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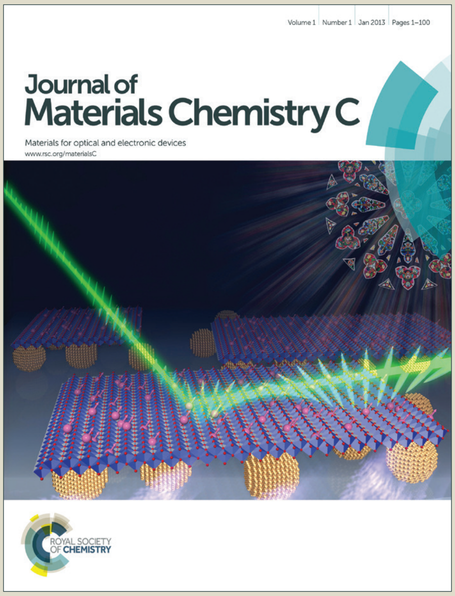

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Fabrication of transparent conducting films composed of In^{3+} doped CuS and their application in flexible electroluminescent devices

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Transparent conductive films composed of CuS were formed by wet deposition on PET at room temperature followed by annealing at 100 °C for 1 hour. The resistance of the films was tuned by doping with In^{3+}. A decrease of over an order of magnitude of the sheet resistance was obtained, from 1721 Ω sq^{-1} for undoped CuS film to 109 Ω sq^{-1} for In^{3+} doped. Transparency of the conducting films could be tuned by a proper selection of reaction time and In^{3+} concentration. It was found that films containing 10 mol % of In^{3+} ions after reaction duration of 24 hours have sheet resistance ~270 Ω sq^{-1} and transparency ~80 %. The fabricated films are characterized by excellent adhesion to the PET substrate and are shown to be suitable for use as transparent conducting electrodes (TCE) in flexible electroluminescent (EL) devices.

1. Introduction

Thin films of semiconducting metal chalcogenides have gained interest during the past few decades due to immense scientific and technological potential. Copper sulphide is a metal chalcogenide which has various applications including solid state solar cells,[1-2] catalysis,[3] conductive coating,[4-7] dye degradation,[8] electrodes,[9] supercapacitors.[10-12] In warm climatic conditions, CuS films on glass windows act as selective radiation filters.[13] CuS films are also used as counter electrode (CE) for dye and quantum dot sensitized solar cell.[14,15] Cu_xS exists in five stable phases at room temperature, namely chalcocite (Cu_2S), djurleite ($Cu_{1.96}S$), digenite ($Cu_{1.85}S$), anilte ($Cu_{1.75}S$) and covellite (CuS).[16] Cu_xS particles of various sizes and shapes such as hollow spheres,[17] rods,[18] tubes,[19] flowers,[20] plates,[21] disks,[22] and ribbons[22] were successfully deposited on various substrates by various techniques such as atomic layer deposition (ALD),[23] chemical vapor deposition (CVD),[24] metal organic deposition (MOD),[25] successive ionic layer adsorption and reaction (SILAR),[26] spray pyrolysis,[27] chemical bath deposition (CBD),[1,2,28-30] and hydrothermal/solvothermal method.[31] Among these techniques, chemical bath deposition technique is simple, cost effective, ease of doping, large area can be coated and does not require sophisticated instruments. While using this technique, the CuS films can be deposited on polymers (PET, PVA, PTFE, polysulphone), metals, insulators, and semiconductors of any size and shape, whose surface is either modified or used as is.[11,32-34] In most of the reports, the deposition of CuS films was performed in the presence of bases such as NaOH or NH_4OH at a reaction temperature higher than 40 °C, and the films obtained were further annealed in presence of N_2 or in vacuum at 150 to 400 °C.[35,36] The annealing step at high temperature is not suitable for plastic devices.

In addition to a proper conductivity, it is desirable to fabricate flexible films with high transparency to be utilized in optoelectronic devices as transparent conductive electrodes (TCE).[37-39] Nowadays, the most common materials used for the fabrication of such films are metal oxides, while the most widely used oxide being indium tin oxide (ITO), with a market share of more than 97%.[37] However, ITO films have several disadvantages, such as high production cost, the need for special etching processes in order to achieve patterning, and brittleness which prevents their utilization for the fabrication of flexible, stretchable, and bendable devices. Therefore, much effort is being made to find alternatives for ITO, which are based on low cost materials, and which can be deposited directly on various substrates. One of such materials can be CuS.

It has been shown that CuS films deposited on glass modified by organosilane (R-SiX_3, where R is an organic group) at 40 °C using a base such as NH_4OH, followed by post heat treatment at 160 °C, possess sheet resistance of 154 Ω sq^{-1} and optical transparency of 80%.[34] Cardoso et al. have deposited CuS films on Kapton polyimide tape at 150-400 °C under N_2 flow in the presence of ammonia and NaOH, and the films obtained had a sheet resistance of 10–50 Ω sq^{-1}, but the transparency was very low, only 25–35%.[35] Grozdanov et al. have deposited CuS on glass, and the sheet resistance and transparency were found to be 105 Ω sq^{-1} and 60 %, respectively.[36]

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In our work, undoped CuS films and In^{3+} ions doped CuS films on PET were prepared by simple chemical bath deposition with low cost materials at room temperature followed by low temperature (100 °C) sintering that makes a procedure suitable for fabrication of transparent conductive films on heat sensitive substrates. We demonstrated for the first time the effect of In^{3+} ions doping on the sheet resistance, transparency and band gap of CuS films synthesized at room temperature. These films acted as hole transport layers and were used as transparent conducting electrode (TCE) for fabricating electroluminescent (EL) device. They can also find application in the field of solar cells.

2. Experimental

2.1 Materials

Copper acetate monohydrate (99.9%, Sigma Aldrich), indium acetate (99.99%, Aldrich), triethanolamine (98% min., Sigma), thiourea (>99%, Acros Organic), PET (Jolybar) and triple distilled water (TDW) were used as reagents without additional purification.

2.2 Procedure

PET films were cleaned with TDW and purged with nitrogen prior to deposition. One side of the PET films was covered with cello tape so films are deposited only on one side. Stock solutions of copper acetate, indium acetate and triethanolamine were prepared using TDW, and their concentrations were kept 0.4 M. Thiourea solution with the same concentration was freshly prepared each time.

In a typical procedure for deposition of CuS film, the required amount of copper acetate solution and 92.5 mL TDW were added in a 100 mL beaker followed by an addition of triethanolamine solution and finally, freshly prepared thiourea solution. After each addition, the reaction mixture was thoroughly stirred with a glass rod. Final concentration of copper acetate and thiourea was maintained at 2.5 mM and that of triethanolamine was maintained at 25 mM. The PET films were dipped vertically for 24 or 48 hours into a 100 ml beaker without stirring under ambient conditions. Finally, the films were washed thoroughly with TDW to remove unreacted reactants and loosely adhered CuS. To increase the robustness of the film, it was heated in the oven at 100 °C for 1 hour. In case of In^{3+} doped CuS films, the required amount of indium acetate solution was added along with Copper acetate solution. For TEM measurements, the films were deposited on a silicon grids coated with silicon nitride using the same procedure.

2.3 Characterization

TEM, HRTEM, SAED images and EDS were acquired using transmission electron microscope Tecnai F20 G^2 (FEI). The thickness of films was measured by using the Veeco Dektak 150 Surface Profiler. The sheet resistances were measured using a 4-point probe (Jandel HM21, UK). The carrier concentration (n) and mobility (μ), were measured using van der Pauw geometry in a physical property measurement system (PPMS, Quantum Design) at 300 K. The absorbance spectra of the films were measured using a UV-Vis spectrophotometer (Cary 100 model, Varian), and we used the transmittance at 555 nm, as a measure for transparency. The adhesion test was performed by the ASTM D 3359 standard tape test (Elcometer). All the measurement were made at 25±2 °C.

2.4 Fabrication of electroluminescent device

Transparent electrode consists of In^{3+} doped CuS films on PET. Electroluminescent (EL) layers were screen printed on top of it. The first was a layer of ZnS paste on CuS film (Type E80-01HB, MOBI*Chem* Scientific Engineering Ltd.), and then two layers of dielectric paste (Type D80-01, MOBI*Chem* Scientific Engineering Ltd.) were deposited. The samples were dried at 100 °C for 15 min after printing of each layer. The counter electrode was inkjet printed (OmniJet 100 printer, UniJet, Korea) using colloidal silver ink (Xjet Solar 30%) on top of the EL layer. The sintering of the counter electrode was done by immersing the device in 20% NaCl solution for 60 seconds, and then the device was heated in oven at 100 °C for 15 min. EL devices were also fabricated on ITO coated PET using similar procedure.

3. Results and discussions

Films of undoped and In^{3+} doped CuS on PET were prepared at room temperature using Cu^{2+}, In^{3+}, thiourea and triethanolamine. A proposed reaction mechanism is as follows: Cu^{2+} and In^{3+} ions form complex with triethanolamine (TEA) as presented in steps 1 and 2. Then the complex ions combine with thiourea to give In^{3+} ions doped CuS along with triethanolmine and urea as seen in step 3.

$$Cu(CH_3COO)_{2(aq.)} + n(TEA)_{(aq.)} \longrightarrow [Cu(TEA)_n]^{2+}{}_{(aq.)} + 2CH_3COO^{-}{}_{(aq.)} \text{..(1)}$$

$$In(CH_3COO)_{3(aq.)} + n(TEA)_{(aq.)} \longrightarrow [In(TEA)_n]^{3+}{}_{(aq.)} + 3CH_3COO^{-}{}_{(aq.)} \text{...(2)}$$

$$(1-x)[Cu(TEA)_n]^{2+}{}_{(aq.)} + H_2O_{(l)} + x[In(TEA)_n]^{3+}{}_{(aq.)} + (NH_2)_2CS_{(aq.)} \longrightarrow Cu_{1-x}In_xS_{(s)} + n(TEA)_{(aq.)} + (NH_2)_2CO_{(aq.)} + 2H^{+}{}_{(aq.)} \text{.....(3)}$$

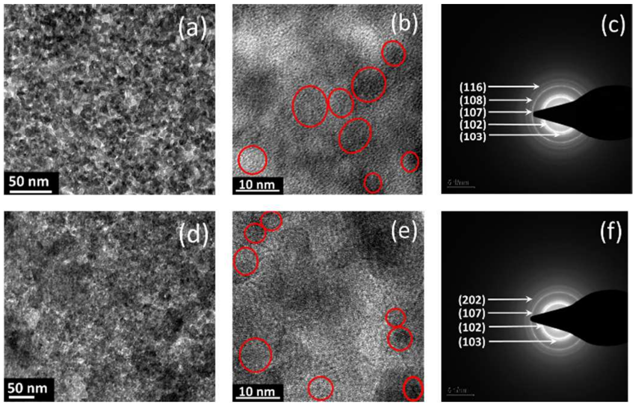

Fig. 1 (a) TEM image, (b) HRTEM image and (c)SAED pattern of undoped CuS films; (d) TEM image, (e) HRTEM image and (f) SAED pattern of 2.5 mol. % In^{3+} doped CuS films on silicon grid (red circles indicate nanocrystallites of undoped (b) and doped (e) CuS).

To evaluate the microstructure of films, CuS films were deposited on silicon grids coated with silicon nitride as described above. Fig. 1a shows the TEM image of undoped CuS films. As seen from the HRTEM image (Fig. 1b), the size of primary CuS particles is in the range of 5–10 nm (indicated by red circles in Fig. 1b). Aggregates with sizes in the range of 50–100 nm are also clearly seen. The SAED pattern (Fig. 1c) reveals that these particles are polycrystalline with d-spacing values corresponding to the covellite structure of CuS. EDS (Fig. S1) confirms the 1:1 stoichiometry of synthesized CuS. Figs. 1d and 1e show TEM and HRTEM images of CuS film containing 2.5 mol. % of In^{3+} ions. The SAED pattern (Fig. 1f) is similar to that for undoped CuS film, testifying to the preservation of covellite structure after incorporation of In^{3+} ions into the lattice, and In^{3+} ions occupy Cu^{2+} site in the lattice. The morphology and average particle size also remain the same as seen in Figs. 1d and 1e (indicated by red circles). EDS of a film prepared in presence of 2.5 mol. % of In^{3+} clearly indicates the presence of In^{3+} ions in the CuS matrix (Fig. S2). The thicknesses of undoped and doped films measured using a profilometer, were found to be in the range of 40–60 nm for a dipping time of 24 hours (Fig. S3).

The observed sheet resistances and transparencies of undoped and In^{3+} doped CuS films on PET obtained at deposition time of 48 hours are shown in Fig. 2. The sheet resistance of undoped CuS films was found to be 1721 Ω sq^{-1}. It is clearly seen that incorporating of In^{3+} ions into CuS films results in abrupt decrease of the sheet resistance to 109 Ω sq^{-1} for films containing 0.75 mol. % of In^{3+} ions. At higher concentrations of In^{3+} ions, the sheet resistance increases and reaches 295 Ω sq^{-1} for films containing 10 mol. % of In^{3+} ions. However, the transparency of these doped films was found to be low, around 30–60%, which means that they are not really applicable in the field of optoelectronic devices. To evaluate the dependence of transparency on the dipping time, the films were dipped for time intervals of 8, 12, 24 and 48 hours. The sheet resistance and transparency of undoped CuS films with various deposition times are shown in Table 1. From the table, it is clear that for an 8-hour reaction time, the transparency is very high but sheet resistance is also high. To reach a good balance between the transparency and sheet resistance, the reaction time was fixed at 24 hours.

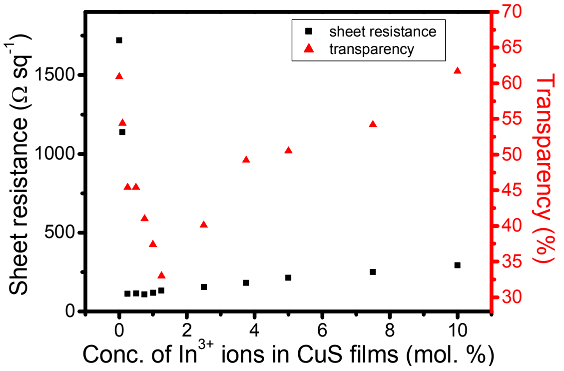

Fig. 2 Sheet resistances and transparencies of CuS films at deposition time of 48 hours as a function of In^{3+} concentration.

Fig. 3 presents the sheet resistance and transparency of CuS films at reaction time of 24 hours as a function of In^{3+} concentration. The sheet resistance abruptly decreases from 1914 Ω sq^{-1} for undoped film to 162 Ω sq^{-1} for film containing 2.5 mol. % In^{3+} ions, and then increases reaching the value of 272 Ω sq^{-1} for film containing 10 mol. % of In^{3+} ions. This is in agreement with results that cationic vacancies are created in CuS lattice by incorporation of In^{3+} ions which occupy Cu^{2+} sites. As In^{3+} ion concentration increases, the number of cationic vacancies also increases that leads to increase in carrier concentration (electrons) and therefore, to increases in conductivity and decrease in sheet resistance. Beyond certain concentration of In^{3+} ions, cationic vacancies carrying negative charge trap the holes, that in turn decreases the mobility of carriers and the conductivity decreases (sheet resistance increases).[40]

Dipping time for undoped CuS films (hour)	Avg. Sheet resistance (Ω sq^{-1}) ± 10 Ω sq^{-1}	Transparency (%) ± 1%
8	2.83×10^6	96.6
12	3177	91.3
24	1914	87.2
48	1721	60.9

Table 1. Sheet resistance and transparency of undoped CuS films obtained at various dipping durations.

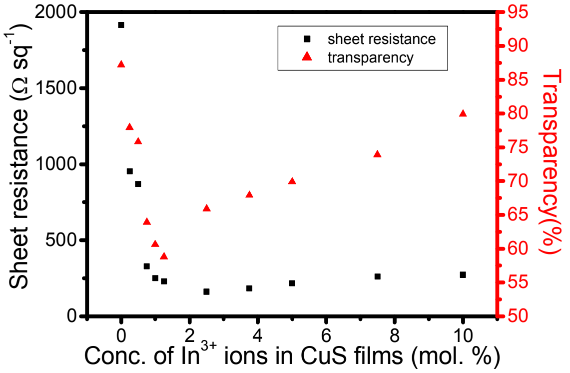

Fig. 3 Sheet resistances and transparencies of CuS films at deposition time of 24 hours as a function of In^{3+} concentration.

To confirm this aspect, Hall measurements were carried out as discusses above and values of carrier concentration and mobility are listed in table 2. The carrier concentration and mobility for 0 % In^{3+} ions doped CuS is found to be 3.6×10^{20} cm^{-2} and 1.5 $cm^2V^{-1}s^{-1}$ respectively. The carrier concentration increases by incorporation of In^{3+} ions as seen in table 2, whereas the mobility increases until 0.25 % In^{3+} ions doped CuS and then decreases. These results are in good agreement with the trend of sheet resistance of these films.

Conc. Of In^{3+} ions in CuS film (mol. %)	Carrier concentration (n) (cm^{-3})	mobility (μ) ($cm^2V^{-1}s^{-1}$)
0.0	3.6×10^{20}	1.5
0.25	4.1×10^{20}	2.7
0.5	4.7×10^{20}	2.5
1.0	2.5×10^{21}	2
3.75	3.0×10^{21}	1.8
7.5	5.3×10^{21}	0.7

Table 2. Carrier concentration and mobility of CuS films with varying concentration of In^{3+} ions.

To further substantiate the study, absorbance spectra in UV-Vis range, which are useful to measure the optical band gap of the material, were studied. Fig. 4 shows the absorbance spectra of CuS films containing 0.0, 0.5, 1.25, 2.5, 5.0 and 10 mol. % In^{3+} ions (dipping time 24 hours). The absorbance shows a red shift with an increase in concentration up to 2.5 mol. % of In^{3+} ions, and further shifts to shorter wavelengths. To evaluate this aspect, the direct and indirect band gaps of CuS films were calculated using the Tauc's plots.[41] The band gaps were calculated by plotting $(\alpha h\nu)^{1/n}$ vs hv, where n = ½ for direct band gap. The extrapolation of the straight line to $(\alpha h\nu)^n = 0$ gives the band gap of the material. The direct band gap of undoped CuS films was found to be 2.71 eV that is higher direct band gap of bulk CuS, possibly a result of quantum confinement effect due to the presence of very small crystallites in the film.[42] The direct band gap of CuS was found to decrease with the an increase in concentration of In^{3+} ions. It decreases to 2.56 eV by incorporation of 2.5 mol. % of In^{3+} ions into the CuS lattice and then increases to 2.69 eV as seen in Table 3.

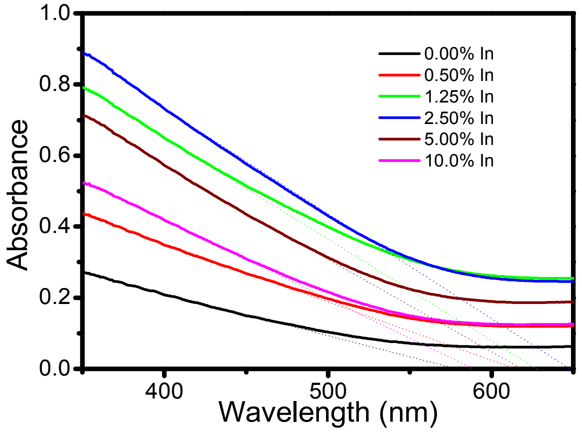

Fig. 4 Absorbance spectra of CuS films containing 0.0, 0.5, 1.25, 2.5, 5.0 and 10 mol. % of In^{3+} ions.

Conc. Of In^{3+} ions in CuS film (mol. %)	Direct band gap (eV) (±0.01)
0.0	2.71
0.25	2.67
0.5	2.65
1.0	2.63
1.25	2.61
2.50	2.59
3.75	2.56
5.0	2.62
7.5	2.65
10	2.69

Table 3. Direct band gaps of In^{3+} doped CuS films obtained at dipping time of 24 hours.

It is worthwhile to mention that the band gap of CuS films was tuned by doping with In^{3+} ions. The direct band gap values show a trend similar to the observed sheet resistance for CuS films: if the band gap of the material decreases, the conductivity increases and sheet resistance decreases.

Both, In^{3+} doped and undoped CuS films on PET showed excellent adhesion, which was tested by the scotch test using ASTM standard tape. It was found that the tape showed no visual changes, and the sheet resistance and transparency of the films before and after the peeling test remained almost unchanged with variations of less than 1%. That makes these films good candidates for fabrication of transparent electrodes for flexible electronic devices. We have demonstrated this by fabricating an EL device. The device was composed of a transparent electrode made of 0.75 mol. % In^{3+} doped CuS on PET, and the electroluminescent layers were screen printed over it. The second electrode was formed by inkjet printing of colloidal silver ink. Fig. 5 shows the images of electroluminescence from a device at different frequencies of electric field and voltage ±100 V. As seen from this figure, the film color changes gradually from deep green to deep blue with the increase in frequencies. The reason for such color changes is excitation of different centers in the electroluminescent material.[43-44] At lower frequencies, the green center is excited, whereas at higher frequencies, the blue center is excited.[44]

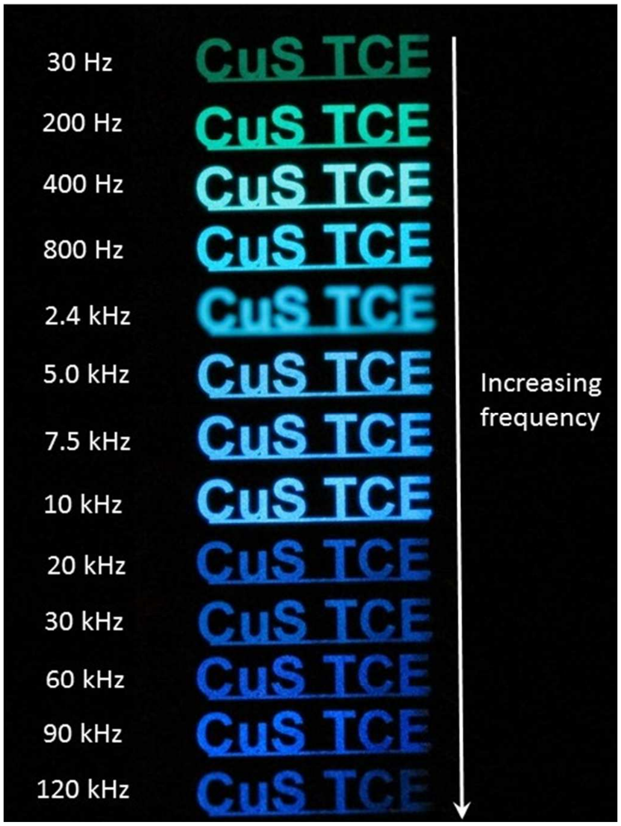

Fig. 5 Images of EL device under different frequencies ranging from 30 Hz to 120 kHz and applied voltage of ±100 V.

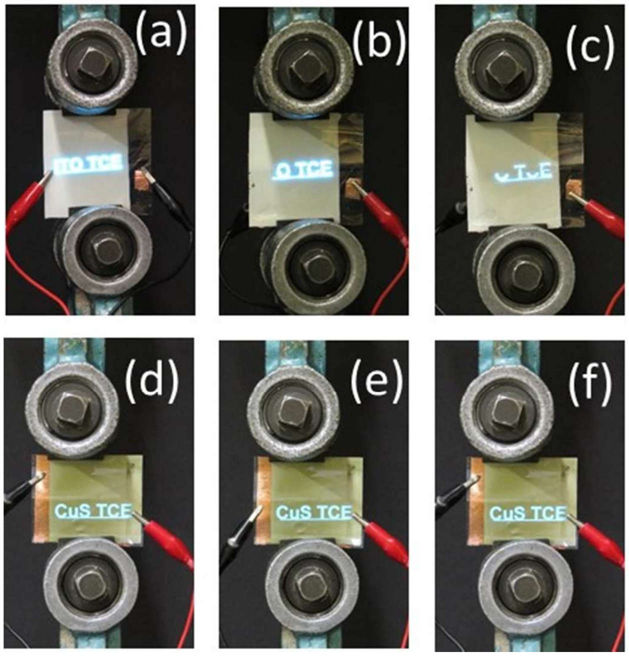

Fig. 6 Photographs of working EL devices with ITO electrodes after (a) 0, (b) 200, and (c) 400 bending cycles and In^{3+} doped CuS electrodes after (d) 0, (e) 400, and (f) 1000 bending cycles.

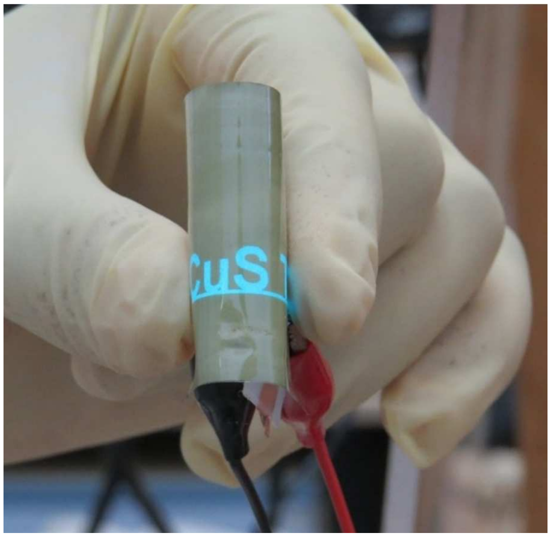

Fig. 7 Photograph of working EL device after 1000 bendings.

These films were also compared with commercial ITO-coated PET films for bending stability. It was found that ITO-based device were damaged after 200 bendings, whereas devices fabricated using In^{3+} doped CuS films were stable upto 1000 bendings (Fig. 6). It is worthwhile to mention that bending of the EL device does not affect the conductivity of the electrode and the luminance as well. Fig. 7 shows the photograph of flexible electroluminescent device after 1000 bendings.

4. Conclusions

Transparent, flexible and conducting films play a key role in the field of optoelectronic devices. Undoped and In^{3+} doped CuS transparent conductive films were prepared at room temperature using the chemical bath deposition technique. The band gap and sheet resistance of CuS films were tuned by incorporation of various amounts of In^{3+} ions into the CuS lattice. Doping with 0.75 mol. % of In^{3+} ions resulted in a decrease in the sheet resistance of CuS films from 1721 to 109 Ω sq^{-1}. Transparency of the films could be improved by decreasing the reaction time from 48 to 24 hours and increasing the concentration of In^{3+} ions to 10 mol. %, resulting in a sheet resistance of 272 Ω sq^{-1} and transparency around 80 %. It was demonstrated for the first time that these films can be used for fabrication of flexible EL devices. Excellent adhesion, low sheet resistance and stability after multiple bendings make these films applicable as flexible transparent conducting electrodes (TCE) for other electronic devices.

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In^{3+} ions doped CuS transparent conducting films were fabricated at room temperature on PET and were used to fabricate a flexible electroluminescent (EL) device.