Solar energy has received increasing interests as an alternative to other sources of energy such as fossil fuels and nuclear energy.1–6 One of the most promising solar cells is based on the polycrystalline Cu(In,Ga)Se2 (CIGS) fabricated by using vacuum based process whose conversion efficiency is up to 21.7%.7 Although vacuum techniques seem to be an excellent method for fabricating high efficiency CIGS solar cells, it is challenging to scale up the process for commercial production. While sputtering and evaporation techniques could be used for large-area deposition, however, the production process is limited by expensive vacuum equipment as well as large energy consumption.8–11 For low-cost fabrication of semiconductor thin films, electrodeposition has been proved to be a viable approach for large scale manufacturing of CIGS.10–13 However, most of the works are carried out in aqueous solvents, in which hydrogen evolution reaction (HER) leads to low plating efficiencies and pinholes in the plating layer.14,15 To avoid the occurrence of HER, researchers in recent years have turned to deep eutectic solvents (DES) as an electrolyte for electrochemical reactions due to their wide electrochemical window, high ionic conductivity, high solvation capacity, negligible vapor pressure and excellent stability.16–20 Besides, DES, as a subclass of ionic liquids (ILs), is a green electrolyte for application in electroplating.14,16,21–23 Reline, as one application of DES, is formed by choline chloride and urea at the ratio of 1: 2. Harati et al. fabricated successfully stoichiometric CIGS thin film by the one-pot electrodeposition in Reline as electrolyte.21 Malaquias et al. used Reline to deposit In–Ga on Cu and Mo electrodes and finally obtained solar cell with a 7.9% efficiency.15 Chen et al reported a 3.87% CZTS solar cell efficiency by co-electrodepositing Cu–Zn–Sn precursor thin films in Reline.22 Although these studies show the viability of Reline as a green electrolyte, its micro-structure and electrochemical properties are rarely reported and the formed metal complexes in Reline are also needed further investigation. Meanwhile DES is cheap and easy to synthesize and has wide applications in the electrochemical industry, understanding of the above mentioned is important to control or improve the electrolysis process. This work is such an attempt. In this work, we detected the fundamental properties of the ionic liquid and ligand forms of metal ions in Reline. The electrochemical properties show prospects to control the composition of three metals in a one-step electrodeposition. Based on these achievements and optimizations, we introduce a facile, additive-free and Ga-controllable method to fabricate Cu-In-Ga precursor by co-electrodeposition in Reline and achieved a best CIGS solar cell with an efficiency of 10.1%.

Experimental

Experiments related to synthesis and electrochemistry tests were performed in a nitrogen atmosphere glove box (MBrann Umlab) to avoid the influence of moisture. Water and oxygen contents inside the glove box were lower than 1 ppm. The ionic liquids were prepared by mixing choline chloride (ChCl, Alfa Aesar, 99.99%) and urea (Alfa Aesar 99%) at a molar ratio of 1: 2 at the temperature of 65 °C. Analytical reagent grade chemicals 30 mM CuCl2 (Alfa Aesar, 99.999%), 35 mM InCl3 (Alfa Aesar, 99.999%) and 60 mM GaCl (Alfa Aesar, 99.999%) were added successively in the Reline ionic liquid with 200 rpm stirring. After the solution turned blue indicating a stable state, electrochemistry tests and deposition were carried out. A three-electrode system was used with Mo coated soda glass as the working electrode, a platinum foil as the counter electrode and a silver wire as the reference electrode. The Cu-In-Ga precursors were electrodedeposited in a one-step process by differential pulse potentiostatic method controlled by a potentiostat (Tatreplate CP, MPC, France). After the deposition, the substrate was removed from the electrochemical bath, rinsed with methanol and deionized water, then dried under argon flow. The post-annealing treatments were carried out in a graphite container with excess selenium particles (purity, 99.999%), which was inserted into a quartz tube filled with Ar at 30 Torr. The precursor films were heated up to 550 °C rapidly and held at this temperature for 1 hour in a rapid thermal process (RTP) furnace. The CIGS absorber films were further processed into photovoltaic devices by chemical bath deposition of CdS, sputtering of i-ZnO and Al: ZnO, and evaporation of the Ni/Al contact grid.

The optical properties of thin films were characterized using a Shimadzu UV-3101PC spectrophotometer at room temperature. Fourier transform infrared spectroscopy (FTIR) analysis was carried out using a Nicolet 6700 FTIR Spectrophotometer (Thermo Nicolet Corporation, Madison, USA) in liquid films using KBr tablets. X-ray diffraction (XRD) measurements were carried out on a Shimadzu XRD diffractometer with Cu Karadiation. The morphologies and compositions of the samples were observed using a Hitachi S5200 field emission scanning electron microscope (SEM) equipped with energy dispersive.Current-voltage (I-V) measurements were performed using a Keithley 2400 General Purpose Sourcemeter under a Xe-based light source solar simulator providing AM 1.5G illumination (100 mW cm−2).

Results and Discussion

In order to analyze the micro-structure of the deep eutectic solvent of Reline, FTIR analysis is carried out as shown in Fig. 1. From

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the FTIR spectroscopy, the interaction between different groups can be learned so as to analyze and identify the structures of chemical bonds from the frequencies. Fig. 1 presents the FTIR spectroscopy of ChCl(s), Urea(s) and Reline (l), respectively. It can be noted that the IR spectrum of Reline is generally the combination of frequencies in the IR spectra of ChCl and Urea except for some frequency shifts. The frequencies at 3258 cm$^{-1}$ in ChCl are assigned to the -CH$_3$ stretching vibrations. The frequencies at 3447 cm$^{-1}$ and 3347 cm$^{-1}$ in Urea are related to coupled vibrations of –NH$_2$ symmetric stretching vibration and antisymmetric stretching vibration, respectively. The band at 3250 cm$^{-1}$ is attributed to the –NH$_2$ bending vibration. Meanwhile, frequencies at 1683 cm$^{-1}$ and 1631 cm$^{-1}$ are the characteristic frequency vibration of –CONH$_2$. In the sample of Reline, those bands shift toward an obvious lower frequency and change to broader bands. The observation indicates the formation of hydrogen bonds between ChCl and Urea. The existence of hydrogen bonds reduces the bond force constants of the original bonds, a reduction which results in the declining and broadening of the absorption frequency. The hydrogen bonds in the system could exist as –NH... OH–, –NH... NH–, –OH... OH–, –OH... NH–.

The IR spectrum confirms the presence of hydrogen bonds which explains why the deep eutectic solvent of Reline exists as liquid state at room temperature. This solvent is transparent and colorless as shown in Fig. 2a. After CuCl$_2$, InCl$_3$ and GaCl$_3$ were added to the solvent and dissolved, the color of Reline changed from colorless to yellow as presented in Fig. 2b. However, it then changed to green and finally became stable in blue with the increase of stirring time, as shown in Figs. 2c and 2d. The color of yellow is indicative of the presence of chlorocomplex anion of Cu$^{2+}$ ion and the blue color maybe of Cu$^{2+}$ ion or the complexes of Cu$^{2+}$ and –OH, –NH$_2$, –C=O, because when InCl$_3$ or GaCl$_3$ was added separately the liquid was colorless. The green color points to a mixture of different complexes. How the liquid changed from yellow to blue will be explored in the next section.

UV-vis absorption is a very simple method and applicable to exploring the structure formation and changes of the complex. In order to get more information on the structure of the solutions, UV–vis absorption spectra were recorded. To simplify the solution system, different color of Reline ionic liquids dissolved only with CuCl$_2$ are investigated. Fig. 3 shows the UV-vis absorption spectra of yellow, green and blue Reline ionic liquids dissolved with CuCl$_2$. In the ultraviolet-visible region between 200 and 450 nm, intense charge-transfer bands can be observed. In the sample of the yellow ionic liquid, one main peak at 275 nm and a small peak at 386 nm are observed in the UV–vis part of the absorption spectrum. These absorption bands can be attributed to the [CuCl$_4$]$^{2-}$ complex. Because light is absorbed in the blue spectral region, this absorption band is responsible for the typical yellow color of [CuCl$_4$]$^{2-}$ solutions. With increasing time of stirring, the peak at 386 nm decreases in intensity and the main peak at 275 nm shifts to blue, a color change which occurred when Cu$^{2+}$ combined with a stronger ligand, thus causing a larger orbit splitting energy. According to the analysis of ligand field theory (I$^-$ < Br$^-$ < S$^{2-}$ < SCN$^-$ < Cl$^-$ < NO$_3^-$ < N$_3^-$ < F$^-$ < OH$^-$ < C$_2$O$_4^{2-}$ < H$_2$O < NCS$^-$ < CH$_3$CN < pyridine < NH$_3$ < ethylenediamine <

**Figure 2.** Photographs of Reline and color changed after dissolving CuCl$_2$, InCl$_3$ and GaCl$_3$ with stirring.

**Figure 3.** UV-vis absorption spectra of yellow, green and blue Reline ionic liquids dissolved with CuCl$_2$.
2, 2’-bipyridine < 1, 10-phenanthroline < NO2− < PPh3 < CN− < CO2−. The absorption band observed at 258 nm as shown in the blue ionic liquid absorption spectrum most likely is the [Cu(NH2CONH2)2]+. Due to the π → π∗ transitions, the absorption shows a blueshift in the spectra due to the polarization within the chromophore caused by the metal–ligand interactions. Therefore the color change we observed agrees well with the theory. Referring to previous work reported by Cristina Trujillo et al. and combined with our analysis above, we could deduce that the structure of Cu2+ exists as the following structure, [Cu⋅⋅⋅O]=C(NH2)2]+. The deduction is also applicable to In3+ and Ga3+.

From the analyses of FTIR and UV-Vis, the structure of Reline and the forms of metal complex are confirmed. The existence of hydrogen bonds between Urea and ChCl explains why Reline is in a liquid state at room temperature. The analyses also show that the metal ions are complexed with urea and become more stable so that other additives are not required in this electrochemical system. Therefore Reline can be used as a green electrolyte in the CIGS electrochemical industry. In addition, understanding the electrochemical properties such as the potential window of Reline and the cathode potential of Cu, In and Ga dissolved could help to control the ratio of metal elements in the precursor which could be used to fabricate stoichiometric CIGS films for high efficiency solar cell devices, as described in our work below.

In order to study the electrochemical properties of the deep eutectic solvent of Reline and cathode potential of Reline-metal elements, cyclic voltammetry (CV) experiments were carried out. Cyclic voltammetry was performed using a three-electrode system composed of the same two 2 cm × 2 cm Pt foils as the working electrode and the counter electrode, Ag wire as the reference electrodes in a scan rate of 20 mV s−1 at the temperature of 65 °C. The CV measurements were conducted after the open circuit voltage of the electrochemical cell was stabilized. To explore the interaction on electrochemical behavior between the elements, CVs from electrolytes containing 30 mM CuCl2, 30 mM CuCl2 + 60 mM GaCl3 and 30 mM CuCl2 + 60 mM GaCl3 + 35 mM InCl3, respectively, were studied as shown in Fig. 4. The voltammograms were recorded in the range from 2 V to −2 V. In Curve (a), corresponding to a Reline ionic liquid, the potential window of Reline ranges from 0.85 V to −1.7 V, which is in accordance with a previous study by A. P. Abbott.

In Curve (b), corresponding to a Reline-CuCl2 solution, a cathodic peak potential at E = −0.55 V (c1) is recorded. Wave c1 corresponds to the deposition of metallic Cu on the forward scan. Curve (c), corresponding to a Reline-CuCl2-GaCl3 solution, shows a similar shape. The potential for c1 does not move forward or backward, which indicates that the cathodic potentials for Cu2+ is not changed after GaCl3 was added to the Reline ionic liquid. However, the measured current density for Cu2+/0 in Curve (c) becomes lower than that in Curve (b) (2.9 mA cm−2 and 3.6 mA cm−2, respectively). Meanwhile, a new cathodic peak potential is detected, where the deposition of Ga occurred at E = −1.60 V (wave c2). Curve (d) presents the CV of Reline-CuCl2-GaCl3-InCl3 solution. The cathodic peak potential for In3+/0 at −1.04 V (wave c3) is recorded. The c1 and c2 waves is not the current densities for Cu2+/0 and Ga3+/0 are reduced compared to in Curve (c).

From these plots, the diffusion coefficients for Cu2+/0, Ga3+/0 and In3+/0, D0 could be calculated using an abbreviated form of the Randles-Sevcik equation, Eq. 1:

\[ i_p = K c^* A \sqrt{n F D_0 v} \]  

\[ K = 0.4463 \sqrt{ \frac{F^3}{RT} } \]

Where \( i_p \) is the peak current, \( n \) is the number of electrons, \( A \) is the area of electrode, \( c^* \) is the bulk concentration, \( v \) is the potential sweep rate, \( F \) is the Faraday constant, \( R \) is the gas constant, and \( T \) is the absolute temperature. Table I depicts the calculated diffusion coefficients of Cu, In and Ga atoms at 65 °C.

### Table I. The calculated diffusion coefficients of Cu, In and Ga at 65 °C.

| Solution | Ion      | \( i_p \) / mA cm\(^{-2}\) | \( D_0 \) / cm\(^2\) s\(^{-1}\) |
|----------|----------|-------------------------------|----------------------------------|
| Reline-CuCl2 | Cu\(^{2+/0}\) | 3.6 | 1.4 \times 10^{-6} |
| Reline-CuCl2-GaCl3 | Cu\(^{2+/0}\) | 2.9 | 9.2 \times 10^{-7} |
| | Ga\(^{3+/0}\) | 2.6 | 5.5 \times 10^{-8} |
| Reline-CuCl2-GaCl3-InCl3 | Cu\(^{2+/0}\) | 2.5 | 6.8 \times 10^{-7} |
| | Ga\(^{3+/0}\) | 1.8 | 2.6 \times 10^{-8} |
| | In\(^{3+/0}\) | 1.3 | 4.0 \times 10^{-8} |

From the analysis of CVs and Table I, it could be concluded that while those three chloride salts in Reline had hardly any effect on the cathode potentials for each metal elements, they changed the speed of the diffusion to the working electrode. The diffusion of Cu\(^{2+/0}\) is found to be much larger than the other two elements. Meanwhile, the ion transfer kinetic of Ga\(^{3+/0}\) is lower than that of In\(^{3+/0}\), which is in agreement with the research of Malaquias. These results could bused to change the composition ratios in Cu-In-Ga precursor by using Fick’s 1st Law as described by Eq. 2:

\[ J = -D \frac{dC}{dx} \]
### Table II. The composition of the precursors controlled by different electrodeposition pulses after selenization characterized by EDS.

| Samples ID | Cu  | In  | Ga  | Se  | Elemental composition (at%) | Composition ratio |
|------------|-----|-----|-----|-----|-------------------------------|-------------------|
|            | Cu% | In% | Ga% | Se% | Experimental Cu/(In+Ga) | Experimental Ga/(In+Ga) | Calculated Ga/(In+Ga) |
| S1         | 22.67 | 26.14 | 0.07 | 51.12 | 0.86                  | 0.01               | 0                  |
| S2         | 23.98 | 24.32 | 3.16 | 48.54 | 0.87                  | 0.12               | 0.16               |
| S3         | 21.60 | 21.40 | 4.40 | 52.60 | 0.84                  | 0.17               | 0.22               |
| S4         | 22.66 | 18.23 | 6.73 | 52.38 | 0.91                  | 0.27               | 0.36               |
| S5         | 24.78 | 15.16 | 12.48 | 47.58 | 0.90                  | 0.45               | 0.53               |

Figure 5. J-V characteristics of the best CIGS solar cell device and the cross-section morphology.

length (cm). According to Fick’s Law,\(^1\) \(v_0\), the rate of electrodeposition \((\text{mol s}^{-1})\) could be described as Eq. 3:

\[
v_0 = -D_0 C_0 A. \tag{3}
\]

Where \(D_0\) is the diffusion coefficient \((\text{cm}^2 \text{s}^{-1})\), \(C_0\) is the concentration of the electroactive species \((\text{mol L}^{-1})\), \(A\) is the area of diffusion. According to the CV test, when cathodic potential is greater than \(-1.6\) V, both the gallium and indium could be deposited. Combined with the values of \(D_0\) and \(C_0\), the rate of electrodeposition ratio of In:Ga = 1:1.11. That is, the max value of Ga/(In+Ga) ratio is 1.11/(1+1.11) = 0.53. When the cathodic potential is between \(-1.6\) V and \(-1.04\) V, the content of gallium is 0. According to the established equation, we are able to finely control the Ga/(In+Ga) ratio over a large composition range by controlling the electrodeposition time at each pulse of each element. We are able to finely control the Ga/(In+Ga) ratio over a large composition range by controlling the electrodeposition time at each pulse of each element. The Ga/(In+Ga) ratio could be tuned from 0 to 0.53 theoretically.

Table II presents the composition ratios of the precursors after selenization controlled by different electrodeposition pulses characterized by EDS. Different pulse potentiostatic methods were applied to the samples S1~S5. The detailed information is shown in Fig. S2 in the Supplemental Material. From the EDS test results, it is concluded that the experimental composition of Ga/(In+Ga) is from 0.01 to 0.45. Because the real electrodeposition system in Reline is complicated, the calculated ratios are not quite equal to the experimental ones. It still has the guidance to the experiment.

The CIGS absorber films were then processed into photovoltaic devices by chemical bath deposition of CdS, sputtering of i-ZnO and Al:ZnO, and evaporation of the Ni/Al contact grid. Fig. 5 shows the J-V characteristics for the best CIGS solar cell under AM1.5G illumination. The best device represents a power conversion efficiency (PCE) of 10.1% with the illumination area of 0.22 cm\(^2\). The device gives an open circuit voltage of \(V_{oc} = 472.7\) mV and photocurrent of \(J_{sc} = 34.0\) mA/cm\(^2\) where the fill factor (FF) is 62.6%.

The J–V behavior of a CIGS solar cell can be described by a general single exponential diode equation:

\[
J = J_0 \exp \left[ \frac{q}{AKT} (V - RJ) \right] + GV - JL \tag{4}
\]

For the case of constant JL, the series resistance R and shunt conductance G are lumped circuit model representations of the losses that occur in series or parallel with the primary diode, respectively. According to Equation 4, the diode parameters as the shunt conductance G, the series resistance R, the ideality factor A and the saturation current density \(J_0\) can be further extracted from the dark J-V curve. A shunt conductance G of 0.7 and the saturation current density

Figure 6. The derivative dV/dJ against \((J + J_{sc})^{-1}\) (a) and a semilogarithmic plot of \(J + J_{sc} - GV\) against \(V - RJ\) (b).
In this work, a green and low cost electrolyte named Reline was employed to electrodeposit Cu-In-Ga precursor for CIGS solar cells. The micro-structure property of Reline and the metal complex forms were studied, which gives the chemical insights to explain why Reline could be used as a green electrolyte in the electrochemical industry. The metal complex system of Reline and metal salts is deduced by FTIR and UV-Vis absorption spectroscopy analyses. The theoretical diffusion coefficients of Cu, In and Ga at 65 °C are calculated by Cyclic voltammetry. According to Fick’s law, the Ga/(In+Ga) ratio has been successfully controlled over a large theoretical composition range (0 ~ 0.53) by different pulse potentiostatic methods. With this approach we have achieved a champion CIGS solar cell with an efficiency over 10%.

Conclusions

Acknowledgments

This work is supported by the National Nature Science Foundation of China (51202227 and 51502015), the Synergistic Innovative Joint Foundation of AEG-SCU (xtxc2011008), the National High Technology Research and Development Program of China (2012AA050704) and the Science and Technology Development Foundation of China academy of Engineering Physics (2014B0302054).

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