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Vacuum Deposition and Polymerization of Acetylene Substituted Fluorenes

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2-Ethynylfluorene and 2,7-diethynylfluorene were prepared and tested for the vacuum deposition polymerization. The deposited solid films were heat treated to induce polymerization in which the acetylene group is involved in the polymerization step. Photoluminescence of the resultant films was examined by UV-vis and fluorescence spectrophotometer.

Keywords: vacuum deposition polymerization; ethynylfluorene

INTRODUCTION

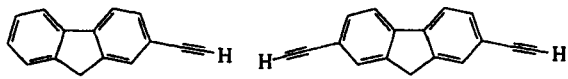
An efficient and thermally stable emitting and hole transport layers are critical to the electroluminescence devices. Although many aromatic chromophores can serve as excellent hole transport or emitting materials,

they are lack of thermal stability, which results in device degradation. Vacuum deposition polymerization is a useful technique to produce thin films of polymers, which have been tested for the preparation of piezoelectric sensors[1,2] and intermetallic dielectric layers in semiconductor devices.[3,4] This technique has advantages over the conventional spin coating process such that no solvent is used during the process and contamination by dust can be minimized.

In this letter, we report the preparation of polymer thin films using postpolymerization of vacuum deposited acetylene substituted fluorenes.

RESULTS AND DISCUSSION

Acetylene substituted fluorenes were synthesized from the corresponding fluorene iodide or bromide with trimethylsilylacetylene followed by desilylation reaction using KOH methanol solution and the structure of the 2-ethynylfluorene (**1**) and 2,7-diethynylfluorene (**2**) used in this study are shown below.



1 **2**

DSC of **1** and **2** are shown in Figure 1 (a). Exothermic peaks which are corresponding to the polymerization are found to be at 225℃ and 198℃ for **1** and **2**, respectively. After vacuum deposited on the substrate, **1** and **2** were polymerized by heat treatment at 210℃ for 20 min. under N_2 atmosphere. TGA thermogram of the resulting poly(2-ethynylfluorene) (**3**) and poly(2,7-diethynylfluorene) (**4**) are also shown in Figure 1 (b). Thermal decomposition of **3** and **4** started at 208℃ and 385℃, respectively. Thermal stability of polymers **4** was better than that of **3**.

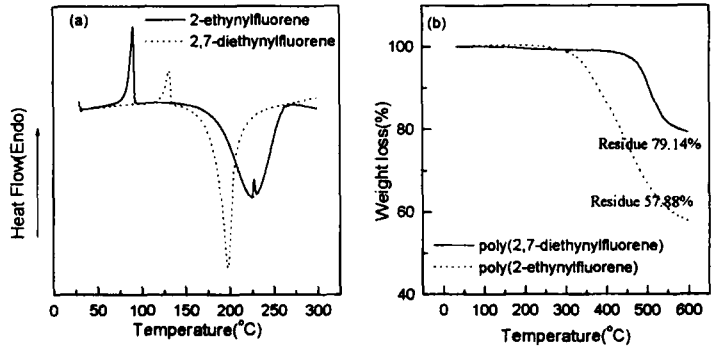


FIGURE 1. DSC (a) and TGA (b) thermogram of acetylene substituted fluorenes and their polymers.

FT-IR spectra of acetylene substituted fluorenes and their polymers are shown in Figure 2. Before heat treatment, **1** and **2** had a strong peak at 3283 cm^{-1} which corresponds to the ≡C-H stretching vibration and a weak peak at 2099cm^{-1} which corresponds to C≡C stretching vibration. After heating, e.g. polymerization, the peaks at 3283cm^{-1} and 2099cm^{-1} related to the acetylene group were disappeared and new peaks at 1600-

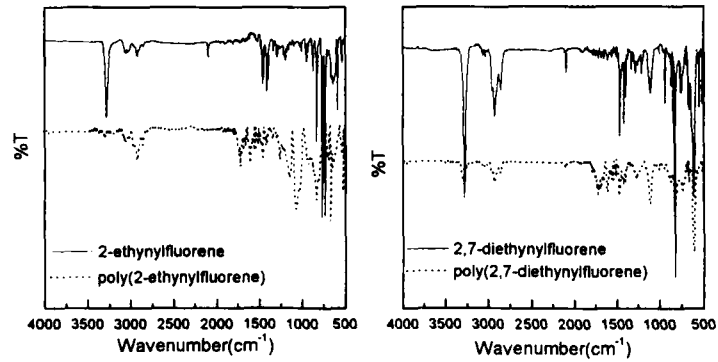


FIGURE 2. FT-IR spectra of acetylene substituted fluorenes and their polymers.

1700cm^{-1} which correspond to double bond were appeared.

UV-vis absorption and fluorescence spectra of **3** and **4** are shown in Figure 3. UV maximum of **3** and **4** were found to be at 318nm and at 320nm, respectively. Very weak fluorescence was observed for both poly(2-ethynylfluorene) and poly(2,7-diethynylfluorene). UV and fluorescent spectra of **3** and **4** are red shifted compared to that of **1** and **2**.

In summary, thermally stable poly(2-ethynylfluorene) and poly(2,7-diethynylfluorene) which can be used as emitting materials were prepared by means of vacuum deposition polymerization. Degradation temperatures of **3** and **4** were at 208 and 385℃, respectively

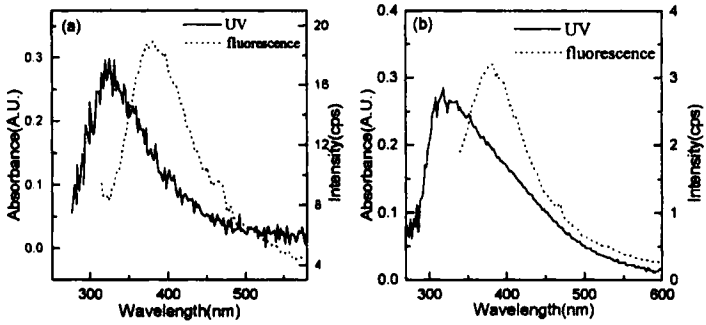


FIGURE 3. UV and fluorescence spectrum of poly(2-ethynylfluorene) film (a) and poly(2,7-diethynylfluorene) film (b).

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