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**CuInSe₂-BASED PHOTOELECTROCHEMICAL CELLS: THEIR
USE IN CHARACTERIZATION OF THIN CuInSe₂ FILMS,
AND AS PHOTOVOLTAIC CELLS PER SE**

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ABSTRACT

Photoelectrochemistry has been employed to characterize the p-CuInSe₂ component of the CdS/CuInSe₂ photovoltaic cell. Using the junction between CuInSe₂ on-metal and a nonaqueous electrolyte containing a redox couple not specifically adsorbed onto the semiconductor, we can test the films for photovoltaic activity and obtain effective electronic properties of them, before CdS deposition, in a nondestructive manner. Electrochemical decomposition of CuInSe₂ was investigated in acetonitrile solutions to determine the mechanism of decomposition (n & p) in the dark and under illumination. Electrochemical, solution chemical and surface analyses confirmed at the light-assisted decomposition of CuInSe₂ resulted in metal ions and elemental chalcogen. On the basis of the results from the electrochemical decomposition, and studies on the solid state chemistry of the (Cu₂Se)_x(In₂Se₃)_{1-x} system and surface analyses, the CuInSe₂/polyiodide interface was stabilized and up to 11.7% conversion efficiencies were obtained.

INTRODUCTION

Single crystal and thin film CuInSe₂ - based solar cells have shown appreciable conversion efficiencies (>10%) and impressive output stabilities (1,2). The Cd(Zn)S/CuInSe₂ thin film solar cell is a multilayer device. Up to 6 layers are used in state-of-the-art thin film devices (1). The reproducible fabrication of the complete cell depends on the many factors that control the preparation of each of these layers. Up to now the photovoltaic quality of the layers could be assessed only by measuring the characteristics of the complete devices. Therefore, we developed a photoelectrochemical (PEC) system which allows tests of the photovoltaic activities of the individual CuInSe₂ on Mo films and layer patterns in a non-destructive way. Different redox electrolytes might need to be chosen due to different energetics and chemical behavior of the semiconductor.

Respectable performances were obtained also for n-CuInSe₂/polysulfide electrochemical photo-

voltaic cells (3). n-CuInS₂ and CuInSe₂, I-III-VI₂ materials with the chalcopyrite structure (4), have shown excellent output stabilities at reasonable conversion efficiencies, when used as single crystal photoanodes in aqueous polysulfide (5). It is apparent that kinetic factors limit the performance of this system. Therefore, we searched for a redox couple with fast kinetics as compared to the sulfide/polysulfide couple. The iodide/polyiodide is such a couple that has been used with elemental or binary semiconductor photoanodes (6). However, redox couples with electrochemical potentials positive of the sulfide/polysulfide couple generally do not protect low bandgap photoanodes from photodecomposition (7). Here we report on the stabilization of efficient n-CuInSe₂ photoanodes in aqueous polyiodide solution (8).

EXPERIMENTAL

p-CuInSe₂ thin films were prepared by multisource vacuum evaporation (1) on alumina substrates, that were metallized with a thin film of Mo. They were kindly supplied by R.A. Mickelsen (Boeing Engineering and Construction Co.) and by R. Axton (SERI). The films were about 3.3 μm thick and were deposited in two layers; the first (2.5 μm) at a substrate temperature of 350°C, and the second (0.8 μm) at 450°C. The p-CuInSe₂ crystals were grown by C. Rincon, and ohmic contact was made by evaporating Au onto one surface and using silver epoxy to connect the crystal to a copper wire. Except for one working face, the others were covered by epoxy (Devcon Corp., Danvers, Mass.) or sometimes by tar. Because the p-CuInSe₂ films were deposited on Mo, direct pressure contact or contact using Ag epoxy was made directly to the Mo. n-Type CuInSe₂ crystals, obtained from C. Rincon (9) and K. Bachmann (10), had 10¹⁶ - 10¹⁷ cm⁻³ majority carrier density, 600-850 cm²v⁻¹sec⁻¹ Hall mobilities and 1 - 10 Ω-cm resistivities. Most of the n-crystals were cut along the (112) plane. p-Type CuInSe₂ crystals were obtained from multicrystalline chunks, prepared by F.A. Thiel (A.T.&T. Bell Labs). They were cut along random directions, to isolate simple crystalline zones. They had an acceptor density of ca. 10¹⁸cm⁻³ and a resistivity of ca. 10⁻²Ω-cm. The stoichiometry of all crystals and thin films was checked by electron beam-induced wavelength-dispersive X-ray fluorescence, after calibrating this method by wet chemical analyses on some

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samples. All crystals were polished and ohmic contact was made with to one side with In-Ga alloy (n-type), or by evaporating Au (p-type). Except for one face, the crystals were covered by "Torr-Seal" epoxy.

A PAR Model 173 potentiostat with Model 175 Universal Programmer was used for voltammetric measurements in the dark and under illumination. The light source was a 150 W tungsten-halogen lamp. An H-cell with optical window was employed for the photoelectrochemical measurements. A helium-neon laser with scanning set-up (11) was used to test the spatial homogeneity of p-CuInSe₂ films. The experimental set-up for Mott-Schottky measurements was as described previously (12).

RESULTS AND DISCUSSION

Evaluation of Photovoltaic Activity

The primary purpose was to develop a diagnostic method for non-invasive evaluation of the photovoltaic quality of p-CuInSe₂ on Mo films, of the kind used for thin film solar cells. We chose to use acetonitrile containing Co(bpy)₃(ClO₄)₂ as the redox couple and tetraethylammonium perchlorate (TEAP) as supporting electrolyte (CAT). Using an outer sphere redox couple, such as this one, decreases the probability of specific adsorption, while use of a non-oxygenated organic solvent makes rapid surface oxidation less likely. In this way we could evaluate the effect of several surface treatments, the quality and homogeneity of films, and determine some effective optical and electronic properties of them. Such information was used for better control over, and improvement of preparation of the semiconductor films. For comparison, single crystals were studied, too.

In order to find out if the redox potentials of Co(bpy)₃^{3+/2+} and Co(bpy)₃^{2+/1+} fulfill the energetic requirements (hole [electron] transfer from n[p] type CuInSe₂, respectively) we used single crystals to determine the dark and light I-V characteristics, as we can expect minimal dark currents for such electrodes. Figure 1 (a,b), shows the dark and photo I-V curves of n-CuInSe₂ and p-CuInSe₂ crystals. The same on-set potential at ~ -0.2 V vs. SCE was obtained for n- and p-type crystals. Therefore, the semiconductors as used here are not ideal ones. However, the results indicate that the redox potential (~ -0.9 V vs. SCE) of Co(bpy)₃^{2+/+} is located within the bandgap of p-CuInSe₂ and the redox potential (~ 0.3 V vs. SCE) of Co(bpy)₃^{3+/2+} falls within the bandgap of n-CuInSe₂ (SCE: Standard Calomel Electrode, -0.24V vs. the normal hydrogen electrode).

Thin films of p-CuInSe₂ as obtained, showed very small photocurrents in the absence of the redox agent, Co(bpy)₃²⁺ (Fig. 2a). In the presence of the latter, significant photocurrent was observed which started at ~ -0.2 V vs. SCE (Fig. 2b). Fig. 2c shows the improved photovoltaic activity of the film after etching in Br₂/MeOH, with significant photocurrent starting around zero V vs. SCE. Several methods are available for the measurements of the flatband potential and here we

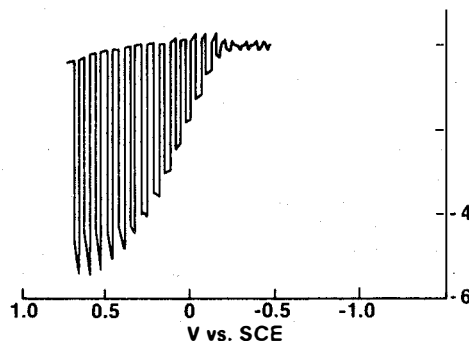
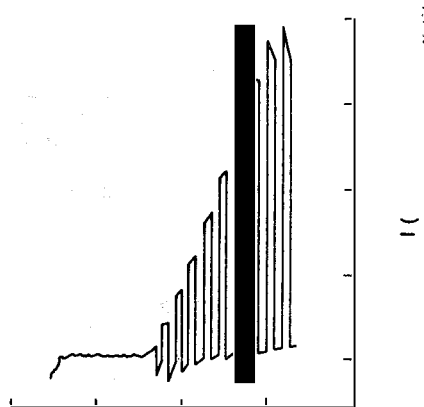


Figure 1. Chopped Current-Voltage Plots for: (a) p-CuInSe₂ crystal (0.35 cm²); (b) n-CuInSe₂ crystal (0.36 cm²), after etching in 2% Br₂/MeOH for 30 sec and heating in air at 90°C for 1 hour. Co(bpy)₃(ClO₄)₂ concentration 0.2 M, 0.1 M TEAP in acetonitrile. Light intensity 1.5-2 × AM1.

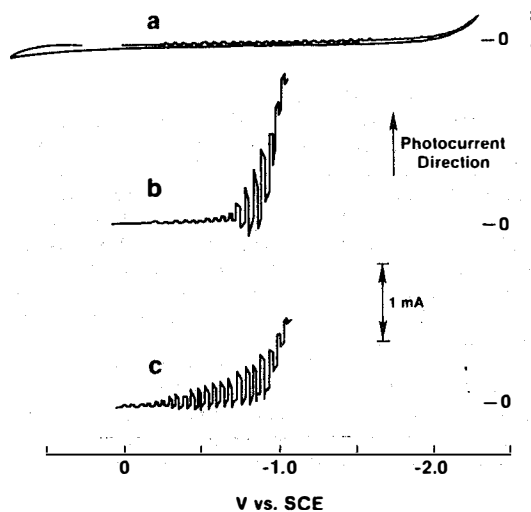


Figure 2. Chopped Current-Voltage Plots for p-CuInSe₂ (0.28 cm²) in 0.1 M Co(bpy)₃(ClO₄)₂, 0.1 M TEAP in acetonitrile. (a) without redox couple; (b) without etching; (c) after 30 sec etch in 0.5% Br₂/MeOH.

report on results from capacitance-voltage (Mott-Schottky) measurements. Fig. 3 shows Mott-Schottky plots for an as-grown film (that had been exposed to air for some time), and for a Br_2/MeOH etched film. The values obtained for the flatband potentials are -0.2 V and 0.0 V vs. SCE, respectively. These results agree fairly well with the potentials at which photocurrent starts (onset potentials) that can be obtained from Fig. 2.

The spatial homogeneity of a pattern of CuInSe_2 squares on Mo was tested in CAT. As shown in Fig. 4, six of the nine squares (area of each square = 1 cm^2) on the checker board were evaluated for their individual photovoltaic activity, using illumination from a tungsten-halogen lamp. The lateral structure of the as-deposited films is also shown. Electrical contact was made directly to the Mo on alumina, between the squares. The photocurrent at -0.3 V vs. SCE was measured for each individual film. The significant difference in the photocurrents between the left squares and the right squares showed the inhomogeneity of these films. The spatial homogeneity of an individual film was tested using laser scanning (11). Fig. 5 shows the results from such tests on a "Boeing" thin film ($11 \text{ mm} \times 4 \text{ mm}$). The top gave better photoresponse than the bottom, while the best photoresponse was obtained in the middle. The results from these tests of spatial homogeneity of films or film patterns could be understood in terms of non-homogeneity of the deposition process (for the layer pattern) and from the effects of previous surface treatments (individual film).

Photoelectrochemical Decomposition Studies

The use of organic solvent, e.g., acetonitrile, which has a wide working potential range

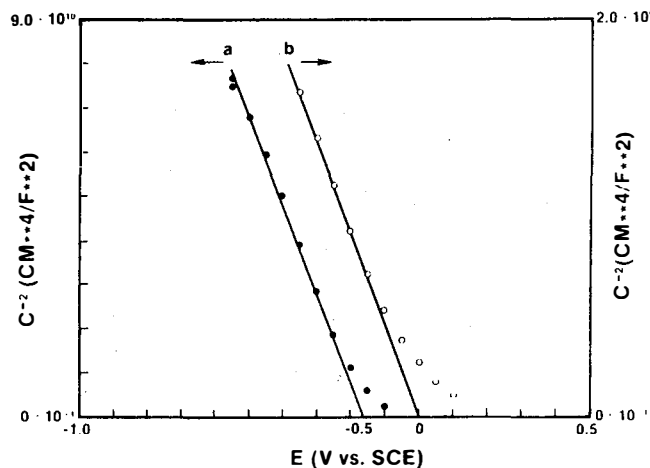


Figure 3. C^2 -V Plots of $p\text{-CuInSe}_2$ Thin Film in $0.1 \text{ M Co}(\text{bpy})_3(\text{ClO}_4)_2$, 0.2 M TEAP in acetonitrile. (a) as-grown thin film; (b) etched in 0.2% Br_2/MeOH for 10 minutes.

(from -2.0 V to $+0.75$ V vs. SCE for CuInSe_2 as shown in Fig. 2a), is useful for (photo) electrochemical studies of semiconductor electrodes. Therefore, many (photo) electrochemical decomposition products could be detected *in situ*, without solvent interference. It is known that CuInSe_2 can be oxidized after exposing it to air (13). Using etchants to produce reproducible surfaces for solar cells should be an important process to improve the cell performance.

While several etchants were identified previously (3), Mott-Schottky and I-V plots indicated that they did not always yield stable surfaces and

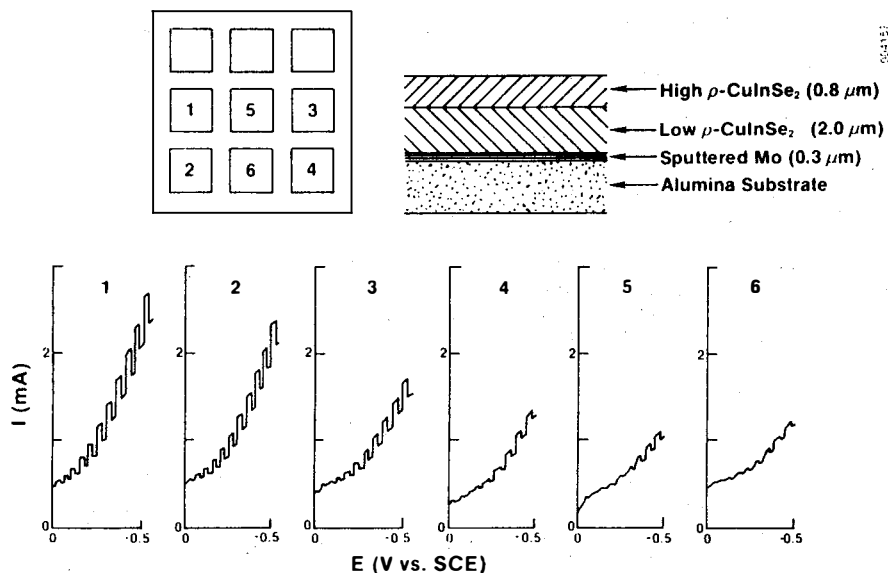


Figure 4. Top: Schematic pictures of $p\text{-CuInSe}_2$ on Mo checkerboard. Bottom: Results of PEC tests of six of the nine squares in CAT. Numbers of the IV curves refer to the numbers of the squares.

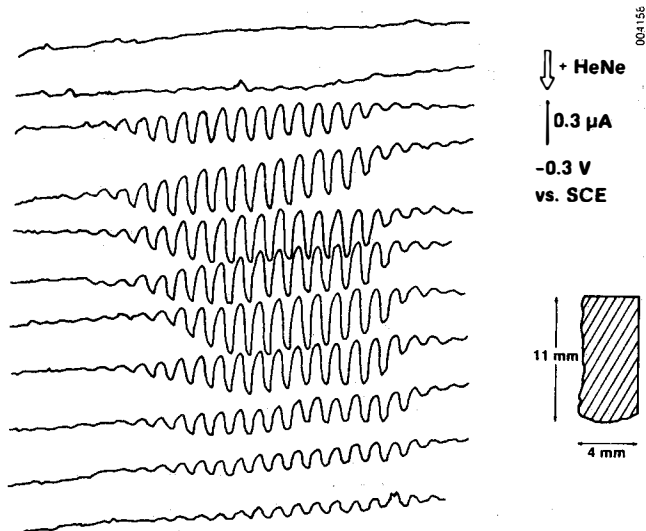


Figure 5. Laser Scan (intermittent illumination, spot size 25 μm) of p-CuInSe₂ Film (outline shown on bottom right) in CAT. A 50 mW HeNe laser was used. Scan rates were 200 $\mu\text{/sec}$ left to right and top to bottom. Note that the measurements were done at -0.3 V vs. SCE. The film was etched in 0.5% (V/V) Br₂/MeOH before measurement.

therefore we searched for alternative etching procedures. Etching with Br₂/MeOH was found to yield results more consistent than those obtained with the previously used acid/H₂O/KCN/H₂O etch. Analyses of etching solutions showed that 2% (V/V) Br₂/MeOH removes the constituent elements in a near-stoichiometric ratio. Surface analyses confirmed that a virtually clean surface, except for adsorbed oxygen and carbon, was obtained. After etching, it was annealed 3 hours at 150°C in air. On the basis of comparative surface analyses using the oxygen and indium Auger parameters, the In 3d line width, MNN Auger line shape, the relative X-ray photoelectron, and Auger intensities, we confirmed earlier indirect evidence (3b,5c,d), that an In-O bond formed upon air anneal, indicating the presence of indium oxide on the surface.

Results from investigations on the photoelectrochemical decomposition of CuInSe₂ and its binary components, Cu₂Se and In₂Se₃ are shown in Figs. 6,7. Figure 6a shows relevant results for Cu₂Se crystals in the dark. They are consistent with the anodic decomposition reaction:

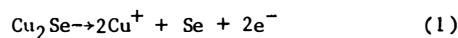
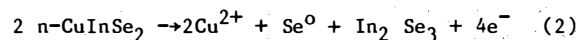
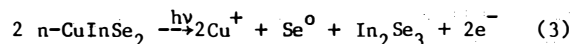


Figure 6b shows no electrochemical decomposition for In₂Se₃ crystals in acetonitrile. Fig. 7 shows relevant results for n-CuInSe₂ crystals in the dark and under illumination. After anodic decomposition, three cathodic waves related to the reduction of Cu²⁺, Cu⁺ and Se⁰ were detected in the dark. Atomic absorption analyses indicate the presence of only Cu in the acetonitrile solution after anodic decomposition. This is consistent with the anodic decomposition reaction:



Under illumination, two cathodic waves related to the reduction of Cu⁺ and Se⁰ were detected after anodic decomposition. Therefore, the photooxidation is:



The above interpretation of the electrochemical results is supported by results from analyses of the contents of the solutions, after electrode decomposition. Surface analyses showed that the

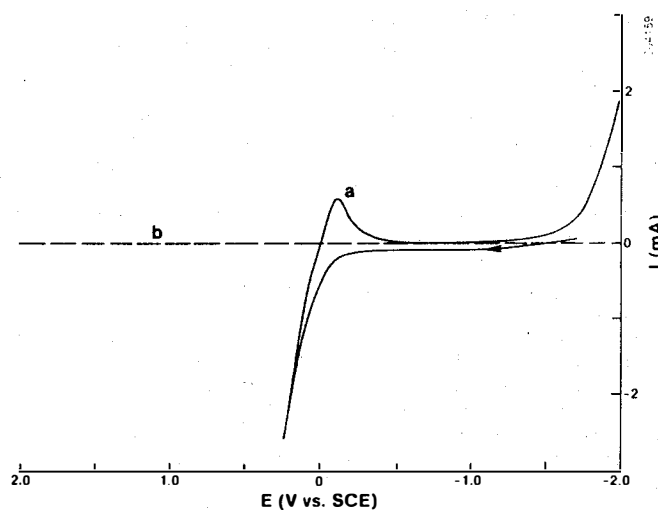


Figure 6. Voltammetric Curves of Cu₂Se (—) and In₂Se₃ (---) in Acetonitrile of 0.1 M TEAP. Electrode area: 1 cm². Scan rate: 50 mV/sec.

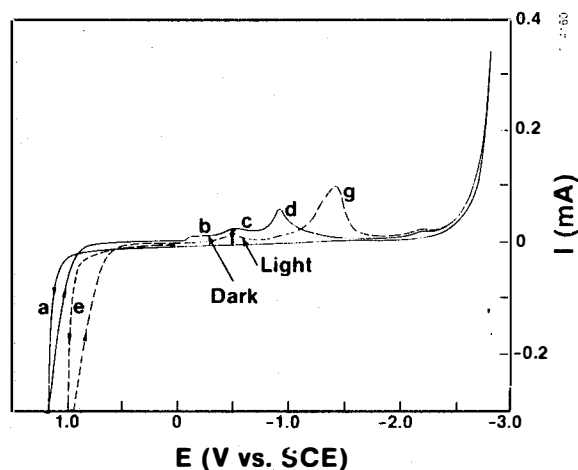


Figure 7. Voltammetric Curves of an n-CuInSe₂ Crystal in Acetonitrile of 0.1 M TEAP. Electrode area: 0.1 cm². Scan rate: 50 mV/sec. Illumination intensity: 100 mW/cm² tungsten-halogen light a & e: Oxidation of lattice selenide to Se⁰. b: Reduction of Cu²⁺ to Cu⁺ c & f: Reduction of Cu⁺ to Cu⁰. d & g: Reduction of Se⁰ to Se²⁻.

top layer of the electrodes was partially depleted of Cu and In. From the above one can conclude that breaking of the Cu-Se bond is the primary step of anodic decomposition of CuInSe_2 . Anodic decomposition was also found in aqueous polyiodide solution. A golden film of Se^0 was observed after photo-decomposition. AES profile analyses show Cu and In depletion on the surfaces. Solution analyses revealed the presence of some Cu, and possibly some In. Anodic decomposition of p-CuInSe_2 shows the same decomposition pathway as eq. (2) in the dark and under illumination. The rather facile removal of Cu from CuInSe_2 may be connected with the considerable range of stoichiometry, along the $\text{Cu}_2\text{Se-In}_2\text{Se}_3$ tie line, for " CuInSe_2 ", as determined from a series of solid state and structural chemical experiments. In fact, the chalcopyrite structure was found to be preserved down to $x = 0.43$ (where x is the formula percent of Cu_2Se in $(\text{Cu}_2\text{Se})_x(\text{In}_2\text{Se}_3)_{1-x}$) and related cubic structures could be found for stoichiometrics with x values as low as 0.25 (ref. 8, paper RNP 511; ref. 14).

Photoelectrochemical Solar Cells

A 30 sec etch with 2% (V/V) Br_2/MeOH was found to be optimal for photovoltaic activity in aqueous polyiodide (Fig. 8 a,b). From experiments in polysulfide electrolytes we noted the beneficial effect of an air heating treatment on photoelectrode performance (3b, 5c,d). The treatment was optimized (Fig. 8 c,d) to yield an increase in the short-circuit current, fill factor, and open-circuit voltage, after annealing 3 hours at 150°C . The formation of a more stoichiometric and more homogeneous indium oxide layer probably contributes to such effect. The combined effect of the surface etch and air anneal has a 30-fold increase of photovoltaic activity in aqueous polyiodide. While efficiencies well over 10% were measured for this system, decay in the short circuit photocurrents under $\sim 1.25 \times \text{AM1}$ illumination ($\sim 50 \text{ mA/cm}^2$) after 10-30 minutes, was significant.

On the basis of results from these experiments and those from photoelectrochemical decomposition and solid state chemical studies, we investigated the effects of the addition of Cu^+ and/or In^{3+} ions to the polyiodide solution. Cu^{2+} was added to the iodide solutions with which it reacted to give Cu^+ (15). In such solutions a dramatic decrease in photocurrent decay was observed. Addition of Cu^{2+} and In^{3+} to iodide solutions produced an even stronger effect, and in some cases completely prevented photocurrent decay, without effecting the I-V characteristics. Most of these studies were conducted in nearly neutral aqueous solutions. At $\text{pH} < 9$, no obvious pH effect on the I-V was found. However, at $\text{pH} > 9$, poor cell performance was observed for the iodide/polyiodide solution probably due to slow charge transfer kinetics and iodide hydrolysis. Since illuminated iodide solutions are sensitive to air oxidation, especially at lower pH values (16), the pH of the solutions was always between 4 and 9.

To obtain, in a completely reproducible way, total electrode output stability, we electrodeposited a thin ($0.3 \mu\text{m}$ based on total geometric

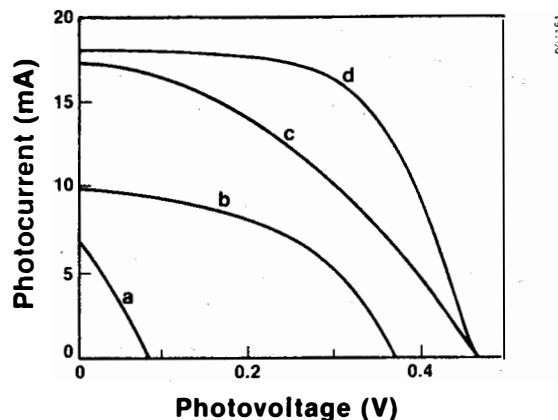


Figure 8. Photocurrent-Photovoltage Curves for n-CuInSe₂ Single Crystal Electrodes in Aqueous 6M KI, 0.5 M I₂ Solution at pH ~ 7, as Function of Surface Treatment a. Polished electrode. b. After subsequent 30 sec etch in 2% (V/V) Br₂/MeOH. c. After subsequent 1 hour air anneal at 150°C . d. As C, but for 3 hours. Solution redox potential (I₃/I⁻) is + 0.30 V vs. SCE. Electrode area: 0.36 cm²; Light intensity: 1.5 × AM1.

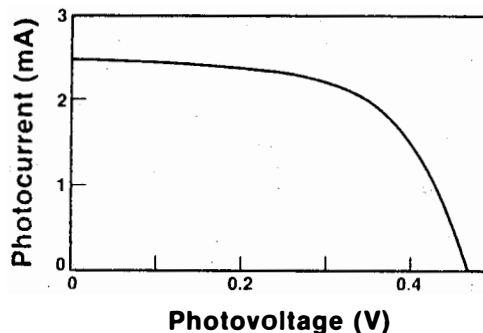


Figure 9. Photocurrent-Photovoltage Curves for n-CuInSe₂ Electrodes in 6 M KI, 0.2 M Cu²⁺ and 0.1 M In³⁺, pH ~ 7. The electrodes were optimized according to curve d in Fig. 8. Subsequently In was plated on the surface, and the plated electrodes were heated in air at 90°C for 3 1/2 hours. Electrode area: 0.05 cm². Illumination intensity was 120 mW/cm² from tungsten-halogen source.

area) layer of In on a previously optimized electrode and air-heated the plated electrode at 90°C for 3 1/2 hours. Such electrodes showed, consistently, no photocurrent decay in the modified polyiodide solutions, while retaining the favorable I-V characteristics of optimized electrodes (Fig. 9). Power conversion efficiencies of 10-11.7% were measured (under tungsten-halogen illumination of 120-145 mW/cm², cf. legend to Fig. 9) for these systems. At 50 mA/cm² short-circuit current, some 20 kC/cm² photocharge passed with no change in the output parameters and no visible change in the electrode surface. Similarly, at 40 mA/cm², max-

imum power current, more than 15 kC/cm^2 photocharge passed while all output characteristics were retained. These results are thus comparable with those for CuInS_2 and CuInSe_2 in polysulfide electrolyte, but at significantly higher conversion efficiencies.

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