



Original article

Synthesis and properties of ionic conduction polymer for anodic bonding



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ARTICLE INFO

Article history:

Received 15 August 2014

Received in revised form 7 October 2014

Accepted 22 October 2014

Available online 5 November 2014

Keywords:

Packaging

Polyethylene oxide

Anodic bonding

Solid electrolyte

ABSTRACT

In this study, powders of polyethylene oxide (PEO) and lithium perchlorate (LiClO_4) were used as the raw materials for producing the ionic conduction polymer PEO– LiClO_4 with different complex-ratios and used for anodic bonding through high energy ball milling method, and meanwhile, X-ray diffraction, differential scanning calorimetry (DSC), ultraviolet absorption spectrum test analysis, and other relevant methods were adopted to research the complexation mechanism of PEO and LiClO_4 and the impact of the ionic conduction polymer with different complex-ratios on the anodic bonding process under the action of the strong static electric field. The research results showed that the crystallization of PEO could be effectively obstructed with increased addition of LiClO_4 , thus increasing the content of PEO– LiClO_4 in amorphous area and continuously improving the complexation degree and the room-temperature conductivity thereof, and that the higher room-temperature conductivity enabled PEO– LiClO_4 to better bond with metallic aluminum and have better bonding quality. As the new encapsulating material, such research results will promote the application of new polymer functional materials in micro-electro-mechanical system (MEMS) components.

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1. Introduction

With the development of microelectromechanical systems (MEMS) technology, more and more materials have been applied to MEMS in order to achieve effective microsystems [1]. Encapsulation is one of the key technologies for the industrialization of MEMS technologies since the reliability of MEMS components greatly depends on the encapsulation quality [2]. At present, the bonding technology is one of the most challenging technologies among MEMS technologies, and the relatively mature bonding technologies include anodic bonding technology, silicon-silicon bonding technology, gold-silicon co-melting bonding technology, aluminum-silicon co-melting bonding technology, and cold welding bonding technology, of which anodic bonding technology has been widely applied [3]. Anodic bonding can achieve solid connection between glass and silicon or metals, as well as the alloy thereof, under the action of a static electric field, and such technology also offers a simple process, high bonding strength, good sealing performance, etc. [4]. However, anodic bonding requires the encapsulating materials to have ionic conduction in order to ensure the mutual

diffusion between positive and negative ions under the action of the electric field during the bonding process. As the cathodic bonding material, only Pyrex7740 glass produced by US Corning Corporation can meet the bonding requirements of the present anodic bonding process. As a kind of solid electrolyte material, Pyrex7740 glass contains alkali metal ions, thus basically satisfying the ionic conduction requirement [5]. However, Pyrex7740 glass can achieve ideal bonding only under high bonding temperature that accordingly causes great bonding stress and damages the front-end process and the structure of MEMS components; meanwhile, Pyrex7740 glass is high in cost. Solid polymer electrolyte (also called ionic conduction polymer) is a kind of new solid electrolyte material developed in recent years [6]. Because solid polymer electrolyte materials feature ionic conductivity, viscoelasticity, small thermal expansion coefficient, workability, etc., both the bonding temperature and the residual encapsulation stress can be reduced during the bonding process, thus improving the encapsulation quality of MEMS components [7]. At present, there is no research on the encapsulation achieved through the bonding of solid electrolyte polymer materials and metals, thus limiting the application of polymer functional materials in MEMS components [8]. Therefore, the research results of this paper are significant for expanding the application of anodic bonding technology, developing new uses for polymer materials, and promoting the development of MEMS.

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2. Experimental

Preparation of PEO–LiClO₄ used for anodic bonding: Raw materials of this experiment: solid polyethylene oxide (PEO) powder with relative molecular mass of 5,000,000, purity >99.36%, granularity <80 μm, which was pre-dried in vacuum at 50 °C; LiClO₄, analytical reagent, purity >99.6%, granularity <50 μm, which was pre-dried in vacuum at 150 °C. The above two powders were put into a ball milling tank in different proportions with absolute ethyl alcohol as the grinding agent and agate balls 8 mm, 5 mm, and 3 mm in diameter as the ball milling media. The proportion of the three kinds of balls was 1:1:1, the proportion of the balls and the materials was 8:1, the rotating speed was 280 rpm, the ball milling time was 12 h, and the ball milling operations were completed on a QM-3SP planetary high-energy ball mill. After the ball milling process, the powdered material was dried and sieved to obtain a fine powder, which was then pressed into a round sheet with a diameter of 25 mm and the thickness of 3 mm in a cylinder type mold cavity.

Anodic bonding experimental device: The device for the anodic bonding experiment of PEO–LiClO₄ and metallic aluminum is shown in Fig. 1, and the bonding device is mainly composed of a high voltage power supply, bonding room, and a heating and temperature-regulating device, wherein the bonding room is internally equipped with a heating plate, lower and upper electrodes, an assembling platform, bonding power supply, and a temperature-regulating cabinet; a 10–1000 V continuously regulated DC pulse bandwidth filtering power supply is adopted as the bonding power supply; TRC current protection, microcomputer data collection, as well as an arithmetical unit are also configured; a SiC heating element and thermocouple temperature monitor are adopted for the heating furnace; and a triaxial parallel locating device and three-dimensional adjustment machinery are adopted for the assembly platform.

3. Results and discussion

3.1. XRD analysis

According to XRD analysis, increasing the content of the lithium salt has great impact on the conductivity of the polymer electrolyte; here Fig. 2(a) is the XRD Graph of PEO, Fig. 2(b) is the XRD Graph of PEO–LiClO₄ generated by the ball-milled PEO and LiClO₄ with the mass content of 5% through complexation reaction, and Fig. 2(c) is the XRD Graph of PEO–LiClO₄ generated by the ball-milled PEO and LiClO₄ with the mass content of 10% through complexation reaction. The scanning scope (2θ) is 10–50°. Two obvious diffraction peaks respectively appear at 2θ = 19° and 2θ = 23° in the graphs. According to the analysis, the addition of lithium salt does not change the position of the diffraction peak,

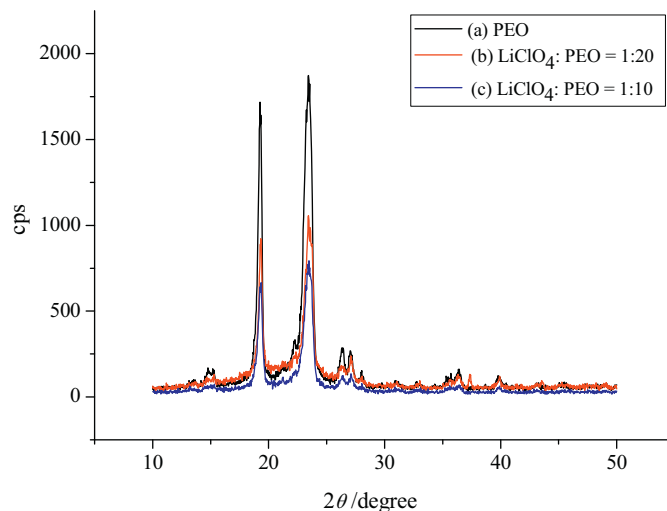


Fig. 2. XRD spectra of PEO–LiClO₄.

but compared to PEO, the strength of the diffraction peak thereof is reduced and the strength of the diffraction peak is more greatly reduced along with the increment of LiClO₄. Since the conductivity of the polymer electrolyte mainly relies on the proportion of the amorphous phase high-elastic area, such phenomenon indicates that the increment of LiClO₄ can effectively obstruct the crystallization of the solid polymer electrolyte, thus increasing the content of the amorphous area and favorably improving the room-temperature conductivity thereof.

3.2. DSC analysis

According to DSC results shown in Fig. 3(a), the melting temperature of the pure PEO is 80 °C, and after adding LiClO₄ and PEO for complexation, the thermal properties of PEO significantly change. Fig. 3(b) is the DSC curve of PEO–LiClO₄ generated by the ball-milled PEO and LiClO₄ with the mass content of 5% through complexation reaction, and Fig. 3(c) is the DSC curve of PEO–LiClO₄ generated by the ball-milled PEO and LiClO₄ with the mass content of 10% through complexation reaction. An obvious peak is located at the position of 74 °C in Fig. 3(b) while an obvious peak is located at the position around 65 °C in Fig. 3(c), which is regarded as the transition point of PEO from the crystalline state to the amorphous

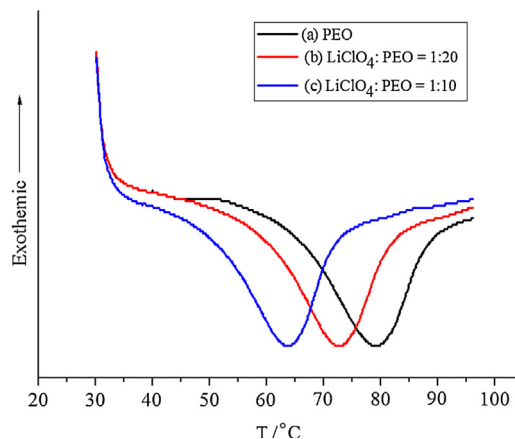


Fig. 3. DSC of PEO–LiClO₄.

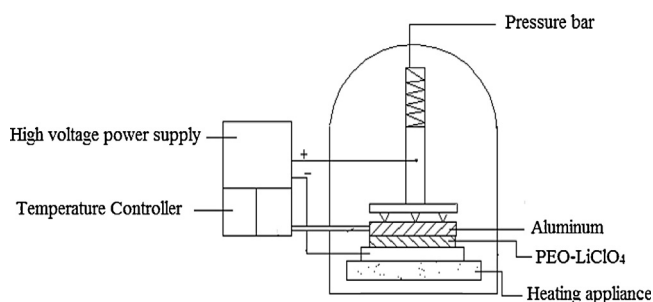


Fig. 1. Schematic diagram of anodic bonding device.

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