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A STUDY ON THE HUMIDITY SUSCEPTIBILITY OF THIN-FILM CIGS ABSORBER

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ABSTRACT

The susceptibility of a thermally co-evaporated CuInGaSe_2 (CIGS) thin-film absorber to humidity and its consequence on composition, morphology, electrical and electronic properties, and device efficiency was investigated. CIGS films on Mo-coated soda lime glass were degraded either in the ambient at $\sim 21^\circ\text{C}$ and $\sim 21\%$ relative humidity (RH) for a period of several months or in damp heat (DH) at 85°C and 85% RH briefly for 15-30 min; then the films were processed simultaneously into devices in a batch that included an unexposed control. In addition to severe delamination on some samples of the absorber films, prolonged ambient exposure resulted in numerous "spot" formations that lost CIGS with scale-like disintegration rippling around the spots and showed a significant presence of Na. Exposure in DH for 5 h was able to reproduce the spot formations on the CIGS films. A significant to large decrease of cell efficiency was observed from 14%–16% for the unexposed control to 8%–11% for the CIGS absorber exposed in DH for 15 and 30 min and 1%–4% for the ambient-degraded CIGS with high series resistance and very low shunt resistance.

INTRODUCTION

Long-term performance reliability of thin-film photovoltaics such as CIGS and CdTe is highly important to their success in gaining broad market acceptance. Packaging capable of blocking or minimizing moisture ingress is essential for PV modules to protect an absorber such as CIGS, window layer such as ZnO, and contact electrode such as Mo from humidity-induced damages and therefore to ensure their durability in the field. For example, Siemens Solar (which is no longer operating) employed a moisture-blocking desiccant-type edge sealant and a TPAT (Tedlar/polyester/aluminum foil/Tedlar) on its CIGS modules. Edge sealants are also beneficially used on glass/glass laminates of CIGS and CdTe modules. The Olson group has developed a multilayer moisture-barrier coating to successfully protect CIGS from DH at $85^\circ\text{C}/85\%\text{RH}$ for more than 1500 h [1-2]. On the side of materials stability, several groups have conducted accelerated stress tests on either complete cells or mini-modules in damp heat, light/dark cycles, or both DH/light exposures [3-11], to better assess the performance stability and understand the degradation mechanisms of CIGS absorbers or devices. Longer-term outdoor performance reliability of CIS and CIGS modules was reported by del Cueto et al. recently [12]. Various factors or mechanisms ranging from increased defect density [3-5]

to increased resistance of ZnO window layer [10,11] have been proposed to explain the differences observed in CIGS device or module degradation behaviors. However, a common issue in such approaches is that using complete devices or (mini-) modules make it difficult to properly differentiate, and/or separate, the factors that contribute to degradation from among the different component materials. In recognizing the instability issues imparted by the main CIGS components, Wennerberg et al. attempted to improve the performance and stability by design modifications [13]. Previously we have demonstrated the instability of Mo back electrode and intrinsic and Al-doped ZnO upon DH exposures [14-16]. To eliminate the ambiguity of uncertain contributions from various degrading factors encountered in testing complete CIGS cells or mini-modules, we chose in this work to only investigate only the humidity susceptibility of the absorber by subjecting the CIGS samples to ambient environment over a prolonged period of several months and to DH condition over a very brief period of hours or less. The ambient and DH-exposed CIGS absorber samples, along with an unexposed control, were then processed simultaneously in a batch into devices. This approach effectively removed the potential variations and uncertainty that would have been found had the devices were fabricated separately. Meanwhile, other studies at NREL also examined the lifetime and stability of the CIGS absorber and partial device structures exposed to the air or stored in a N_2 dry box by using photoluminescence analysis [17-18].

EXPERIMENTAL

Samples and cell fabrication: CIGS absorbers were deposited by NREL's three-stage thermal co-evaporation process on Mo-coated soda lime glass. The standard device fabrication process involved CdS by chemical bath deposition, intrinsic-/Al-doped bilayer ZnO window by sputter deposition, and Ni/Al contact electrode by e-beam evaporation.

Exposures: Some absorber samples placed in clear plastic boxes were allowed to expose to the ambient for 8-12 months inside the laboratory building, which was typically at $\sim 21^\circ\text{C}$ and $\sim 21\%$ RH. Another set of absorber samples was cut from a large piece into four portions, one of which was stored in a N_2 dry box as the control until the moment of device fabrication. The other pieces were exposed in a Blue M chamber operated at 85°C and 85% RH briefly for either 15 or 30 min. Smaller specimens for morphological and microscopic studies were DH-exposed accumulatively from 0.5 to 1, 2, and 5 hours.

Characterization: Surface morphological features and changes were examined by an optical microscope at low magnification factors, a WYKO interference optical microscope at high resolutions, and an FEI Nova NanoSEM 630 scanning electron microscope (SEM). Localized small-area composition analysis was performed by using a JEOL 8900 Superprobe electron probe microanalysis (EPMA) with a typical $\sim 20\text{-}\mu\text{m}$ probe size. Distribution of work function was examined using scanning Kelvin probe force microscopy (SKPFM) [19,20] with fine resolutions of several tens of nanometers. SKPFM is based on the non-contact mode of atomic force microscopy (AFM), and measures surface potential/work function by measuring a coulomb force between the AFM tip and sample. Admittance spectroscopy measurements were conducted by using an Agilent A4294 impedance analyzer, typically with an AC modulation voltage of 45 mV_{rms} at a frequency of 10 kHz.

RESULTS AND DISCUSSION

Compositional Changes. The CIGS absorber films upon prolonged ambient or 5-h DH exposures exhibited numerous visible spots. Some ambient-degraded films even became wrinkled and delaminated. Some results of localized composition analysis by EPMA at 10KeV are given in Table 1 for two samples (A and B) before and after prolonged degradation by ambient exposure. Sample A was a Ga-rich film, and sample B had a typical CIGS film composition. When elemental weight percentages are compared, the compositions of the ambient-degraded but still smooth regions on sample A are similar to those of the initial film; however, the spotty areas are relatively low in Cu, Ga and Se. Greater compositional changes are seen for the spotty areas on sample B than that for sample A. Upon normalization to the atomic ratio percentage, the spotty areas are comparatively low in Ga for both samples. Significant presence of Na was also found at the spotty formations, but its concentration was difficult to quantify.

Morphological and Optical Changes. The optical images in Fig. 1 show the visible spots on the CIGS films. As revealed from examination under a WYKO interference microscope, the spot formations are in fact small regions of CIGS that were disintegrated (lost) with finite scale-like lines rippling around the spots (figures not shown). The SEM micro-images in Fig. 2 compare the surface morphology of a still smooth area near the spot formation

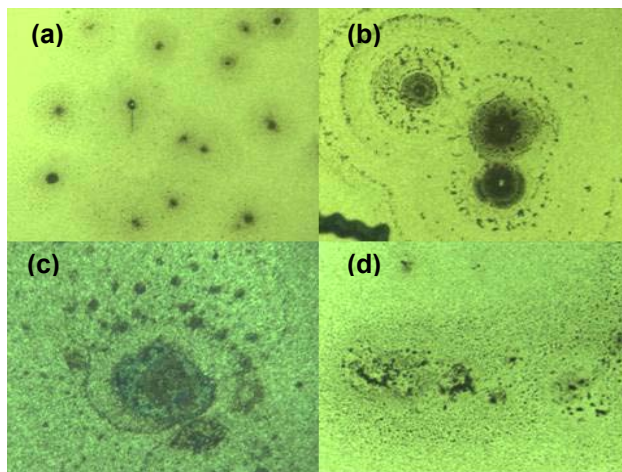


Fig. 1. Optical images with artificial green color of some spot formations on ambient-degraded CIGS films at a magnification of 10X (a), 16X (b), or 40X (c, d).

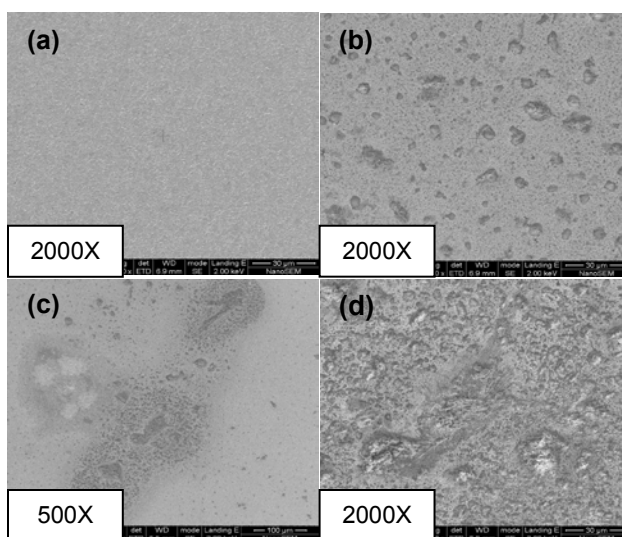


Fig. 2. SEM micrographs for an ambient-degraded sample (#A, Table 1) over several months: (a) a still “smooth” area, (b) an area with a large number of small pits, and (c, d): large pitting spots, which were visible and corresponded to the images seen under the optical and interference microscopes. The magnification factors are indicated in the micrographs.

Table 1. Compositions for CIGS Absorber Films with or without Exposure to Ambient or Damp Heat

Sample ID	Exposure	Area	Weight Percent (Average of 5 points)					Atomic Ratio (Average of 5 points)				
			Cu	In	Ga	Se	Total	Cu	In	Ga	Se	Total
A	No (Initial)	smooth	17.23	22.76	10.27	51.25	101.50	21.42	15.66	11.64	51.28	100.00
	Ambient	smooth	17.20	23.44	9.82	51.15	101.60	21.42	16.16	11.15	51.28	100.00
	Ambient	spotty	15.59	21.07	8.74	47.61	93.01	21.10	15.82	10.79	52.29	100.00
B	Ambient	smooth	18.97	26.57	7.21	50.30	103.05	23.50	18.21	8.14	50.15	100.00
	Ambient	spotty	12.19	17.89	4.40	34.39	68.87	22.37	18.43	7.11	52.08	100.00

and the spot for an ambient-degraded CIGS sample. A large degree of morphological (and compositional, above) disintegration was present, especially around the spot formations. Optically, despite the presence of visible spotty formations and some degree of delamination, the ambient-degraded CIGS films showed essentially identical reflectance spectra in the 250-900-nm range, but a consistent peak shift of ~30-40 nm in the interference pattern in the 900-1500-nm range, as seen in Fig. 3. Currently, it can not be clearly determined if this spectral shift is caused by certain changes in film thickness, non-uniformity, optical density, or compositions.

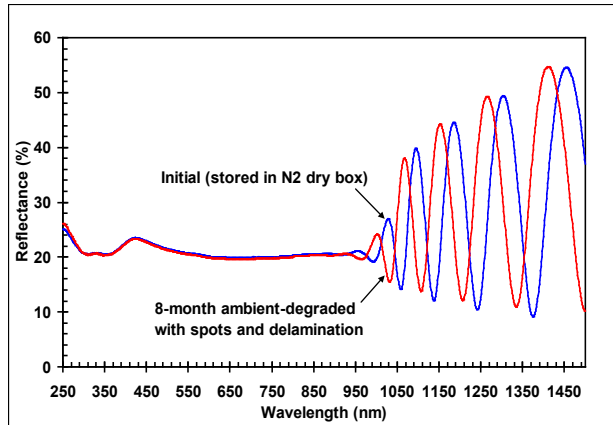


Fig. 3. Reflectance spectra for a CIGS absorber film on Mo/SLG substrate before and after 8-month ambient exposure.

SKP/AFM Analysis. The scanning Kelvin probe/AFM (SKPFM) was employed to examine the effect of DH exposure on the electronic property (work function or surface potential). The SKPFM and AFM images taken before a DH exposure, shown in column (a) in Fig. 4,

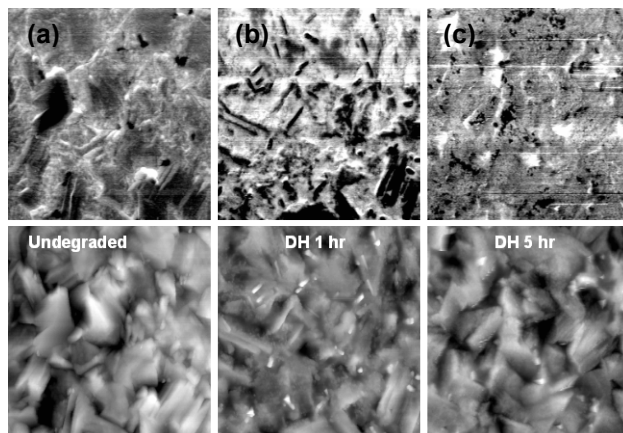


Fig. 4. Corresponding scanning Kelvin probe (SKP, top row) and AFM (bottom row) images for DH-degraded CIGS on Mo/SLG: column (a) undegraded specimen, (b) 1-h DH exposure, and (c) 5-h DH exposure.

exhibit typical CIGS topographic features and a well-defined work-function mapping. The grain topography and work function distribution appear to show well-defined correlations; that is, the work function depends closely on the grains' surface facets and its values on the grain boundaries may depend on specific grain boundary structures. Small spots appeared on the AFM images after 1- and 5-h DH exposures (columns [b, c], bottom). The SKPFM images show significant changes with the DH exposures, suggesting that the work function had gradually lost its correlation with the grain geometric feature upon DH exposure. After 5 h in DH, the initially well defined work-function images became fairly blurred, likely a result of electronic property breakdown on the CIGS grains.

The sensitivity of the CIGS absorber to the environment (humidity and oxygen) was evidenced by the rapid decline of carrier lifetime after exposing it to the air for only one day, as observed by Metzger et al. in their studying the stability of CIGS absorber films using photoluminescence [17,18]. The degradation was greatly reduced if the CIGS was quickly deposited with CdS; the CIGS/CdS was found stable in the air for months [17,18]. The stabilization mechanism of CdS for CIGS is not clear, however. On the other hand, Würz et al. reported the formation of a $\text{Cu}(\text{OH})_2$ surface phase on the CuGaSe_2 that was stored in the ambient for several months [21]. Heske et al. reported the formation of sulfate, which resulted from sulfur oxidation induced by DH in the $\text{Cu}(\text{In,Ga})(\text{S,Se})_2$, CIGSSe-based solar cells [22]. While more in-depth studies are required, it is likely that the CIGS absorber thin films have undergone similar oxidation/hydrolysis when exposed in the ambient as well as in the damp heat.

Admittance Spectroscopy Measurements. To gain understanding of the interface and bulk electrical properties [23,24], we also conducted some capacitance (C) measurements dependent on both frequency (f) and bias voltage (V), i.e., bias dependent admittance spectroscopy on the complete cells. Figure 5(a) shows a 3-D plot of the admittance spectroscopy of a device made of a CIGS sample treated in DH for 15 min as a function of both the reverse-bias voltage and measurement temperature. The signatures of two trap levels are evident: one corresponding to the bulk states with its activation energy (~140 meV) essentially independent of bias voltage and the other was clearly influenced by the increasing bias voltage. Figure 5(b) shows the activation energy for the deep level states as a function of bias voltage for the devices on four different cell samples. The activation energy of those states was obtained from the Arrhenius plot of frequency versus temperature at which the differential capacitance $fdC/d[\ln(f)]$ spectrum peaks. The results show that ambient and DH degradation introduces a deep level of >400 meV. This level is absent in the unexposed reference control sample. Because the activation energy of this deep level increases with reverse bias, the deep level apparently originates from interface states [25,26]. In comparison, Deibel et al. reported an

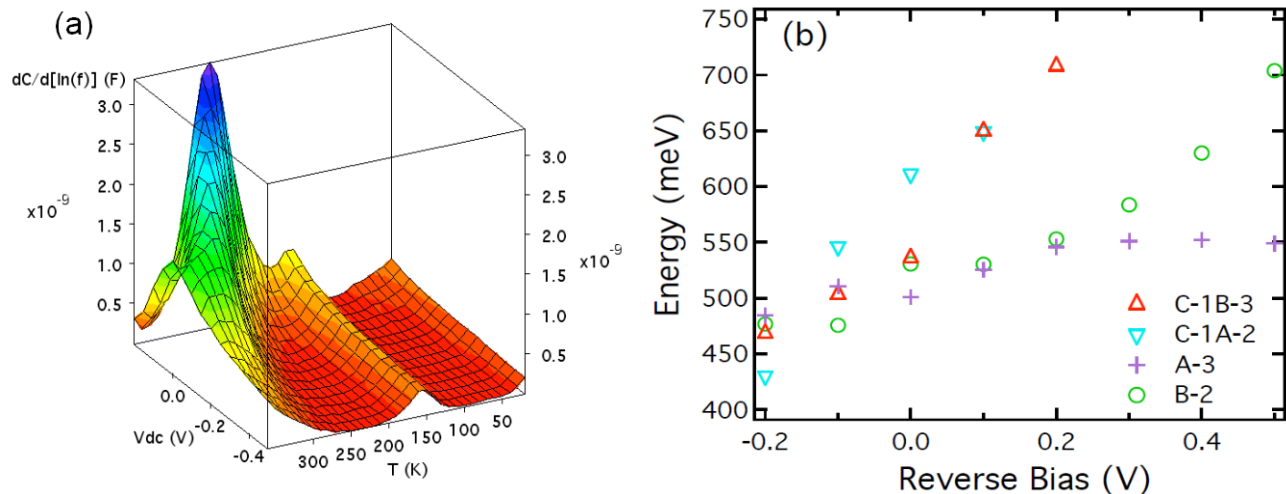


Fig. 5. (a) A 3-D plot of admittance spectroscopy as a function of temperature and bias for a 15-min, DH-treated CIGS device (C-1B-1, Table 2), and (b) the activation energy of deep-level states as a function of bias voltage for the devices on four samples with device numbers indicated in the legends (see Table 2 for the exposure treatments of the CIGS absorber films).

Table 2. Cell Parameters for CIGS Samples with or without Exposure to Ambient or Damp Heat

Sample ID	Exposure	Device No.	Voc (V)	Jsc (mA/cm ²)	FF (%)	Eff (%)	Rs (ohm-cm)	Rsh (ohm-cm)
A	ambient	1	0.415	10.30	34.3	1.5	16.8	58
		3	0.436	6.44	31.6	0.9	33.3	78
B	ambient	1	0.411	22.89	40.5	3.8	6.9	44
		4	0.385	25.67	44.9	4.4	4.5	55
C-1A	DH 15 min	2	0.544	30.84	65.6	11.0	2.5	413
		4	0.560	32.04	66.0	11.8	2.4	628
C-1B	DH 30 min	1	0.512	30.20	64.4	10.0	2.8	504
		3	0.513	30.91	63.7	10.1	2.8	376
C-0A	No (N ₂ dry box)	1	0.654	30.28	71.1	14.1	1.7	4491
		7	0.652	31.39	76.9	15.7	1.4	14126
C-0B	No (N ₂ dry box)	1	0.668	29.78	71.2	14.1	2.1	4228
		5	0.672	31.10	77.2	16.1	1.4	20536

activation energy of 160–180 meV for the interface states in a device made of CIGSSe absorber that was DH-treated for 24 h prior to device fabrication; and the activation energy of the interface states ranged from 85 to 340 meV for their devices exposed to DH ranging from 2 to 438 h, as determined from C-V measurements at 90°K with a frequency of 100 kHz [4,5].

Cell Efficiency Changes. The unexposed and ambient- and DH-exposed CIGS absorber samples were fabricated into cells simultaneously in a batch having a common configuration of SLG/Mo/CIGS/CdS/bi-layer ZnO. The cell efficiencies were also measured all in one session. Some of the cell I-V parameters are given in Table 2. It is obvious that the prolonged ambient exposure (samples A and B) resulted in very poor devices due to substantial compositional and morphological degradations on the absorber films. The devices' high series resistance (Rs)

and low shunting resistance (Rsh) suggest that shorting and shunting had occurred. Even brief exposures in DH for 15 min and 30 min induced significant efficiency losses as evidenced by samples #C-1A and 1B, respectively, when compared to the unexposed control samples (samples C-0A and C-0B). The C-0 and C-1 samples were made from specimens cut from the same piece of CIGS film on Mo/SLG.

CONCLUSIONS

We have shown the detrimental consequences in CIGS absorbers that were subjected to low humidity levels over prolonged periods at ambient temperature and high humidity at a high temperature in a DH environment. The humidity susceptibility of the CIGS absorber resulted in

compositional, morphological, and electrical deteriorations, which in turn caused the loss of cell performance. More work is on-going to further evaluate the sensitivity of CIGS absorber to various RH levels and temperatures.

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14. ABSTRACT (Maximum 200 Words) The report summarizes the research on the susceptibility of a thermally co-evaporated CuInGaSe ₂ (CIGS) thin-film absorber to humidity and its consequence on composition, morphology, electrical and electronic properties, and device efficiency.						
15. SUBJECT TERMS CIGS, thin-film absorber, humidity, reliability, packaging, photovoltaics						
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