_2$ increased the hardness and thermal stability of ZnS. Therefore, these materials might be useful for the development of IR windows in the long wavelength range.

We were also able to show that diamond films can be grown on stainless steel if a silicon buffer layer is first deposited on the steel. It appears that the silicon layer, approximately 3500 Å thick retards graphite formation and allows the diamond to form. The silicon layer must also be pre-scratched before the diamond film can be grown successfully.

Technical Reports Submitted during period of contract

Technical Report 1. Growth and Characterization of Zinc Sulfide Films by Conversion of Zinc Oxide Films with H_2S. Sept., 1989.

Technical Report 2. The Preparation and Properties of Cobalt-Doped II-VI Chalcogenides. September, 1989.

Technical Report 3. Preparation and Characterization of Alumina Films Prepared by a Novel Spray Pyrolysis Method. Jan. 1990.

Technical Report 4. Preparation and Characterization of $Cu_2ZnGeS_{4-y}Se_y$. January, 1990.

Technical Report 5. Growth and Characterization of Gallium(III) Oxide Films. January, 1990.

Technical Report 6. Preparation and Properties of the System $Cu_xPd_{1-x}O$ ($0 \leq x \leq 0.175$). January 1990.

Technical Report 7. The Preparation and Properties of Iron Doped II-VI Chalcogenides. January 1990.

Technical Report 8. Synthesis of Oxides Containing Transition Metals. July, 1990.

Technical Report 9. Preparation and Characterization of Chromium(III) Oxide Films by a Novel Spray Pyrolysis Method. December 1990.

Technical Report 10. Nucleation and Growth of Diamond on Si, Cu and Au Substrates. December 1990.

Technical Report 11. Preparation and Characterization of Dispersed "Cobalt Oxide" supported on γ-Al_2O_3. December 1990.

Technical Report 12. Preparation and Characterization of Iron(III) Oxide Films by a Novel Spray Pyrolysis Method. December 1990.

Technical Report 13. Growth and Characterization of Thin Films of Y_2O_3, La_2O_3 and La_2CuO_4. December 1990.

Technical Report 14. Nucleation and Growth of Diamond on Si, Cu and Au Substrates. January 1992.

Technical Report 15. Preparation and Characterization of Dispersed Cobalt Oxide Supported on γ-Al_2O_3.

Technical Report 16. Preparation and Characterization of Conducting Transition Metal Oxides. January 1992.

Technical Report 17. Preparation and Characterization of Iron(III) Oxide Films by a Novel Spray Pyrolysis Method. January 1992.

Technical Report 18. Preparation and Characterization of Phases Formed by the Reaction of *Nickel(II)* and Cobalt(II) Precursors with MgO, $MgAl_2O_3$and γ-Al_2O_3. March 1992

Technical Report 19. The Crystal Growth and Characterization of the Solid Solutions $(ZnS)_{1-x}(CuGaS_2)_x$. March 1992.

<u>**Journal articles published during contract period of 5-1-89 to 5-31-92**</u>

Y-M. Gao, P. Wu, J. Baglio, K. Dwight and A. Wold, *Mat. Res. Bull.*, <u>24</u>, 1215 (1989).

C-M. Niu, R. Kershaw, K. Dwight and A. Wold, *J. of Solid State Chem.*, <u>85</u>, 262 (1990).

W. J. DeSisto, Y-T. Qian, C. Hannigan, J. O. Edwards, R. Kershaw, K. Dwight and A. Wold., *Mat. Res. Bull.*, <u>25</u>, 183 (1990).

K. Doverspike, K. Dwight and A. Wold., *Chemistry of Materials*, <u>2</u>, 463 (1990).

P. Wu, Y-M. Gao, R. Kershaw, K. Dwight and A. Wold., *Mat. Res. Bull.*, <u>25</u>, 357 (1990).

C-M. Niu, P. H. Rieger, K. Dwight and A. Wold., *J. Solid State Chem.*, <u>86</u>, 175 (1990).

J. DiCarlo, M. Albert, K. Dwight and A. Wold., *J. Solid State Chem.*, 87, 443 (1990).

A. Wold and K. Dwight, *J. Solid State Chem.*, 88, 229 (1990).

Y-T. Qian, R. Kershaw, K. Dwight and A. Wold., *Mat. Res. Bull.*, 25, 1243 (1990).

C-M. Niu, G. Tsagaropoulos, J. Baglio, K. Dwight and A. Wold., *J. Solid State Chem.*, 91, 47 (1991).

C-S. Bai, S. Soled, K. Dwight and A. Wold, *J. Solid State Chem.*, 91, 148 (1991).

Y-T. Qian, C-M. Niu, C. Hannigan, S. Yang, K. Dwight and A. Wold, *J. Solid State Chem.*, 92, 208 (1991).

Y-M. Gao, P. Wu, K. Dwight and A. Wold., *J. Solid State Chem.*, 90, 228 (1991).

C-S. Bai, S. Soled, R. Kershaw, K. Dwight and A. Wold, *J. Solid State Chem.*, 96, 376 (1992).

Y-R. Do, R. Kershaw, K. Dwight and A. Wold., *J. Solid State Chem.*, (1992 In press.)

PERSONNEL PARTICIPATING IN RESEARCH DURING 3-YEAR CONTRACT

Graduate Students	Other Researchers
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J. Papp	K. Dwight
W. DeSisto	J. Baglio
C. Hannigan	P. Rieger
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