Journal of Physics and Chemistry of Solids 73 (2012) 1159–1163

Contents lists available at SciVerse ScienceDirect

Journal of Physics and Chemistry of Solids

journal homepage: www.elsevier.com/locate/jpcs

Preparation, characterization and application of Nano CdS doped with alum composite electrolyte

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ARTICLE INFO

Article history:
Received 23 December 2011
Received in revised form
4 April 2012
Accepted 4 May 2012
Available online 23 May 2012

Keywords:
A. Chalcogenides
C. Infrared spectroscopy
C. X-ray diffraction
D. Electrical conductivity
D. Electrical properties

ABSTRACT

A new composite electrolyte has been developed for electrochemical application and studied in details. The system contains Alum doped with nanosize cadmium sulphide (CdS) particles in the desire ratio. The structural studies were carried out by using X-ray diffraction (XRD) as well as infrared spectroscopy (IR) which affirms the composite nature of the system. The electrical properties, including ion transport studies and complex impedance spectra confirm the ionic nature of sample as well as enhancement in ionic conductivity by CdS doping. The temperature dependence measurement confirms the Arrhenius nature of sample, which is commonly observed in the ionic composite system. The dielectric constant varies with temperature, and this data is used to calculate the number of charge carrier (n/n_o) contributing to conductivity and fits well with emf variation. A cell was fabricated by sandwiching the composite between graphite and stainless steel electrodes, which shows an emf of 7 mV.

1. Introduction

The composite electrolytes are dispersed two phase or multi-phase solid systems, generally obtained by physical mixing of different ionic conductors with a second phase material, which may be either insulating (like, Al_2O_3, SiO_2, Fe_2O_3) or another poor ionic conductor (AgBr, AgCl, KCl, etc.). In year 1973, Liang was the first to observe a remarkable enhancement in the conductivity (100–1000 times) of Li^+ conductivity in LiI-Al_2O_3 system [1]. Since then, a lot of work in this field has been done [2–5]. Based on the nature of host matrix and the dispersoid, composites can be broadly classified into four categories [6]

(i) Crystal–Crystal Composites
(ii) Crystal–Glass Composites
(iii) Polymer–Glass Composites
(iv) Polymer–Crystal Composites

In the present paper, we are concentrated around a novel system containing semiconductor (CdS) doped with hydrated salt (Alum) which falls under the category of crystal–crystal composite.

The commonest alum is a white crystalline powder material with double sulphate of potassium and aluminum, $K_2Al_2(SO_4)_4 \cdot 24H_2O$. It is used in curing animal skins. Other alums are used in papermaking and to fix dyes in the textile industry. The raw material of manufacture of common alums is alum rock, composed chiefly of alum stone.

In crystal–crystal composite the first-phase host-matrices are moderate ionic solids, like silver halides, copper halides, etc.; whereas, the second phase dispersoid is either another ionic solid (such as AgCl or AgBr in AgI) or an inert and insulating material (such as Al_2O_3, SiO_2, ZrO_2, fly-ash, etc.). In case of dispersion of insulating and inert second phase material, it has been found that the smaller the particle size, the larger the conductivity enhancement. The reason suggested for this effect is the increased surface area of the dispersoid particles [7–11]. Following the same strategy we have used alum as a host matrix and nanosize CdS (prepared in own laboratory) as dispersoid. Using maximum conductivity composition we have fabricated a solid-state cell using the same electrolyte.

2. Experimental

2.1. Preparation of the samples

Commercially used alum was recrystallized before use and taken as host material while laboratory prepared CdS was taken as dispersoid. The semiconductor CdS was prepared by chemical

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0022-3697/$ - see front matter © 2012 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.jpcs.2012.05.008

precipitation method in which sulphur exchange reaction of cadmium nitrate and sodium sulphide Na_2S as a precursor source. Detail preparation and characterization of the CdS are reported elsewhere [12]. The ion conducting composites having the different amount of CdS were then prepared by mechanical grinding. The obtained powders of different compositions (Alum+x wt% CdS) were then palletized under a pressure of ca. 5 t/cm^2. All the pellets were of fix area but of different thickness. The samples then were subjected to various characterization techniques. For electrical characterization of the pellets, the conducting silver paste was painted on either side of the pellet and dried at room ambient.

2.2. Characterization of sample

2.2.1. X-ray diffraction (XRD)

The XRD method is used to check the composite nature of CdS doped Alum. In the present study, we have used X-ray diffractometer (Rigaku D/max-2500) at a scan rate of 1° min^{-1}.

2.2.2. Infrared spectroscopy (IR)

The mixing of alum doped with CdS resulting in the formation of a complex is reflected in the IR spectra as either a new peak or shifting in the peak positions of the alum/CdS. The IR spectra of complex samples were recorded by Perkin Elmer (880 model) spectrophotometer. In case of powder samples potassium bromide KBr in 100:1 ratio is mixed with CdS and very thin pellet is prepared.

2.2.3. Ion transport Studies

The total ionic transference number of the samples was measured by the polarization method. A small dc voltage was applied, and the current decay was monitored. Using the initial and final value of the current, t_{ion} was calculated as

$$t_{ion} = (i_{initial} - i_{final})/i_{final}$$

2.2.4. Electrical conductivity Studies

Complex impedance spectroscopic techniques were used to evaluate the ionic conductivities of the solid electrolyte composite pellets. The pellets were sandwiched between two polished stainless steel (SS) electrodes. The Ionic conductivities of the solid pellets were calculated from bulk resistance estimated from the by ac complex impedance plots obtained using Hioki LCR HI tester bridge at frequency 100 kHz. The conductivity (σ) was calculated using the relation $\sigma = G$ (l/A), where l and A are the thickness of the prepared polymer film and area of the electrodes respectively, and G is conductance of sample which is reciprocal of bulk resistance (R_b) obtained from the intercept on the real Z-axis of the impedance data in the complex plane.

3. Results and discussion

3.1. X-ray diffraction studies

The X-ray diffractogram of pure CdS, pure alum and two different compositions of the composite are shown in Fig. 1. It is clearly observed in Figs. 1b, 1c that the peak due to (111) plane of cubic CdS appeared at $2\theta \sim 26^\circ$ and to (221) plane of CdS occur at $2\theta \sim 46^\circ$ is clearly present in composite samples. Some other peaks of CdS are also observed as submerged in the composite. All peaks of the host materials were clearly seen in composite. In addition, no new peak was observed, which confirms that the present system shows composite nature of sample.

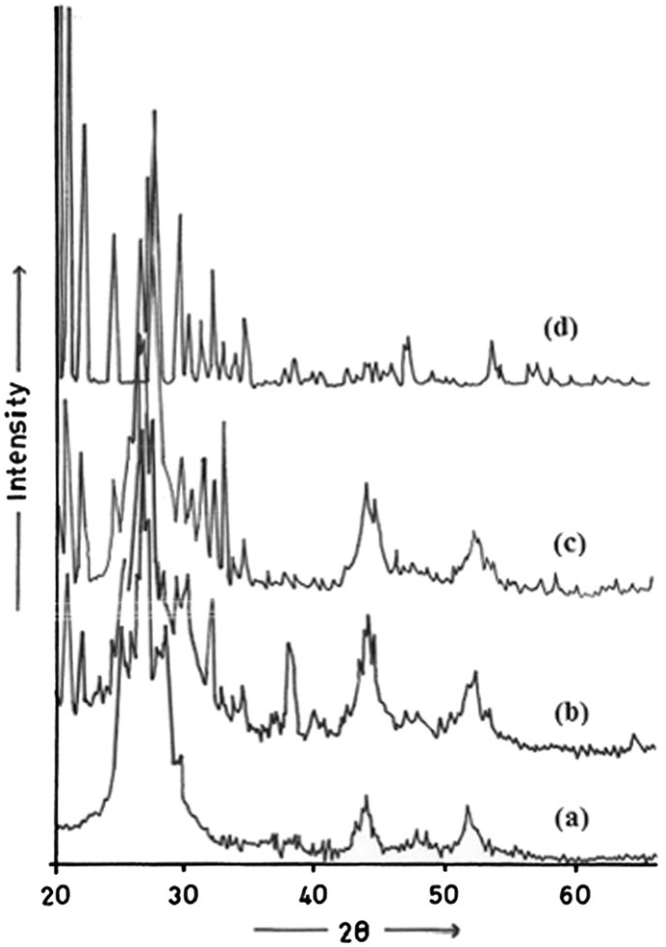

Fig. 1. X-ray diffractogram of (a) pure alum, (b) and (c)composite and (d) pure CdS.

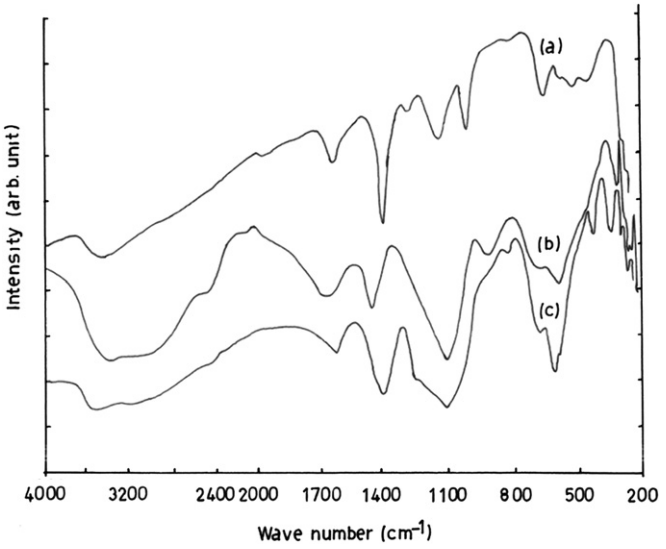

Fig. 2. Infrared Spectra of (a) Pure alum (b) Composite and (c) Pure CdS.

3.2. Infrared absorption studies

The infrared spectra of pure CdS, pure alum and the composite are shown in Fig. 2. In all the samples, presence of the water could be seen clearly by the OH bands near 1600 nm and 3300 nm.