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BROWN UNIV PROVIDENCE RI DEPT OF CHEMISTRY F/G 20/3  
PREPARATION AND PHOTOELECTRONIC PROPERTIES OF THE SYSTEM CD SUB--ETC(U)  
MAY 80 V S NGUYEN, R KERSHAW, K DWIGHT N00014-77-C-0387

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| 4. TITLE (and Subtitle)<br>Preparation and Photoelectronic Properties of the System $Cd_2Ge_{1-x}Si_xO_4$                                     |                                     | 5. TYPE OF REPORT & PERIOD COVERED<br>Technical                           |
|                                                                                                                                               |                                     | 6. PERFORMING ORG. REPORT NUMBER<br>11                                    |
| 7. AUTHOR(s)<br>V. S. Nguyen, R. Kershaw, K. Dwight and A. Wold                                                                               |                                     | 8. CONTRACT OR GRANT NUMBER(s)<br>N00014-77-C-0387                        |
| 9. PERFORMING ORGANIZATION NAME AND ADDRESS<br>Professor Aaron Wold<br>Brown University, Dept. of Chemistry<br>Providence, Rhode Island 02912 |                                     | 10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS<br>NR-359-653 |
| 11. CONTROLLING OFFICE NAME AND ADDRESS<br>Dr. David Nelson, Code 472<br>Office of Naval Research<br>Arlington, VA                            |                                     | 12. REPORT DATE<br>May, 1980                                              |
|                                                                                                                                               |                                     | 13. NUMBER OF PAGES<br>24                                                 |
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|                                                                                                                                               |                                     | 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE                                |

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18. SUPPLEMENTARY NOTES

Submitted to Journal of Solid State Chemistry

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Photoelectrolysis  
Stability  
Semiconductor  
Quantum Efficiency

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

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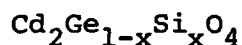
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9 Technical Report #1

6 PREPARATION AND PHOTOELECTRONIC PROPERTIES OF THE SYSTEM



SOLUBLE IN  $\text{H}_2\text{O}$  +  $\text{SiO}_2$  +  $\text{O}_2$  +  $\text{H}_2$

14 Van Son/Nguyen, Robert/Kershaw/ Kirby/Dwight, and Aaron/Wold

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# ABSTRACT

Members of the system  $\text{Cd}_2\text{Ge}_{1-x}\text{Si}_x\text{O}_4$  where  $0 \leq x \leq 0.4$  have been prepared. These compounds were observed to crystallize with the olivine structure, space group Pbnm. The resistivity, Hall mobility, flat-band potential, band gaps, and stability were determined as functions of composition. The variation of these photoelectronic properties can be attributed to the reduction of the cell parameters with increasing silicon substitution. The resulting increase in stability is dramatic.

## INTRODUCTION

There have been several excellent review papers which have summarized recent investigations concerning the photoelectrolysis of water using n-type semiconducting transition metal oxides (1,2). The oxides  $\text{TiO}_{2-x}$  and  $\text{WO}_{3-x}$  (3,4) were shown to have low electron mobilities ( $\leq 0.1 \text{ cm}^2/\text{v. sec}$ ) and high carrier concentrations ( $\sim 10^{19}/\text{cm}^3$ ). The conductivity of other metal oxides, e.g. antimony doped tin (IV) oxide, (5), is due to fewer carriers ( $\sim 10^{18}/\text{cm}^3$ ) with high mobility ( $\sim 20 \text{ cm}^2/\text{v. sec}$ ).

Another example of an oxide which has a high mobility is  $\text{Cd}_2\text{GeO}_4$ . This compound is more stable as an electrode in aqueous solution than  $\text{Cd}_2\text{SnO}_4$  (7). Since the stability is increased by the substitution of germanium for tin, it may be possible to further increase the stability of the electrode in aqueous solution by the preparation of photoanodes from members of the series  $\text{Cd}_2\text{Ge}_{1-x}\text{Si}_x\text{O}_4$ . In addition, the effect of such substitution on the flat band potential and band gap can also be determined.

### SYNTHESIS AND EXPERIMENTAL TECHNIQUES

Samples of  $\text{Cd}_2\text{Ge}_{1-x}\text{Si}_x\text{O}_4$  ( $x = 0.0, 0.2, 0.4$ ) were prepared by the solid state reaction of appropriate quantities of  $\text{GeO}_2$ , (Johnson Matthey, spec. pure),  $\text{CdO}$  (obtained from the decomposition of  $\text{CdCO}_3$ ) and  $\text{SiO}_2$  (obtained by the hydrolysis of  $\text{SiCl}_4$ ). Cadmium carbonate was subjected to thermogravimetric analysis; the weight of cadmium oxide obtained from the thermal decomposition of cadmium carbonate at  $320^\circ\text{C}$  agreed with the calculated value up to  $825^\circ\text{C}$ , but then decreased. As a result of this study, the reaction temperature for the formation of the cadmium compounds is maintained below  $825^\circ\text{C}$  in order to prevent loss of  $\text{CdO}$ . The silicon dioxide was obtained by the hydrolysis of silicon (IV) chloride with the formation of silica gel which was then heated at  $800^\circ\text{C}$  for 48 hours, under vacuum, in order to remove all of the water.

Finely ground mixtures of appropriate amounts of each starting material were heated in evacuated silica tubes at  $800^\circ\text{C}$ . The samples were removed from the furnace and reground thoroughly twice during the heat treatment, which was carried out for a total of 144 hours. Completed reactions were confirmed by x-ray analysis using a Philips Norelco Diffractometer with  $\text{CuK}\alpha$  radiation ( $\lambda = 1.5405\text{\AA}$ ) and at a scan rate of  $0.25^\circ 2\theta \text{ min}^{-1}$ .

Disks of each composition were prepared by pressing aliquots of approximately 200 mg of the product at 60,000 p.s.i. and then heating the green disks in evacuated silica tubes at  $800^\circ\text{C}$  for 72 hours. Well-sintered disks, off-white in color, were obtained by this method despite the low sintering temperature necessitated by the possible loss of  $\text{CdO}$  above  $825^\circ\text{C}$ .

#### PHOTOELECTRONIC PROPERTIES

Seebeck and D. C. Hall measurements showed all of the compounds to be n-type semiconductors; the carrier concentrations ( $n$ ), mobilities ( $\mu$ ) and resistivities ( $\rho$ ) of each compound are reported in Table I. Resistivity measurements were made on representative disks of each composition using the van der Pauw method (8). Indium electrical contacts were applied to each disk by ultrasonic soldering, and ohmic behavior was verified. The thermal variation of resistivity yields an activation energy of 0.017(2) eV for  $\text{Cd}_2\text{Ge}_{1-x}\text{Si}_x\text{O}_4$  ( $x = 0.0, 0.2, 0.4$ ).

Photoanodes were prepared by evaporating thin films of gold onto the back of the disks in order to provide good electrical contact. Anode assemblies were fabricated by first using indium metal to solder these electrodes to platinum wires which had been sealed in small pyrex tubes, and then coating all but the front surface with an electrically insulating resin (Microstop, Michigan Chrome and Chemical Company). Care was taken not to disturb the photoactive surfaces. For photoresponse measurements, these anodes were mounted in a small glass cell described previously (9). The anode was placed in 8 mm from the quartz window. A platinized platinum cathode ( $2.5 \text{ cm}^2$  in area) was mounted 2 cm behind and below the anode and a saturated calomel reference electrode (SCE) above it. The cell was filled with 100 ml of 0.2 M sodium acetate solution which was adjusted to a pH of 13 by the addition of sodium hydroxide. This electrolyte was purged of dissolved oxygen and stirred by means of a continuous stream of 85% Argon-15% Hydrogen gas. A cathode potential of approximately -0.99 V vs S.C.E. was used as

an indication of complete purging of the cell (9).

A quartz lens was used to focus the light from a 150 watt Xenon arc onto an area of 2.25 mm in diameter, and a 1.0 neutral density filter (Oriel Optic Corp.) was used to reduce the irradiation to approximately 40 mW of total power. A monochromator (Oriel Model 7240) with 1 mm slits was used for the spectral studies, and a calibrated silicon photodiode (United Detector Technology) was used to measure the light flux incident on the cell. Anodic bias was applied via a voltage follower having an output impedance of less than 0.1  $\Omega$  and the resulting photoresponse was measured with a current amplifier which inserted a negligible potential drop (less than 1 $\mu$ V/ into the external circuit).

## RESULTS AND DISCUSSION

Tarte (10) reported that  $\text{Cd}_2\text{GeO}_4$  crystallizes with the olivine structure, space group Pbnm. In this structure, independent  $\text{GeO}_4$  tetrahedra are linked to  $\text{CdO}_6$  octahedra by sharing corners and edges. Two crystallographically independent Cd atoms are both octahedrally surrounded by six oxygens; these octahedra share edges and corners with both  $\text{GeO}_4$  tetrahedra and adjacent octahedra. The oxygen atoms form a slightly distorted hexagonal close packed array with the hexagonal axis in the a direction. The cations occupy some of the tetrahedral and octahedral interstices. This structure is shown in Figure 1. A complete discussion of the olivine structure has been reported by Hanke (11).

$\text{Cd}_2\text{SiO}_4$  crystallizes with the  $[\text{Na}_2\text{SO}_4\text{V}]$  structure (room temperature form of  $\text{Na}_2\text{SO}_4$ , space group Fddd). The structure consists of nearly regular and isolated  $\text{SiO}_4$  tetrahedra, which are held together by irregular  $\text{CdO}_6$  octahedra with each  $\text{SiO}_4$  tetrahedra sharing two opposite edges with two  $\text{CdO}_6$  octahedra (12).  $\text{Cd}_2\text{SnO}_4$  crystallizes with the  $[\text{Sr}_2\text{PbO}_4]$  structure, space group Pbam. In this structure the edge-shared  $\text{SnO}_6$  octahedra extend in chains in the c-direction. These chains are held together by  $\text{CdO}_7$  polyhedra (13).

The x-ray diffraction patterns for  $\text{Cd}_2\text{Ge}_{1-x}\text{Si}_x\text{O}_4$  ( $x = 0.0, 0.2, 0.4$ ) indicate that these compounds retain the olivine structure. Table I summarizes the crystallographic data for members of the system which were prepared as single phases and studied. Attempts to prepare compounds where

$x > 0.4$  gave multi-phase products.

The variations of photoresponse with anode potential obtained for the different samples prepared above are shown in Figure 2. Values for the flat-band potentials (relative to SCE) were consistently determined from the onset of photocurrent, and are given in Table I. Although there may be some uncertainty in each of these values, nevertheless, the existence of a progressive change with composition is evident from Figure 2.

The spectral response curves for these samples are shown in Figure 3. It is apparent that in this system, the substitution of silicon for germanium increases the quantum efficiency (obtained by dividing the observed photocurrent of the incident light flux) over a range of wavelengths from 300 to 340 nm. This increase in efficiency is accompanied by an increase in the optical band gaps, which are given in Table I. These values were determined from the spectral response data by using the analysis technique previously reported (4). In addition to the values for this indirect band gap, the analysis yields values for a high-energy, direct band gap in the neighborhood of 4.2 eV.

The variation of relative photocurrent with time is shown in Figure 4. The olivine  $\text{Cd}_2\text{GeO}_4$  is considerably more stable than  $\text{Cd}_2\text{SnO}_4$ , which loses 25% of its photocurrent in the first minute (7), and a further pronounced increase in stability is obtained from the substitution of silicon for germanium in  $\text{Cd}_2\text{Ge}_{1-x}\text{Si}_x\text{O}_4$ . The unsubstituted material loses 25% of its output in the first hour and 35% in the first five hours, compared with 5% and 9%, respectively, for  $\text{Cd}_2\text{Ge}_{0.6}\text{Si}_{0.4}\text{O}_4$ . Although off scale in Figure 4,

the data for  $\text{Cd}_2\text{Ge}_{0.6}\text{Si}_{0.4}\text{O}_4$  extends for an additional fifteen hours and suffers a further decrease of only 1%.

From the above, it appears that stabilization is obtained by the substitution of tetrahedral silicon (IV) ( $0.26\text{\AA}$ ) for tetrahedral germanium (IV) ( $0.39\text{\AA}$ ). The results of x-ray analysis and the preference of silicon (IV) for tetrahedral sites establish that the series  $\text{Cd}_2\text{Ge}_{1-x}\text{Si}_x\text{O}_4$  ( $0 \leq x \leq 0.4$ ) all possess the same olivine structure. It would not be unreasonable to attribute an increase in stability to the strengthening of bonds arising from the decrease in cell parameters given in Table I. This strengthening can also increase the band gap and decrease the flat band potential, as observed.

## SUMMARY AND CONCLUSION

Single-phase samples of  $\text{Cd}_2\text{Ge}_{1-x}\text{Si}_x\text{O}_4$  ( $x = 0.0, 0.2, 0.4$ ) were prepared from  $\text{CdO}$ ,  $\text{SiO}_2$  and  $\text{GeO}_2$  which, in turn, were carefully prepared so as to ensure the greatest possible degree of stoichiometry. It was determined that  $\text{CdO}$  loses weight above  $825^\circ\text{C}$ , and consequently all reactions and sinterings were carried out at  $800^\circ\text{C}$ . X-ray analysis showed all the samples to possess the olivine structure. the cell parameters decreasing with increasing substitution of silicon for germanium.

Measurements were made of the resistivity, Hall effect, and such photoelectronic properties as the current-voltage response, spectral response, and stability. The results listed in Table I follow a systematic variation with composition.

Silicon, prefers tetrahedral coordination and its substitution for germanium in  $\text{Cd}_2\text{Ge}_{1-x}\text{Si}_x\text{O}_4$  presents no anomalies. The variation of the observed properties with composition for this system can be attributed to the decrease in cell dimensions, which increases the strength of chemical bonding. The observed increase in stability is dramatic; from a 25% loss in one hour for  $x = 0.0$  to only a 6% loss in twenty-two hours for  $x = 0.4$ .

$\text{Cd}_2\text{GeO}_4$  maintains the olivine structure for substitution of up to forty atomic percent silicon for germanium. It is the first reported oxide system where such substitutions can modify the fundamental photoelectronic properties of flat-band potential, band gap and stability.

#### ACKNOWLEDGEMENTS

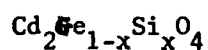
The authors would like to acknowledge the support of the Office of Naval Research, Arlington, Virginia for the support of Van Son Nguyen and Kirby Dwight.

The authors would also like to acknowledge the Solar Energy Research Institute, Golden, Colorado as well as the Materials Research Laboratory Program at Brown University for their support.

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TABLE I



| $n_x$                                       | $\text{Si}_{0.0}$        | $\text{Si}_{0.2}$       | $\text{Si}_{0.4}$        |
|---------------------------------------------|--------------------------|-------------------------|--------------------------|
| a (Å)                                       | 5.20(1)                  | 5.18(1)                 | 5.15(1)                  |
| b (Å)                                       | 11.14(1)                 | 11.13(1)                | 11.10(1)                 |
| c (Å)                                       | 6.57(1)                  | 6.57(1)                 | 6.55(1)                  |
| Resistivity ( $\Omega\text{-cm}$ ) R.T.     | 0.20(2)                  | 0.60(5)                 | 1.60(5)                  |
| Carrier Concentration ( $\text{cm}^{-3}$ )  | $1.05(5) \times 10^{18}$ | $5.1(2) \times 10^{17}$ | $2.85(5) \times 10^{17}$ |
| Mobility ( $\text{cm}^2/\text{V-sec}$ )     | 30(3)                    | 21(2)                   | 14.0(2)                  |
| Flat-band potential<br>(V vs SCE @ pH = 13) | -0.80(5)                 | -0.85(5)                | -1.05(5)                 |
| Optical Band Gap (eV)                       | 3.15(5)                  | 3.25(5)                 | 3.35(5)                  |
| Direct Band Gap (eV)                        | 4.15(5)                  | 4.20(5)                 | 4.45(5)                  |
| $I/I_0$ after 1 hour                        | 0.75                     | 0.90                    | 0.95                     |
| $I/I_0$ after 5 hours                       | 0.65                     | 0.88                    | 0.91                     |

FIGURE CAPTION SHEET

- Figure 1        -        The olivine structure
- Figure 2        -        Photoresponse under "white" xenon arc irradiation  
of  $1.0 \text{ W/cm}^2$  in pH  $\approx$  13 electrolyte.
- Figure 3        -        Quantum efficiency measured at an anode potential  
of -0.2 V vs SCE in pH  $\approx$  13 electrolyte.
- Figure 4        -        Stability under "white" xenon arc irradiation of  
 $1.0 \text{ W/cm}^2$  in 0.2 M sodium acetate adjusted to  
pH  $\approx$  13.

# The $A_2 BX_4$ Structure

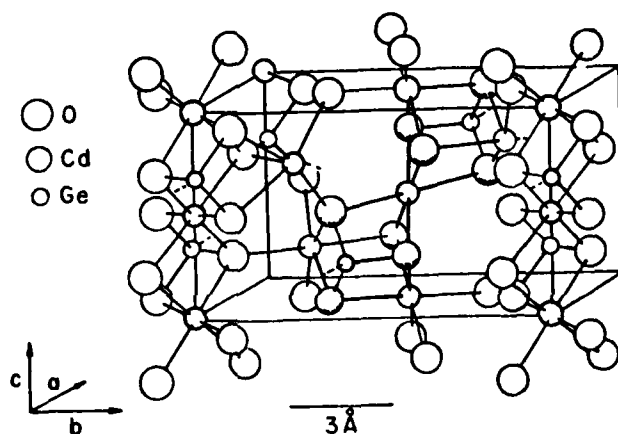
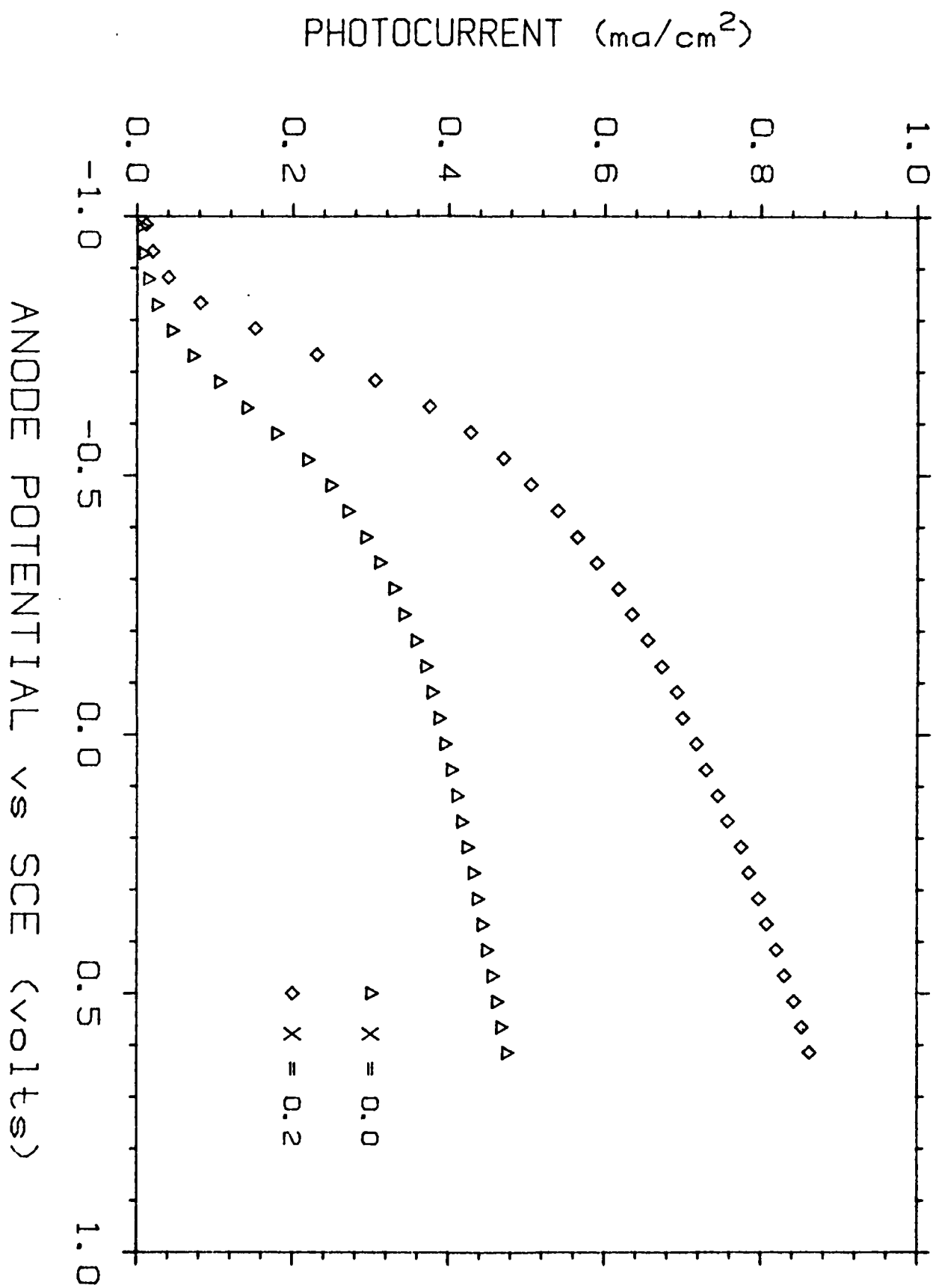
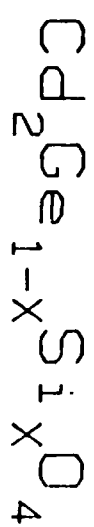
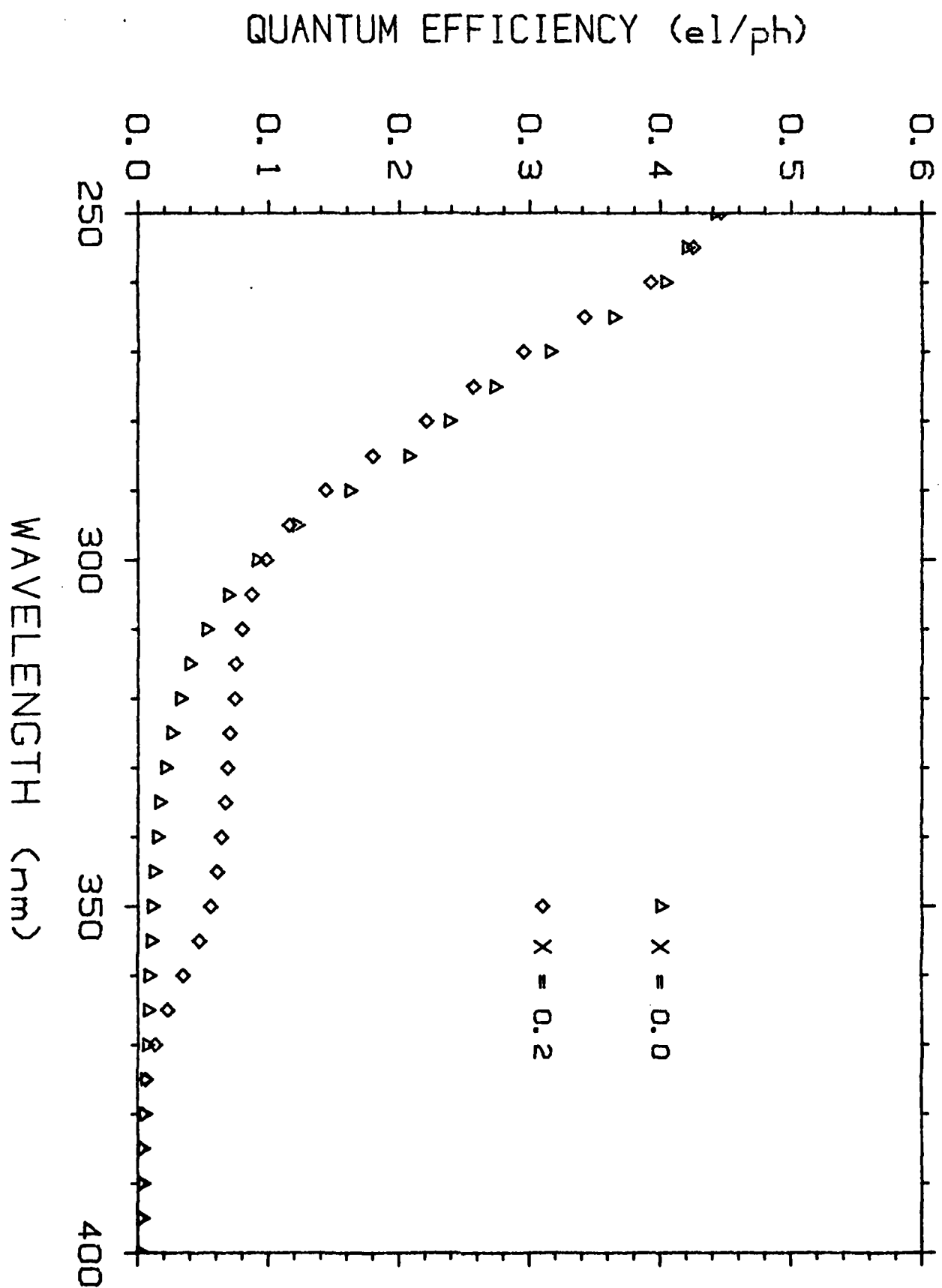


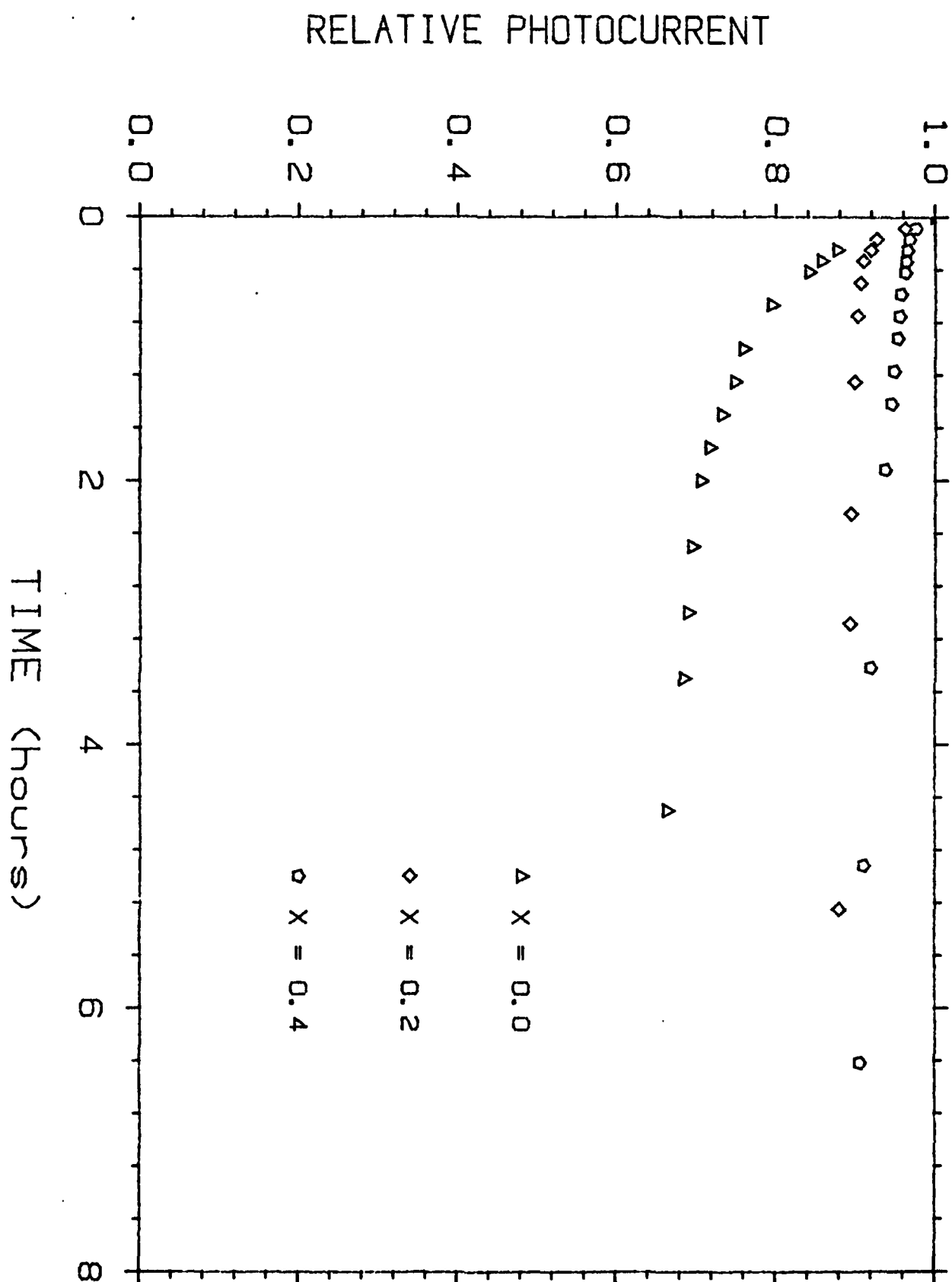
FIG. 1 The Olivine Structure  $Cd_2GeO_4$ . Note the edge-sharing of the distorted  $CdO_6$  Octahedra.



# $\text{Cd}_2\text{Ge}_{1-x}\text{Si}_x\text{O}_4$ SPECTRAL RESPONSE



# STABILITY of $\text{Cd}_2\text{Ge}_{1-x}\text{Si}_x\text{O}_4$



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