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SYNTHESIS, EVALUATION, AND DEFECT COMPENSATION OF
TETRAHEDRAL GLASSES AS POSSIBLE SOLAR CELL MATERIALS

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ABSTRACT

This report describes the work performed during the second quarter of the project to synthesize $A^{II}B^{IV}C^V$ glasses and evaluate their potential use as solar cell materials. The major effort was directed towards preparation of these films by thermal evaporation of compounds synthesized during the first quarter. Films were produced from $CdAs_2$, $CdSnAs_2$, $CdSiAs_2$, $ZnSiAs_2$, and $ZnAs_2$. Only films from the last compound had a stoichiometry comparable to the starting material. The ternary films were deficient in the group IV element and films produced from $CdAs_2$ indicated the sample had undergone extensive decomposition. In addition to having good stoichiometries, the X-ray analysis of the $ZnAs_2$ films indicated they were amorphous. The work thus represents the first reported results of preparation of amorphous films of $ZnAs_2$. The optical and electrical properties of this material will be characterized in the next quarter.

DESCRIPTION OF PROJECT

The goal of this project is to evaluate the potential of the $A^{II}B^{IV}C_2^V$ tetrahedral glasses as solar cell materials. The chalcopyrite glasses have structural and electronic properties which allow their chemical modification for ideal photovoltaic characteristics. The research is directed towards four principal tasks.

1. Synthesis and characterization of bulk quantities of $A^{II}B^{IV}C_2^V$ compounds.
2. Production of amorphous thin films of these materials.
3. Evaluation of optical and electrical properties of the amorphous materials.
4. Construction and testing of solar cells.

The work will concentrate on the series $CdMP_2$, $CdMAs_2$, $ZnMP_2$ and $ZnMAs_2$ where M is Ge, Si or Sn, since these materials are the least expensive and have the most promising band gaps.

TABLE OF CONTENTS

<u>Section</u>	<u>Page</u>
ABSTRACT.	i
DESCRIPTION OF PROJECT.	ii
I. INTRODUCTION.	1
II. EXPERIMENTAL.	2
III. RESULTS	6
IV. DISCUSSION	8
V. REFERENCES	11

I. INTRODUCTION

Thin films offer one of the most promising methods of making solar cells using large scale production because of reduced material requirements and ease of covering large areas. Whereas considerable difficulties are encountered in making single crystal thin films, amorphous films are more easily obtained. The ternary $\text{AlIBIVC}_2\text{V}$ compounds which are the subject of current research have exhibited a tendency to form glasses. The work this quarter, therefore, was directed towards making thin amorphous films using the compounds prepared as described in the previous quarterly report.

A computer search of the literature on $\text{AlIBIVC}_2\text{V}$ compounds revealed that thin films have been made by liquid phase epitaxy (LPE) (1,2), explosion in vacuum (3), and sputtering of polycrystalline materials (4). Since the goal of the LPE work was to produce orientated crystalline materials, it is not directly relevant to our fabrication of amorphous films. One of the methods described, however, used a vertical dipping technique in an open tube system (1). The substrate was single crystal material and careful consideration was given to the cooling rate and speed of withdrawal of the substrate from the melt. The method is inherently so simple that it was thought worthwhile to attempt to make amorphous films using a similar technique employing glassy substrates and rapid quenching of the film.

Perhaps the most promising method for formation of amorphous thin films is vacuum deposition. CdGeAs_2 , ZnGeAs_2 and CdGeP_2 have been formed by explosion in vacuum and ZnGeAs_2 by sputtering. The first method did not result in formation of films with the same stoichiometry as the bulk material; the films were deficient in Ge, the highest melting constituent of the compounds (3). Sputtering of ZnGeAs onto an aluminum substrate, however, produced stoichiometric amorphous films (4). These results indicate that vacuum evaporation of the ternary compounds will probably lead to nonstoichiometric films. Better results, however, might be expected for the higher melting ternaries such as ZnSiAs_2 (m.p. 1096°C) (5). In addition, the films will provide samples for developing techniques to measure optical band gaps, chemical composition and electrical properties.

II. EXPERIMENTAL

Amorphous Film Formation from Solution

An apparatus has been designed to attempt to make amorphous films by dipping of substrates into melts of the ternary compounds. Figure 1 shows the quartz reaction tube which fits into a vertical tube furnace. The furnace will attain a maximum temperature of 1200°C. A quartz substrate is attached to the bottom of the center tube. The tube is hollow to accept a thermocouple for measuring the substrate (and melt) temperature. A second tube along the side of the vessel is used to purge the vessel of oxygen before the heating cycle and to direct cold gas on the substrate for rapid quenching of the film as it is withdrawn from the melt.

The dipping apparatus has not yet been tried due to the difficulty in securely attaching the quartz substrate to the inner tube. CdGeAs₂ (m.p. = 650°C) will be the first ternary tried because of its known ease for glass formation. Other ternary compounds which will be used are CdSiAs₂ (m.p. = 715°C), and ZnSnAs₂ (m.p. = 775°C). Since CdSnP₂ forms peritectically, the film has to be deposited from Sn solutions and the excess Sn removed by chemical methods.

Amorphous Film Formation by Vacuum Evaporation

An Edwards automatic evaporator, Model 12EA, is used for the vacuum deposition. The unit consists of a 12" bell jar with a 4" oil (D.C. 704) diffusion pump which pumps down to 2×10^{-5} torr in about an hour. Ultimate vacuum with the present Peltier thermoelectric cooled baffle is 5×10^{-7} torr after eight hours of continuous pumping. The system is fitted with a low voltage, high current power supply for resistive heating of boats and filaments. Several different sample holders have been tried including tungsten shallow dimple boats, filament heated alumina crucibles and deep tungsten baskets. The latter has been most successful in producing films with minimal loss of material from jumping out of the boat. It also provides the most directed vapor transport towards the substrate.

Recently a quartz lamp heater was installed to heat the substrate prior, during, or after the deposition. A thermocouple is attached to the substrate to monitor the temperature. An accessory is on order which will permit ion cleaning of the substrate in vacuo prior to deposition. Also on order is a multiple turret deposition source which will allow successive deposition of four different materials without breaking vacuum. There is still no provision for monitoring the thickness of the film during deposition.

Fig. 1: Quartz apparatus for preparing amorphous thin films by dipping substrate into molten ternary compounds.

The general procedure for evaporation was as follows: A small amount of finely ground sample was placed in the heating element. The substitute in all cases was a pyrex slide which had been cleaned in an ultrasonic bath of ethanol, rinsed with acetone, and dried in air. The height of the substrate above the sample could be varied between 3" (low) and 5" (high). The slide was protected during the initial heating stages by a metal shutter. After mounting the sample and substrate, the bell jar was evacuated to less than 10^{-5} torr. Current was passed through the refractory metal. When the desired amount and pressure was reached, the shutter was opened and the material allowed to coat the bottom of the slide. For the results reported here, there was no control or monitoring of the temperature of the substrate during deposition. The detailed experimental conditions for different compounds is given below.

CdAs₂. A film of CdAs₂ was evaporated from a tungsten boat. After obtaining a pressure of 1×10^{-5} torr, the current was slowly raised to 55A over a period of 10 minutes. The shutter was opened and the slide coated at this current for 10 minutes.

CdSnAs₂. Two films were prepared from CdSnAs₂, using a tungsten boat. The same sample was used for both films. For the first film, the system was evacuated to a pressure of 4×10^{-6} torr and the current brought to 45A over a 5 minute period. The shutter was opened and the film allowed to form for 3 hours. The second film was formed by rapidly bringing the current to 50A when the pressure reached 5×10^{-6} torr. The evaporation continued at this current for 6 hours.

CdSiAs₂. Three films were prepared from a single sample of CdSiAs₂, using an Al₂O₃ crucible. For the first film, the current was brought to 10 amps over a 10 minute period with the pressure at 8×10^{-6} torr. The shutter was opened and evaporation proceeded for 1 hour. A second film was formed by immediately raising the current to 35A. After 5 minutes at this current, the shutter was opened and evaporation took place for 30 minutes at a pressure of 6×10^{-6} torr. The third film was deposited at a pressure of 1.5×10^{-5} torr. The current was brought to 45A over 15 minutes. The shutter was opened and current increased to 50A over the next 30 minutes. Evaporation continued for 45 minutes; however, only a small area of film formed on the substrate. It is presumed that the slide itself became too hot to allow the film to form, since the interior of the bell jar was well coated. A fourth film of CdSiAs₂ was prepared from a fresh sample of starting material. For this film, the substrate was raised to the higher position, about 5" above the source. The sample was contained in an Al₂O₃ crucible. The current was raised quickly in 2-3 minutes to 45A. As soon as the pressure reached 1.0×10^{-5} torr, the shutter was opened and evaporation proceeded for 2 hours.

ZnSiAs₂. A single film of ZnSiAs₂ was prepared. The homogeneity of the starting material was suspect even though the atomic emission results show relatively good stoichiometry. It was only partially reacted

during its synthesis from the starting elements because the ampoule was not held at the high temperature for a long enough time period. To prepare the film, the sample was contained in an Al_2O_3 crucible with the substrate held 5" above the source. The current was raised over 5 minutes, and the shutter was opened. Evaporation proceeded for 2 hours.

ZnAs₂. A number of films were made from polycrystalline ZnAs₂ which were obtained from Cerac with a stated purity of 99.99%. The conditions used for film production are given in Table 1. All samples were evaporated from a deep tungsten basket onto pyrex slides.

TABLE 1
SUMMARY OF EXPERIMENTAL CONDITIONS FOR PRODUCTION
OF FILMS FOR ZnAs₂

<u>Sample No.</u>	<u>Sample Composition</u>	<u>Heating Current (amp)</u>	<u>Heating Time (hrs)</u>	<u>Pressure during Deposition (torr x 10⁻⁵)</u>	<u>Substrate Position</u>
070479A	ZnAs ₂	55	0.5	1.25	Low
072479A	"	52	1.0	0.8	High
972679A	"	45	6.0	5.0	Low
070679A*	"	80	0.3	8.0	Low
072579A*	"	65	0.5	0.7	High

* No films were produced under these conditions.

III. RESULTS

The films produced from all the materials had a metallic mirror finish which generally adhered well to the substrate. They were insoluble in simple inorganic acids. There was no sign of deterioration of the material when stored in air, although some of the films did begin to peel off the glass substrate. Observation of the film prepared from CdAs_2 with a metallograph at 20X magnification showed many pits and striations in the film. At this stage, however, the physical properties of the film are of secondary importance vis-à-vis their chemical and structural characteristics.

Atomic emission was used to determine the elemental compositions of the films and estimate their thicknesses. The samples were prepared by dissolving the film in hot aqua regia and diluting with 1% HCl. The approximate film thicknesses were calculated from the weight of the dissolved material using an average density of 5 g/cm^3 . The results for the films produced from four starting compounds are given in Table 2 and from ZnAs_2 in Table 3.

Goryunova has shown that tetrahedral glasses retain enough short range order to provide characteristic infrared spectra (7). Also, glasses and crystals of the same material exhibit different spectra. Attempts were made to characterize some of the films on the basis of IR spectra. Since the films were on pyrex substrates, a second pyrex slide was placed in the reference beam. In most cases, the films were too thin to obtain any spectra. Sample No. 072679A, however, showed absorption bands at 750 and 420 cm^{-1} .

The X-ray diffraction pattern of Sample No. 072679A using $\text{MoK}\alpha$ radiation has no fine structure. The lower limit of X-ray diffraction is about $30\text{\AA}$ which is much less than the estimated thickness of the film (see Table 3). The indication, therefore, is that the film of this sample is amorphous.

TABLE 2
ELEMENTAL COMPOSITION AND CALCULATED FILM THICKNESS
OF FILMS DETERMINED BY ATOMIC EMISSION

<u>Starting Compound</u>	<u>Composition of Film</u>	<u>Approximate Film Thickness (μm)</u>
$\text{Cd}_{1.0}\text{As}_{2.3}$	$\text{Cd}_{1.0}\text{As}_{3.9}$	0.2
$\text{Cd}_{1.0}\text{Sn}_{0.8}\text{As}_{1.5}$	$\text{Cd}_{1.0}\text{Sn}_{2.8}\text{As}_{8.3}$	0.04
	$\text{Cd}_{0.5}\text{Sn}_{2.8}\text{As}_{0.43}^*$	0.07
$\text{Cd}_{1.0}\text{Si}_{1.2}\text{As}_{1.8}$	$\text{Cd}_{1.0}\text{Si}_{0.1}\text{As}_{0.3}$	0.2
	$\text{Cd}_{1.0}\text{Si}_{0.1}\text{As}_{1.8}$	0.3
	$\text{Cd}_{1.0}\text{Si}_{1.0}\text{As}_{1.3}$	insufficient material
	$\text{Cd}_{1.0}\text{Si}_{0.3}\text{As}_{0.4}$	0.05
$\text{Zn}_{1.0}\text{Si}_{0.3}\text{As}_{1.8}^{**}$	$\text{Zn}_{1.0}\text{Si}_{0.1}\text{As}_{0.1}$	0.11

* Only a trace of cadmium was detected.

** The sample may not have had a homogeneous stoichiometry

TABLE 3
ELEMENTAL COMPOSITION OF ZnAs_2 FILMS
DETERMINED BY ATOMIC EMISSION

<u>Sample No.</u>	<u>Original Composition</u>	<u>Thin Film Composition</u>	<u>Calculated Film Thickness (μm)</u>
070479A	ZnAs_2	ZnAs_2	0.5
072479A	ZnAs_2	$\text{ZnAs}_{0.7}$	0.09
072679A	ZnAs_2	$\text{ZnAs}_{1.4}$	0.6

IV. DISCUSSION

There are three primary controllable factors in vapor deposition: deposition rate, sample temperature, and substrate temperature. The deposition rate is controlled to a large extent by the heating rate. Substrate temperatures can be controlled by the distance from the heating source in the absence of an additional heating element.

Other variables to consider are the type of source holder and the length of the deposition time. Some source containers provide more directed vapor transport than others. For example, containers such as crucibles with high straight sides are superior to boats with shallow wells, which tend to deposit the material over a wider area. In theory, the longer the deposition time the thicker the film. However, other factors are affected by longer deposition times. If the substrate is hot enough, decomposition of the initially deposited film may occur on the substrate to produce films of changing stoichiometry. Another possibility is that during the longer times, the materials may undergo a change from amorphous to polycrystalline since the molecules may have time to order themselves into a preferred structure.

Most of the necessary data for evaluating a priori the best set of conditions, i.e., vapor pressure, decomposition temperature, etc., are unavailable for most of the compounds. Our initial attempts at vacuum deposition of the ternary compounds, therefore, were primarily of a trial and error nature to delineate the problems. The composition of the evaporated films for the compounds listed in Table 2 is decidedly different from the starting material. This result is consistent with what is known about the thermal decomposition of the compounds.

CdAs_2 has been shown to decompose to Cd_3As_2 and As_4 at temperatures below the melting point of CdAs_2 (8). Since the vapor pressure of Cd_3As_2 is much lower than As_4 , for example, 1 torr vs. 60 torr at 525°C , a higher concentration of As is expected in the film than in the original compound. If dissociation to the free elements occurred, the composition of the film would be close to that of the compounds, since As and Cd have almost the same vapor pressure. Indeed what is surprising is that there is not a higher concentration of As. A possible explanation is that the substrate temperature was sufficiently high that the As did not stick and was pumped away. If this process were totally efficient, however, only Cd_3As_2 would be left giving a film stoichiometry of $\text{CdAs}_{0.7}$. An additional complication is that Cd_3As_2 itself dissociates in the gas phase, although to a lesser degree than CdAs_2 (8).

CdSnAs_2 is a relatively well behaved ternary compound which melts congruently. Decomposition pressure as a function of temperature for the liquid state has been reported (9). The pressure is 28 torr at the melting point of 595°C , with the vapor phase probably consisting primarily of Cd and As since the vapor pressure of Sn is much lower than the other two elements. Our observed composition for the first film of $\text{Cd}_{1.0}\text{Sn}_{2.8}\text{As}_{8.3}$ has an unexpectedly large Sn to Cd ratio. This anomaly may be explained by loss of the Cd from the film initially formed because of prolonged heating of the substrate. Alternatively, phase separation may have occurred during heating or film formation with some SnAs being formed. This material has a lower vapor pressure than Cd and would lead to increased Sn/Cd ratios. The second film contained almost no cadmium indicating that the Cd has been lost from the sample in formation of the first film. The As ratio is also very low, probably for the same reason.

CdSiAs_2 melts peritectically at 830°C with extensive decomposition. The decomposition pressure is 400 torr at its melting point (9). At the low current (low temperature) conditions used for deposition of the first two films, no Si was deposited as expected in view of the low vapor pressure of Si. A significant fraction of Si was found only in the film produced at the highest temperature. At this temperature, however, relatively little film deposit was produced on the substrate. The near agreement, therefore, between the composition of the starting material and the third film must be regarded with suspicion. The experiment with the new material to produce the fourth film confirmed the general results of the first two depositions that the films are produced with low proportions of silicon.

The higher melting point of ZnSiAs_2 might be expected to favor production of films with stoichiometries more nearly similar to the starting material. The dissociation pressure at the melting point, however, is one atmosphere higher than the other two ternary compounds (9). This vapor probably consists largely of Zn and As and it is understandable, therefore, that the film produced is deficient in Si. The low As proportion may be due to loss of the initially deposited arsenic during the evaporation period or may just reflect the poor homogeneity of the starting material.

A more detailed investigation of the parameters affecting formation of ZnAs_2 films was made. Nesmeyanov (10) studied the vapor pressure of ZnAs_2 at temperatures below 400°C . ZnAs_2 does not dissociate in the vapor phase below this temperature. In a later report, Lyons (11) showed that between 600 – 800°C considerable thermal decomposition occurs according to the reaction

where the primary constituent of the vapor is As_4 . To achieve stoichiometric films, deposition must occur at temperatures less than 400°C . At

this temperature the ZnAs_2 vapor pressure is 2.8×10^{-3} torr, so deposition should occur quickly.

As seen in Table 3, the stoichiometry of the ZnAs_2 films is similar to the starting material, especially for the thicker films. Furthermore, the X-ray analysis of one of these films indicated that it was amorphous material. To the best of our knowledge, these results are the first time that thin films of amorphous ZnAs_2 have been produced. These films will provide the initial samples for evaluation of optical and electrical properties of amorphous materials. In addition, we will continue to improve the methods of producing the films and attempt to develop various procedures for incorporating the group IV elements. Particular emphasis will be placed on silicon since the band gap for crystalline ZnAs_2 is 0.93 eV, while that for ZnSiAs_2 is 1.74 eV. Addition of Si, therefore, should lead to an increase in the band gap as observed for incorporation of Ge in CdAs_2 (6,12) for better optimization of overlap with the solar spectrum.

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