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Recycling indium from waste liquid crystal display panel by vacuum carbon-reduction

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HIGHLIGHTS

- The recovery and reuse of indium in waste LCD panel is essential for conservation of resources and environmental protection.
- We recycled indium from waste liquid crystal display panel by vacuum carbon-reduction.
- There is no hazardous materials produced in this process.

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ABSTRACT

This study investigated the recovery of indium from waste liquid crystal display (LCD) panel using vacuum carbon-reduction. First of all, high purity In_2O_3 was investigated. The results indicated that indium can be reclaimed from In_2O_3 using vacuum carbon-reduction in thermodynamics and dynamics. The conditions of 1223 K, 50 wt% carbon addition, 30 min, and 1 Pa were confirmed as the optimal conditions for pure In_2O_3 and high purity indium could be selectively recovered on condensing zone. Based on this, the experiment of the recovery of indium from waste LCD power was performed. The best parameters were confirmed as 1223 K and 1 Pa with 30 wt% carbon addition for 30 min. The recovery rate of indium from LCD powder could reach to 90 wt%. No hazardous materials produced in this process. Therefore, this technique provides the possibility of reutilization of LCD in an environmentally friendly way.

1. Introduction

With the advantages of small volume, light quality and low power consumption, LCD has replaced cathode ray tube (CRT) display in most of appliances. The total global TV shipments were 233 million in 2012, while LCD TV sheared 87.3%, plasma display panel (PDP) TV and CRT TV shared 5.7%, 6.9%, respectively, according to the latest finding published in the NPD Display Search [1]. Besides, the increase in demand for high-end products, such as smart phone, tablet PC and digital product, is considered to drive the flat panel display shipments. Taking account of the short life-cycle, 3–5 using years in general, and the endless requirement for new products, a large number of LCD products are coming into discarding period.

Indium–tin oxide (ITO), a solid solution of indium(III) oxide (In_2O_3) (90–95%) and tin(IV) oxide (SnO_2) (10–5%), is widely used as transparent conductive film in LCD because of its significant properties, such as electrical conductivity and optical transparency. Indium consumption in ITO accounts for over 70% of its total consumption and the greatest demand on ITO is LCD nowadays. However, the global reserve of indium is about 16–19 thousand tons and is only 1/6 of that of gold. In addition, the average content of indium in Zinc blende as the most important indium-carrier mineral ranges from less than 1–100 ppm, while that is about 250 ppm in LCD [2]. Therefore, the recycling and reuse of indium from waste LCD become particularly important.

Various methods for recycling of indium from secondary sources containing indium, particular ITO sputtering waste, have been investigated, mainly based on the hydrometallurgy [3,4], which involves leaching, concentrate and separation, and electrorefining. Among them concentrate and separation involve precipitation, cementation, and solvent extraction. In general, the recovery rate of indium is high through hydrometallurgy. However, the using of various solvents, including corrosive acid and hazardous extraction substances, increases the potential environmental risks.

The recycling of indium scrap has been investigated from ITO through pyrometallurgy, but few from end products. In_2O_3 can be reduced to metallic indium under high temperature condition with reducing atmosphere [5], such as H_2, C. Alloy of indium and tin can be recovered. Pure metallic indium could be separated form alloy through vacuum. Satoshi and Katsuya [6] conducted

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laboratory scale experiments for the recycling of metallic indium from ITO scrap. The process consisted of two stages, reduction of ITO with CO at 1073 K and vaporization of In–Sn alloy at 1373 K under vacuum. Metallic indium drops could be recovered on condensing zone. This method requires high temperature for the separation of indium from In-Sn alloy. Besides the use of CO is a potential safety risk as its asphyxiating.

In addition to leaching-extraction and pyrometallurgy, some researchers have reported the chlorination process of waste LCD panel for the recycling of indium. Takahashi et al. [7] presented a method of recovering indium from waste LCD panel by chloride-induced vaporization. In this process, crushed LCD panel was first treated in HCl solution for altering the indium(III) oxide into a chloride-induced indium compound. Then the chloride-induced indium compound was vaporized at relatively low temperature (673 K) in nitrogen atmosphere. The vaporization rate of indium could reach 84.3 wt%. In addition, chlorinated separation of indium from ITO scrap or LCD panel was also investigated by other researchers [8,9]. Comparing with pyrometallurgy, chlorinated separation has relative lower temperature. However, it needs a lot of additive and subsequent purification procedure for metallic indium. These two methods are still in laboratory stage. Therefore, in order to directly recover metallic indium from waste LCD panel, environmentally friendly methods and high efficiently methods for achieving high purity metallic indium are desired.

Vacuum metallurgy has been widely used in non-ferrous metal smelting [10]. In addition, this method is also reported to separate heavy metal lead from funnel glass [11]. However, there is no study was reported on how this technique is used to recover indium from waste LCD panel. In this paper, we study the feasibility of recycling of metallic indium from In_2O_3 through vacuum carbon-reduction at relative low temperature. Based on the feasibility experiments, the method of vacuum carbon-reduction is applied to recycle indium from discard LCD panel in experimental scale. Here coke power is chosen as reducing agent due to its safety, low price and high calorific value.

2. Materials and methods

2.1. Materials

LCD panels were firstly dismantled from discarded computers by hand, and then polymer films (mainly contain (polyvinyl alcohol) PVC and (Triacetyl Cellulose) TAC) were also removed through manual from these panels. Liquid crystal between two glasses was dissolved by acetone. Take TFT-LCD for example, the main structure of LCD panel is shown in Fig. 1. Last remained glass panels were crushed into power and sieved to smaller than 0.3 mm. Coke powder (0.8 mm, carbon content >80%) were prepared as reductant. Besides In_2O_3 (99.99 mass% purity) were also prepared in this experiment.

2.2. Apparatus

Experiments were carried out in a self-assemble vacuum furnace. The schematic diagram of experiment system is shown in Fig. 2. The main body consisted of tubular electric resistance furnace (chamber dimensions is Ø40 mm × 600 mm), quartz tube reactor (Ø35 mm × 900 mm) and vacuum pump (power is 0.25 kW). Middle part of furnace is heating zone and in the outside of it temperature is gradually decrease with the distance from heating zone, and the end of furnace is condensing zone.

2.3. Methods

Sample and carbon powder were blend well with a certain mass ratio in a quartz boat, and then the quartz boat with mixture was put into a quartz tube. The quartz tube was pushed into furnace heating area, and heated up to experimental temperature in nitrogen atmosphere. After system reached set temperature, it was evacuated to 1 Pa by vacuum pump. Temperatures examined were 1073, 1123, 1173, 1223 and 1273 K. After some definite time, the sample was cooled to room temperature under vacuum atmosphere and

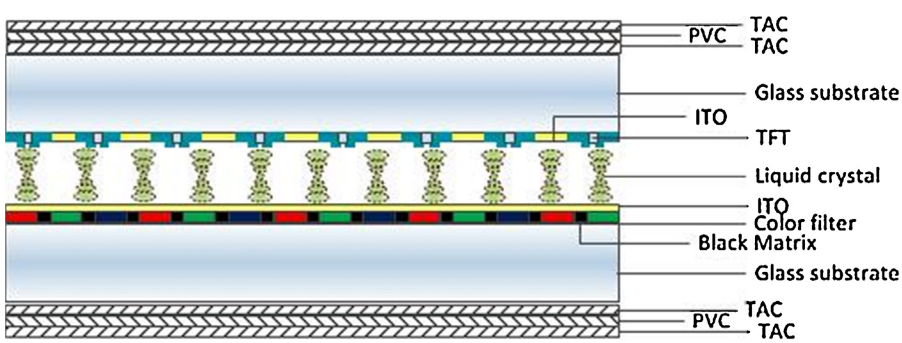

Fig. 1. Structure of TFT-LCD panel.

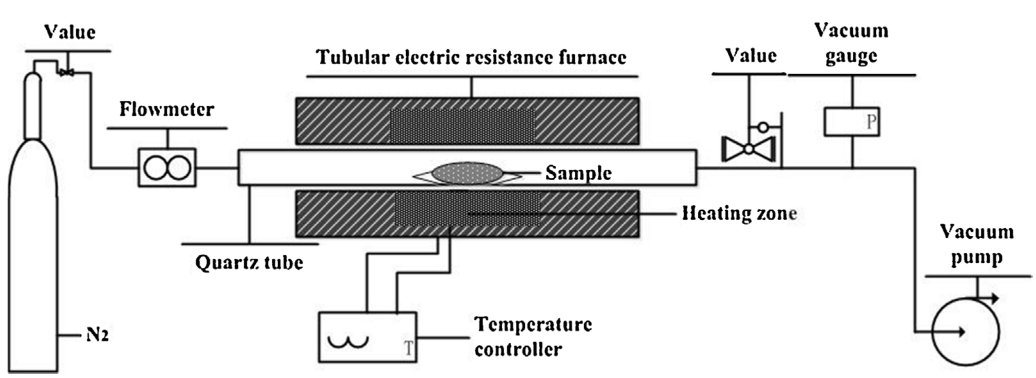

Fig. 2. Schematic diagram of the experimental setup.