

## Cross diffusion of Cd and Zn in Cu<sub>2</sub>S formed on Zn x Cd<sub>1-x</sub> S thin films

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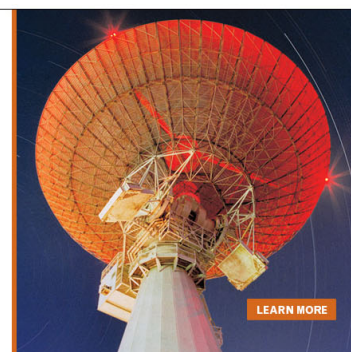
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# Cross diffusion of Cd and Zn into Cu<sub>2</sub>S formed on Zn<sub>x</sub>Cd<sub>1-x</sub>S thin films

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Cadmium and zinc compositions in Cu<sub>2</sub>S formed on Zn<sub>x</sub>Cd<sub>1-x</sub>S films ( $0 < x \leq 0.25$ ) by means of ion exchange have been measured using Auger Electron Spectroscopy (AES), Atomic Absorption Spectroscopy (AAS), and Electron Spectroscopy for Chemical Analysis (ESCA). Net concentrations of Cd and Zn in as-formed Cu<sub>2</sub>S are generally in the  $10^{18}$ – $10^{19}$  cm<sup>-3</sup> range. Heat treatments in both oxidizing and reducing ambients raise the concentrations by over an order of magnitude, with the Zn concentrations increasing more so than those of Cd. Large increases in Zn at or near the Cu<sub>2</sub>S surface were measured subsequent to heat treatment, accompanied by increased oxygen. Following heat treatments, Cd and Zn concentrations in the Cu<sub>2</sub>S "bulk" are found to be less than  $10^{19}$  and  $10^{20}$  cm<sup>-3</sup>, respectively, for all substrate compositions used.

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## I. INTRODUCTION

The highest efficiency thin film solar cells reported are the Cu<sub>2</sub>S/Zn<sub>x</sub>Cd<sub>1-x</sub>S type.<sup>1</sup> These are more efficient than Cu<sub>2</sub>S/CdS junctions due to increased light generated voltage. Light generated currents, however, are usually somewhat less.

It has not been conclusively demonstrated why the currents are less. Various possible current degrading mechanisms for the Cu<sub>2</sub>S/ZnCdS junction have been reviewed,<sup>2</sup> and include a potential spike at the interface,<sup>3,4</sup> decreased electric field and electron mobility in the ZnCdS,<sup>5</sup> poor grain structure of the ZnCdS which feeds through into the Cu<sub>2</sub>S,<sup>6</sup> and excess Zn and Cd impurities in the Cu<sub>2</sub>S. It is the latter area that is addressed in this investigation.

The role of Cd in Cu<sub>2</sub>S and the effect it has on the Cu<sub>2</sub>S/CdS cell have not been established. It is known that copper diffuses into CdS during junction optimization,<sup>7</sup> and this has been incorporated into cell models.<sup>8,9</sup> The presence of Cd and Cd gradients existing in the Cu<sub>2</sub>S has been reported, although for quite thick layers formed on single crystal CdS.<sup>10,11</sup>

AES and AAS studies of Cu<sub>2</sub>S/ZnCdS junction composition have been reported,<sup>12</sup> although these junctions were not heat treated. Measurements of [Cd] and [Zn] in Cu<sub>2</sub>S formed on ZnCdS have, to our knowledge, not been reported in the open literature.

We would like to report measurements of [Zn] and [Cd] in Cu<sub>2</sub>S layers formed on ZnCdS films, before and after heat treatments. Compositional techniques include Auger Electron Spectroscopy (AES) and Atomic Absorption Spectroscopy (AAS). Sheet resistance and electron spectroscopy for chemical analysis (ESCA) measurements were also made on some samples, and will be noted where appropriate.

## II. EXPERIMENTAL TECHNIQUES

The majority of results discussed below pertains to Cu<sub>2</sub>S films formed by aqueous ion exchange on Zn<sub>x</sub>Cd<sub>1-x</sub>S substrates with  $0 < x \leq 0.25$ , obtained from the University of Delaware. Cu<sub>2</sub>S formation conditions were: 6 g/l of CuCl<sub>2</sub>, 2 g/l of NaCl, pH = 3,  $T = 99 \pm 1^\circ\text{C}$ , time 10–30 sec. (Cu<sub>2</sub>S

thicknesses are noted below in the discussion of results.) Several Cu<sub>2</sub>S films formed by the solid state reaction of CuCl were also measured.

Auger measurements were performed on Physical Electronics models 595<sup>13</sup> and 550<sup>14</sup> Scanning Auger microprobes. Survey spectra and some depth profiles by means of Ar<sup>+</sup> milling were taken. First derivative signals for the following elements were monitored, at indicated Auger electron energies: Cu (920 eV), S (152 eV), Cd (376 eV), Zn (994 eV), O(510 eV), C (272 eV) and Cl (181 eV).

AAS measurements were performed on Perkin-Elmer models 703 and 460 spectrophotometers. Analyses were performed for Cd (at 228.2 nm), for Zn (at 213.9 nm) and for Cu (at 324.8 nm). Standards of Zn and Cd in Cu<sup>+</sup> and KCN solutions were measured for possible interference effects<sup>15</sup> and none was discernible.

Cu<sub>2</sub>S layers were removed by argon ion sputtering (ESCA and Auger measurements) and by 0.1 M KCN for the AAS measurements.

Heat treatments were performed either in an air oven or in a tube furnace with a 5% H<sub>2</sub>/N<sub>2</sub> ambient, at temperatures noted below.

## III. RESULTS AND DISCUSSION

For average Cu<sub>2</sub>S composition measurements, Cu<sub>2</sub>S was stripped off in 0.1 M KCN and measured for Cd (on CdS substrates), for Cd and Zn (ZnCdS substrates) and for Cu, from which the effective Cu<sub>2</sub>S thickness could be deter-

TABLE I. Net Cd and Zn concentration in Cu<sub>2</sub>S before and after heat treatments. Measured by AAS.

HT Condition	Substrate	[Cd] ( $10^{19}$ cm <sup>-3</sup> )		[Zn] ( $10^{19}$ cm <sup>-3</sup> )	
		No HT	HT	No HT	HT
Air, 175°C, 20 Min.	CdS	0.58	3.8	—	—
	Zn <sub>0.2</sub> Cd <sub>0.8</sub> S	2.4	10	3.1	18
H <sub>2</sub> /N <sub>2</sub> , 200°C, 22 h	CdS	1.2	4.5	—	—
	Zn <sub>0.2</sub> Cd <sub>0.8</sub> S	0.78	9.4	1.7	19

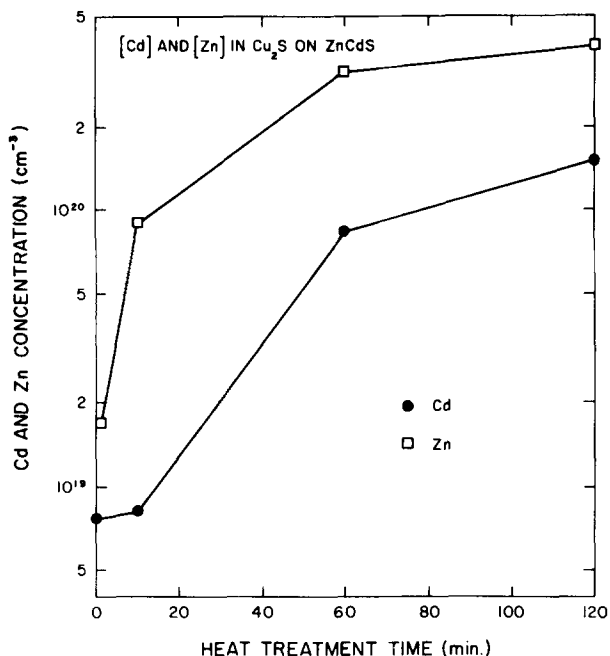


FIG. 1. Average Cd and Zn concentrations in  $\text{Cu}_2\text{S}$  formed on  $\text{Zn}_{0.22}\text{Cd}_{0.78}\text{S}$ .  $\text{Cu}_2\text{S}$  equivalent thickness was  $0.35 \mu\text{m}$ . Heat treatment was air at  $200^\circ\text{C}$ .

mined. Some representative data are shown in Table I, for before and after two types of heat treatment.

Increases in net [Cd] and [Zn] in  $\text{Cu}_2\text{S}$  for more extensive air heat treatments are seen in Fig. 1. An apparent saturation of [Cd] and [Zn] is seen, with the 20 minute values close to those seen in Table I. The net amount of Cd measured in  $\text{Cu}_2\text{S}$  formed on CdS also saturated at  $\approx 10^{20} \text{cm}^{-3}$  when air heat treated for more than 100 min.

[Zn] and [Cd] data determined by AAS for  $\text{Cu}_2\text{S}$  formed by solid-state reaction on CdS and  $\text{Zn}_{0.18}\text{Cd}_{0.82}\text{S}$  are shown in Table II, for air heat treatment at  $180^\circ\text{C}$ .

It is seen that similar ranges of Cd and Zn concentrations before and after heat treatments are measured for aqueous and solid state formed  $\text{Cu}_2\text{S}$ .

The following trends, exemplified by the above results, are apparent from AAS measurements: 1. Net Cd and Zn concentrations are generally in the  $10^{18}$ – $10^{19} \text{cm}^{-3}$  range in as-formed  $\text{Cu}_2\text{S}$ ; 2. Heat treatment (in either air or  $\text{H}_2/\text{N}_2$ ) increases the net concentrations by one to two orders of magnitude; 3. For  $\text{Cu}_2\text{S}$  formed on ZnCdS substrate, [Zn] is always greater than [Cd]; 4. [Zn + Cd] in  $\text{Cu}_2\text{S}$  formed on ZnCdS is always greater than [Cd] in  $\text{Cu}_2\text{S}$  formed on CdS.

TABLE II. Net Cd and Zn concentrations for solid state formed  $\text{Cu}_2\text{S}$ .  $\text{Cu}_2\text{S}$  thicknesses are  $\approx 0.6 \mu\text{m}$ .

Substrate	HT Time (min.)	[Cd] ( $10^{19} \text{cm}^{-3}$ )	[Zn] ( $10^{19} \text{cm}^{-3}$ )
CdS	0	0.64	—
	120	15	—
$\text{Zn}_{0.18}\text{Cd}_{0.82}\text{S}$	0	0.48	0.62
	10	0.45	5.8
	60	3.2	12
	120	7.1	12

TABLE III. ESCA peak intensity data for several elements. No HT = not heat treated; HT = heat treated;  $\text{Cu}_2\text{S}$  = not stripped; ZnCdS =  $\text{Cu}_2\text{S}$  stripped off.

Element (peak)	Sample			
	$\text{Cu}_2\text{S}$		ZnCdS	
	No HT	HT	No HT	HT
Cu 1 (934 eV)	12.3	7.6	—	4.1
Cu 2 (77 eV)	30.0	24.3	—	5.5
Cd (405 eV)	6.7	4.2	31.3	36.6
Zn (1022 eV)	2.3	11.1	14.5	3.1
S (165 eV)	13.0	11.3	38.9	33.8
O (165 eV)	13.2	14.4	15.3	16.8
C (285 eV)	22.4	27.1	—	—
Zn/Cd	0.34	2.6	0.46	0.09

The above Cd and Zn concentrations are averages computed by dividing the numbers of Cd and Zn atoms removed by the  $\text{Cu}_2\text{S}$  volume, and give no information about geometrical distribution in the  $\text{Cu}_2\text{S}$ . We have verified by AAS, AES, and ESCA measurements on different samples that surface segregation, of Zn in particular, occurs as a result of heat treatment.

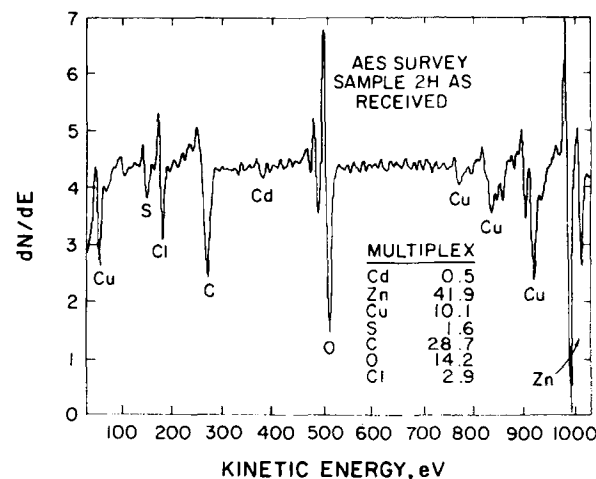
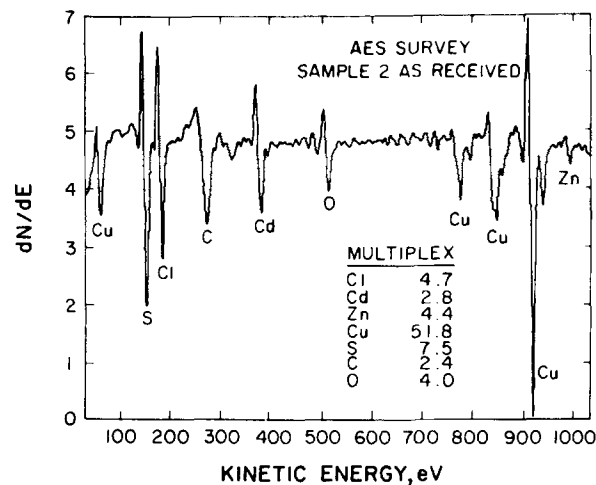


FIG. 2. Auger survey spectra for  $\text{Cu}_2\text{S}$  surfaces formed on ZnCdS. Sample 2: not heat treated; Sample 2H; heat treated (5%  $\text{H}_2/\text{N}_2$ , 18 h,  $200^\circ\text{C}$ ).

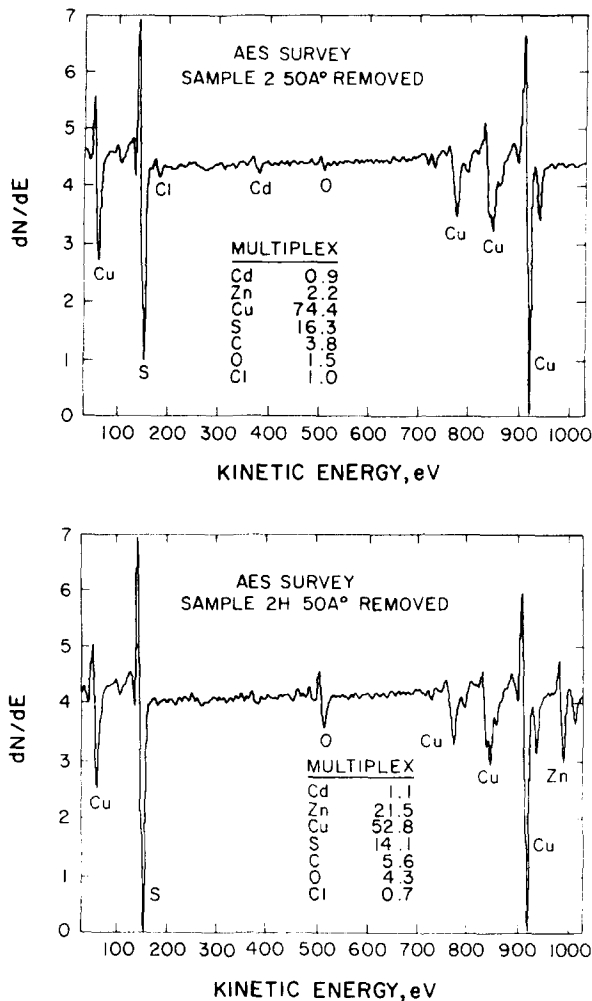


FIG. 3. Auger survey spectra for samples 2 (not heat treated) and 2H (heat treated) following a brief sputter etch.

For an ESCA study,  $\text{Cu}_2\text{S}$  was formed on a  $\text{Zn}_{0.16}\text{Cd}_{0.84}\text{S}$  substrate ( $\text{Cu}_2\text{S}$  thickness  $\approx 0.4 \mu\text{m}$ ). Half of the sample was air heat treated (10 min:  $200^\circ\text{C}$ ). These two samples were cut in half, and a piece of each stripped in 0.1 M KCN. Thus, four samples resulted, two with  $\text{Cu}_2\text{S}$  and two without. Each surface was analyzed. Pertinent intensity data (normal incident beam) are shown in Table III.

The following results are apparent from ESCA measurements: 1. The Zn signal on the  $\text{Cu}_2\text{S}$  surface increases by a factor of  $\approx 5$ ; the Cd signal decreases slightly as a result of heat treatment; 2. An enhanced Cu signal is seen on the ZnCdS surface that was heat treated prior to  $\text{Cu}_2\text{S}$  removal; 3. The Zn/Cd ratio (in terms of peak intensities) increased by a factor of  $\approx 7$  on the  $\text{Cu}_2\text{S}$  surface and decreased by a factor of  $\approx 5$  on the stripped ZnCdS surface subsequent to heat treatment.

Surface analysis with AES combined with  $\text{Ar}^+$  ion milling was used to corroborate these results. Examples showing the surface enhancement of Zn resulting from heat treatment (with slight decrease resulting for Cd) are seen from Figs. 2 to 5.

Figure 2 shows AES survey spectra for  $\text{Cu}_2\text{S}$  on ZnCdS ( $\text{Cu}_2\text{S}$  thickness  $\approx 0.3 \mu\text{m}$ ). The major effects of heat treat-

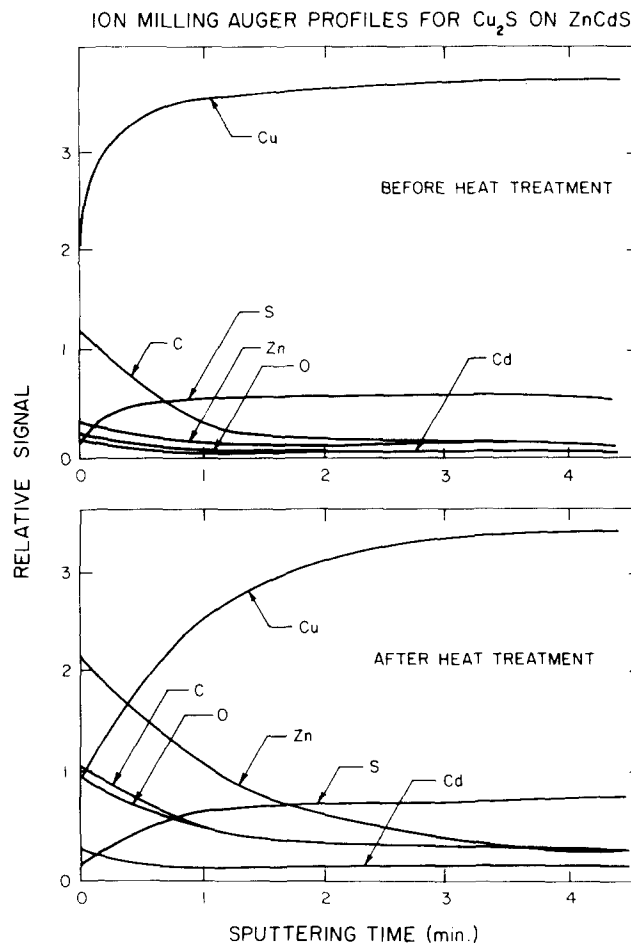


FIG. 4. Ion milling Auger profiles for  $\text{Cu}_2\text{S}$  on ZnCdS film substrate, showing effects of heat treatment (air, 10 min,  $200^\circ\text{C}$ ).

ment were to increase significantly the Zn and O signals, and decrease Cd. This indicates surface segregation of Zn to the exclusion of Cd, with the increased O signal probably resulting from sample exposure to air subsequent to heat treatment in  $\text{H}_2/\text{N}_2$ .

That the increase of Zn seen in Fig. 2 is basically a surface phenomenon is indicated in Figs. 3 and 4. For Fig. 3, the samples of Fig. 2 were briefly sputter etched, and AES survey scans repeated on the same locations. The large decreases in Zn and O for the heat treated sample (2H) subsequent to the sputter etch are readily apparent. Similar trends were seen for a second set of samples that were measured.

Cd, Zn, and O surface compositions (taken from survey scan multiplex data) for heat treated and non-heat treated samples, before and after the ion etch are shown in Table IV.

These results were verified by additional ion-milling profiles, as shown in Fig. 4. The major result of heat treatment is a buildup of Zn and O near the  $\text{Cu}_2\text{S}$  surface.

Thin film  $\text{Cu}_2\text{S}/\text{CdS}$  junctions formed by aqueous ion exchange have been traditionally difficult to sputter etch profile near the junction region due to their large deviations from planarity. In an effort to study the  $\text{Cu}_2\text{S}$  composition of such  $\text{Cu}_2\text{S}/\text{ZnCdS}$  junctions, we used the technique of sequentially etching off layers of  $\text{Cu}_2\text{S}$  in 0.1 M KCN and analyzing the solutions by AAS for Cu, Zn, and Cd. These

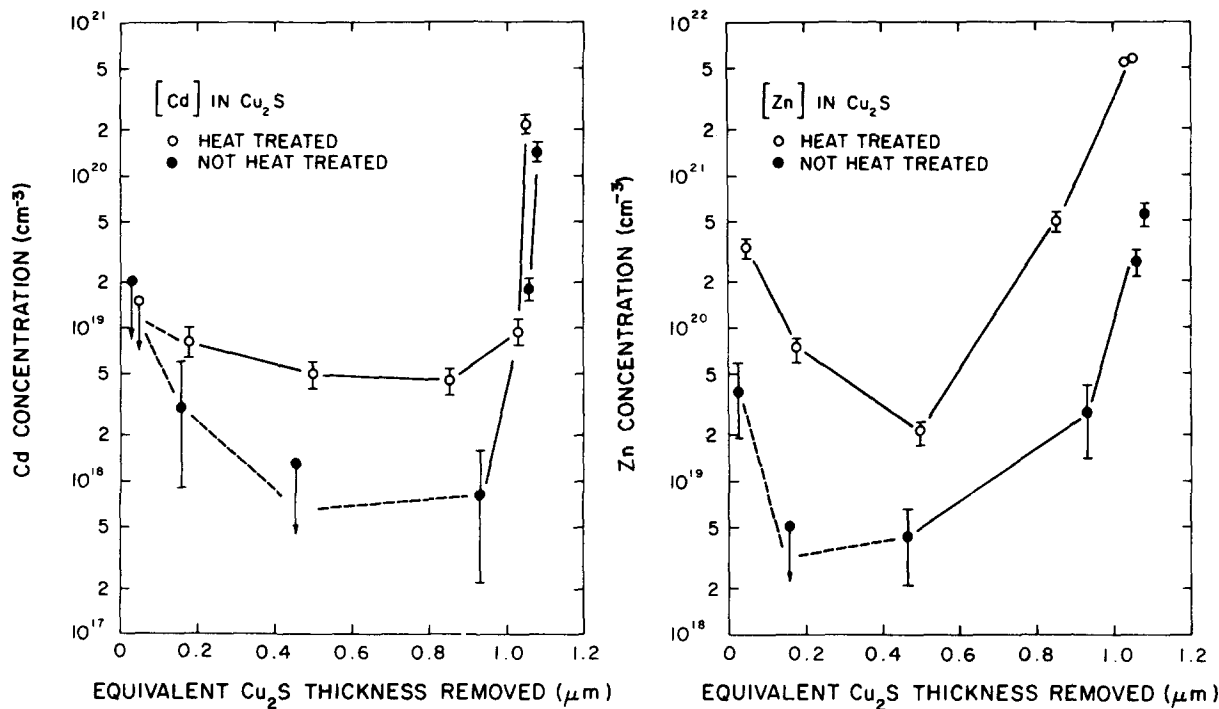


FIG. 5. Concentrations of Cd and Zn in  $\text{Cu}_2\text{S}$  formed on ZnCdS, sequentially etched in 0.1 M KCN and measured by AAS.

results are shown in Fig. 5, for heat treated and non-heat treated samples. The  $\text{Cu}_2\text{S}$  was formed thicker ( $\sim 1\ \mu\text{m}$ ) in an attempt to provide measurable amounts of Zn and Cd for the several AAS samples which were etched off. Even so, for several samples taken from un-heat treated films, there was insufficient Cd or Zn to accurately measure. (These latter points are designated by the vertical arrows in the figures.)

The results shown in Fig. 5 pertaining to at or near the  $\text{Cu}_2\text{S}$  surface are consistent with the AES and ESCA measurements. Note the near equality of  $[\text{Cd}]$  near the surface for heat treated and un-heat treated samples, and the inequality for  $[\text{Zn}]$ . The concentrations of Cd and Zn then decrease into the  $\text{Cu}_2\text{S}$ , for all samples, and subsequently increase.  $[\text{Zn}]$  exceeds  $[\text{Cd}]$  by roughly an order of magnitude.

The sequential removal of  $\text{Cu}_2\text{S}$  by etching in 0.1 M KCN occurs in a very non-uniform manner, if the  $\text{Cu}_2\text{S}$  was formed by aqueous ion exchange. It is known that such  $\text{Cu}_2\text{S}$  consists of a top "skin" and deep protrusions down grain boundaries.<sup>16</sup> When placed in KCN solution, the top skin etches first, with preferential attack occurring at grain boundaries immediately. The grain boundary attack and top skin removal occur simultaneously, until the top skin is entirely removed. Subsequent etching occurs only in the grain

boundaries, until that  $\text{Cu}_2\text{S}$  (which penetrates to a depth of several microns) is also removed. Examples of this etching chronology are shown in Fig. 6, and are presented here in order to aid interpretation of Fig. 5. Figure 6 and similar pictures, coupled with AAS determination of Cu amounts removed, indicate that about 50%–70% of the  $\text{Cu}_2\text{S}$  is in the grain boundaries, below the top surface. It is thus estimated that beyond a  $\text{Cu}_2\text{S}$  equivalent thickness of about  $0.5\ \mu\text{m}$  in Fig. 5, nearly all of the Cu is being removed from grain boundaries, and the  $[\text{Cd}]$  and  $[\text{Zn}]$  increases seen in Fig. 5 pertain to those regions.

Several other points related to Fig. 5 are of interest: 1. The general forms of  $[\text{Zn}]$  and  $[\text{Cd}]$  resemble those of  $[\text{Cd}]$  measured for  $\text{Cu}_2\text{S}/\text{CdS}$  cells by Rutherford backscattering,<sup>17</sup> although the concentrations shown in Fig. 5 are much smaller; 2. The increase in  $[\text{Cd}]$  near the  $\text{Cu}_2\text{S}$  surface has been reported for  $\text{Cu}_2\text{S}/\text{CdS}$  junctions via Auger measurements.<sup>18</sup> However,  $[\text{Cd}]$  values below the surface ( $< 10^{19}\ \text{cm}^{-3}$ , as seen in Fig. 5) are beyond reliable resolution of Auger measurements; 3. The sharp rise in  $[\text{Cd}]$  near the  $\text{Cu}_2\text{S}/\text{ZnCdS}$  interface is consistent with results reported for  $\text{Cu}_2\text{S}$  formed on CdS single crystals.<sup>10,11</sup> The  $[\text{Zn}]$  increase seen in Fig. 5 appears much more severe, extending over about half of the  $\text{Cu}_2\text{S}$  effective thickness. As noted above, this could be largely a grain boundary effect, apparently more extensive for  $[\text{Zn}]$  than for  $[\text{Cd}]$ . 4. The larger increase in  $[\text{Zn}]$  relative to  $[\text{Cd}]$  following heat treatment could be partly a result of Zn excess near the interface due to ion exchange kinetics.<sup>19</sup> It has been established that there is a thermally stimulated redistribution of Zn (see Table III). Any Zn excess at the interface that results from ion-exchange kinetics could act as an available diffusion source during subsequent heat treatments. This would not be so much the case for Cd.

TABLE IV. Compositions from AES multiplex data before and after (in parentheses) ion etch. Average of two samples taken.

	No HT	HT
Cd	2.3(0.8)	0.7(1.1)
Zn	6.0(4.2)	35.1(16.6)
O	3.4(1.5)	10.6(3.2)

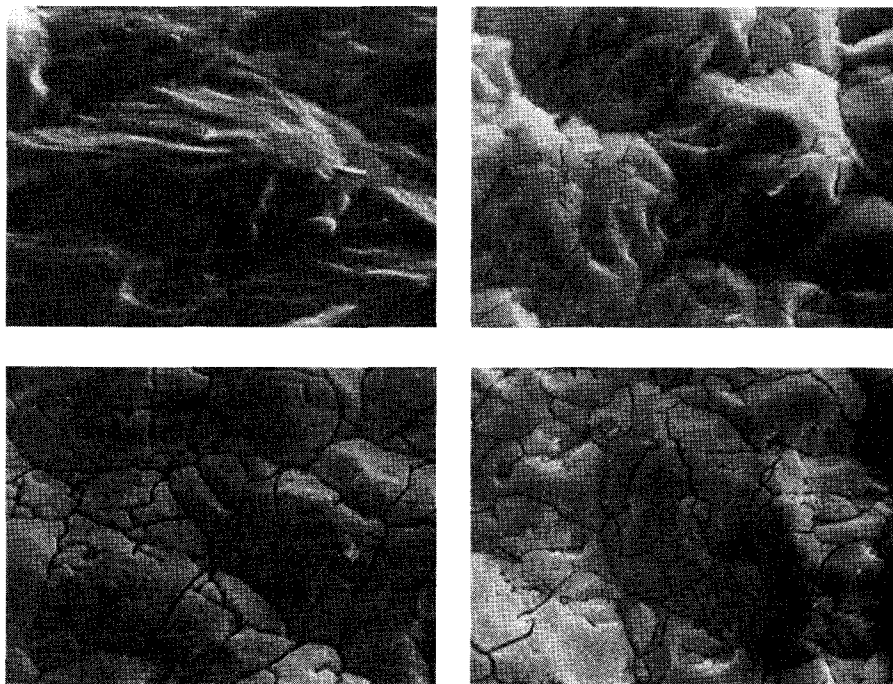


FIG. 6. Scanning electron micrographs of  $\text{Cu}_2\text{S}$  surface sequentially etched in 0.1 M KCN. Etch times and equivalent  $\text{Cu}_2\text{S}$  thicknesses are (clockwise from upper left): not etched; 30 sec.,  $0.10 \mu\text{m}$ ; 50 sec.,  $0.13 \mu\text{m}$ ; 90 sec.,  $0.31 \mu\text{m}$ .

The effects of the modified  $\text{Cu}_2\text{S}$  composition, due to the presence of Zn, on cell response is of interest. Gradients in Zn concentration through the  $\text{Cu}_2\text{S}$  will result in built-in electric fields if the Zn acts as a donor. For Zn segregation near the  $\text{Cu}_2\text{S}$  surface and the  $\text{Cu}_2\text{S}/\text{ZnCdS}$  interface, these fields should be such as to aid electron diffusion away from the  $\text{Cu}_2\text{S}$  bulk. This might be seen as a decrease and increase in the blue and red portions of the spectral response, respectively. However, we have performed sheet resistance measurements on  $\text{Cu}_2\text{S}$  films formed on CdS and ZnCdS, for various heat treatments. Even though the  $\text{Cu}_2\text{S}$  on ZnCdS sheet resistances are larger than for  $\text{Cu}_2\text{S}$  on CdS (after normalization for different thicknesses), it is by less than a factor of two. There are several possibilities to account for this: (a) Zn may be tied up with oxygen near the surface, and with sulfur near the interface; (b) a fraction of the Zn in  $\text{Cu}_2\text{S}$  could be electrically inactive, acting mainly as scattering centers. This would tend to offset the electrically active (donor) fraction; (c) the Zn could be electrically inactive in equilibrium, but act as minority carrier recombination centers when the junction is under illumination.

#### IV. CONCLUSIONS

For  $\text{Cu}_2\text{S}$  formed on  $\text{Zn}_x\text{Cd}_{1-x}\text{S}$  films by means of ion exchange, we have verified the following by means of AES, ESCA, and AAS: 1. Net [Cd] and [Zn] in  $\text{Cu}_2\text{S}$  are less than about  $10^{19} \text{ cm}^{-3}$  immediately after formation; 2. They increase into the  $10^{19}$ – $10^{20} \text{ cm}^{-3}$  range after heat treatment, with  $[\text{Zn}] > [\text{Cd}]$ ; 3. Profiling measurements indicate enhanced Zn and Cd at or near the surface and junction, with  $[\text{Zn}] > [\text{Cd}]$ ; 4. Increase of [Zn] in  $\text{Cu}_2\text{S}$  resulting from heat treatment is accompanied by a decrease in Zn content of substrate under the interface; 5. Increase in surface Zn is accompanied by increase in 0, but not of Cd; 6. [Cd] and [Zn] are less than  $10^{19} \text{ cm}^{-3}$  and  $10^{20} \text{ cm}^{-3}$  in  $\text{Cu}_2\text{S}$  "bulk" following heat treatment.

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