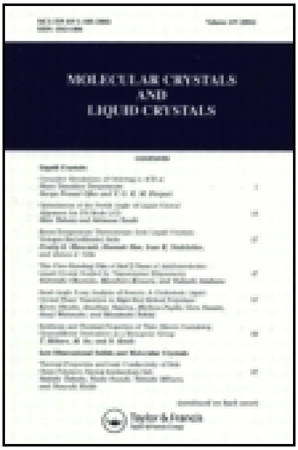

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Preparation and Properties of One-Dimensional C_{60} Nano-Structure in a Zeolite FSM-16

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Preparation and Properties of One-Dimensional C_{60} Nano-Structure in a Zeolite FSM-16

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C_{60} molecules were embedded in the pores of a zeolite FSM-16 by using a liquid phase method. The shift of photoluminescence spectra of C_{60} solid embedded in FSM-16 was discussed using data of ESR, PDS and SAXS.

Keywords: nanostructure; one dimensional C_{60} solids; FSM-16; photoluminescence; ESR; PDS; SAXS

INTRODUCTION

Many kind of nanostructures such as nanocrystalline, porous silicon, carbon nanotubes and quantum dots have been prepared and studied. We have been interested in a zeolite FSM-16 [1,2] as a host material for the nanostrucured host/guest composites which has one dimensional periodic pores of a diameter of 2.7 nm. As a guest materials, we have choosed C_{60} because C_{60} becomes semiconductors in the solid phase and can be vaporized at low temperature such as 400 ℃, and also be dissolved in organic liquids. Therefore it is possible to prepare one dimensional C_{60} nanostructure by embedding C_{60} molecules in the pores of FSM-16 with the vapor phase method and also by the liquid phase method.

Because the vapor phase method was used before for embedding C_{60} in pores of FSM-16 [3], we have tried the liquid phase method in this paper.

SAMPLE PREPARATION AND EXPERIMENTAL METHODS

In the liquid phase method, we used C_{60} as a guest material and FSM-16 as a host material. After the desorption of gases in pores of FSM-16 by a heat treatment at 500 ℃ for 8 hours in a vacuum of about 10^{-5} torr in an ampoule made by pyrex glass [3], the pores of FSM-16 in the ampoule was soaked with a few drops of the C_{60} saturated toluene using a pipet. After the soaking with the saturated liquid , the sample was heated in a vacuum to evaporate the organic solvent toluene at 200 ℃ for 30 minuits. These soaking of the saturated liquid and the desorption of the solvent were continued for ten times to get the brown colored sample of C_{60} embedded in FSM-16, which is written in a simplified form as C_{60}@FSM-16 in this paper.

Photoluminescense spectra of the sample were obtained at 77 K and at room temperature (RT) with an excitation by the argon-ion laser mainly of 488 nm [3,4]. Photothermal deflection spectra (PDS) were obtained to get absorption spectra of C_{60} films, FSM-16 and C_{60}@FSM-16 using the perfluorohexane as a deflection medium [5,6]. For the measurement of PDS of C_{60}@FSM-16, powder-like C_{60}@FSM-16 was fixed on the glass substrate to get a rather flat surface to deflect the laser beam. ESR was measured at RT with using X-band of JEOL;JES=FE1X. X ray diffraction and small angle X ray scattering pattern (SAXS) was obtained by using a system of Rigaku-Denki Co. at RT.

EXPERIMENTAL RESULTS AND DISCUSSION

Photoluminescence (PL) spectra of a C_{60} film and C_{60}@FSM-16 measured at 77 K are shown in fig. 1. Two PL peaks of a C_{60} film at 1.49 and 1.66 eV shift to higher energy of 1.51 and 1.69 eV for C_{60}@FSM-16 . The amount of the PL shifts of 30 and 20 meV for C_{60}@FSM-16 made by the liquid phase method are identical to that made with the vapor phase method by Umehara et al. [3]. These shifts can be attributed to the energy shifts by the quantum size confinment of electrons in the nanostructured

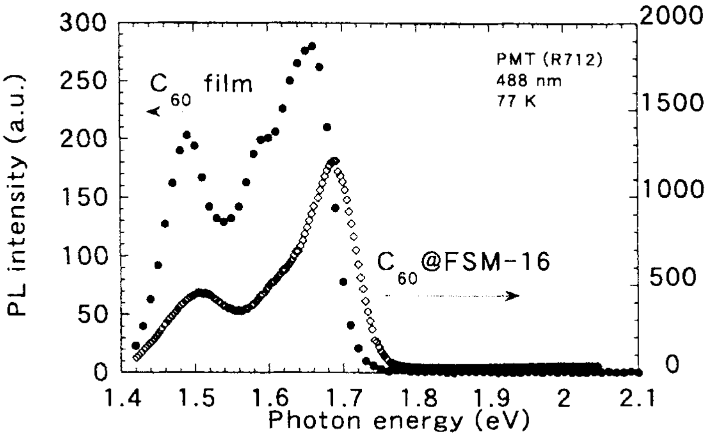

FIGURE 1. **Photoluminescence (PL) spectra of a C_{60} film and C_{60}@FSM-16 measured at 77 K.**

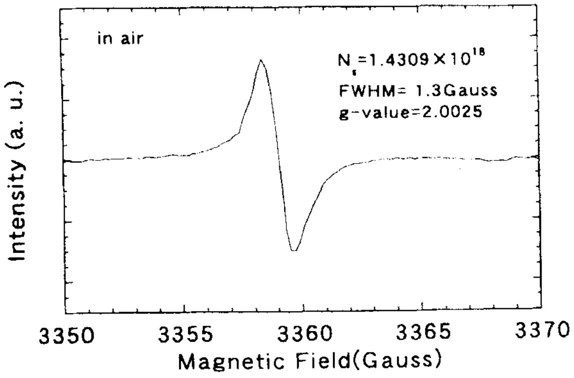

FIGURE 2. **Electron spin resonance of C_{60}@FSM-16 at room temperature.**

semiconductor C_{60} embedded in the fine pores of the diameter 2.5 nm in FSM-16 [3]. This result is one of the verification of embedding C_{60} in the pores of FSM-16, though the filling factor of C_{60} molecules in the pores of FSM-16 has not been obtained yet at present. PL spectra of C_{60}@FSM-16 do not depend on on the excitation wavelength of argon ion laser at 476.5, 488, 496.5, 501.7 and 514.5 nm. Shifts of PL spectra at room temperature (RT) is smaller than that at 77 K.

Electron spin resonance of C_{60}@FSM-16 is shown in fig.2. The g-value of C_{60}@FSM-16 is 2.0031 which is larger than the g-value 2.0028 for a C_{60} film of and that of 2.0025 for FSM-16. Electron spin density and FWHM are 1.4×10^{18} cm^{-3} and 1.3 Gauss respectively. The difference of g-values is also one the indirect verification of the embedding of C_{60} in FSM-16.

Data of X ray diffraction and SAXS are shown in fig. 3 (a) and (b). The data of SAXS of FSM-16 and C_{60}@FSM-16 are used to confirm the periodic honeycomb structure of FSM-16. In fig.3, (100), (110), (200) and (210) of the periodic honeycomb structure are observed. The intensity of SAXS and X ray diffraction pattern for (210) of the honeycomb structure in FSM-16 is almost missing in that of C_{60}@FSM-16 which may be the indirect verification of the embedding of C_{60} in FSM-16, though the origin of the decrease of the scattering for (210) is not clear at present.

PD spectra of PDS for FSM-16, a C_{60} film and C_{60}@FSM-16 are shown in fig.4. PD spectrum of FSM-16 is similar to that of the pure SiO_2 films such as made from the tetraisocyanate silane [$Si(CNO)_4$; TICS] [7]. Several peaks of FSM-16 at lower energy than 1 eV are corresponding to defects and impurities in SiO_2. C_{60} solid film shows an Urbach tail about 44 meV as the same order as that obtained by the constant photocurrent method (CPM) [8] and by PDS before [6]. Absorption of C_{60}@FSM-16 is showing an Urbach energy of 62 meV which is about 1.5 times larger than that of C_{60} films. The increase of the Urbach energy in C_{60}@FSM-16 is showing that the electronic band tails are incresed in C_{60}@FSM-16 compared with that of C_{60} films. This increase may be explained by the interaction of C_{60} molecules in the pores with the walls of pores in FSM-16.

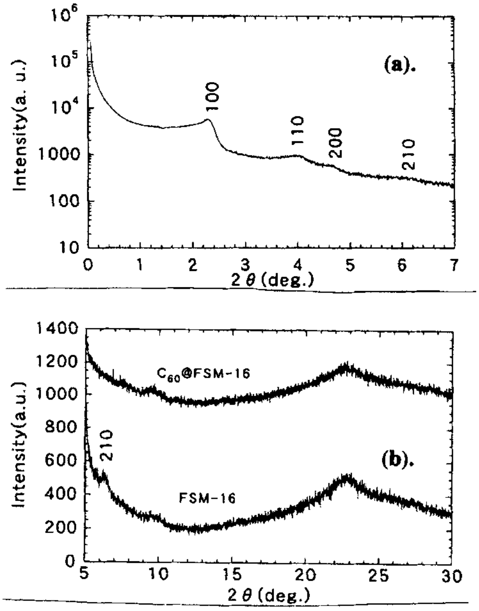

FIGURE 3 (a). Small angle X ray scattering, SAXS, of C_{60}@FSM-16 at RT. (b). X ray diffraction patterns of C_{60}@FSM-16 and FSM-16 at RT.

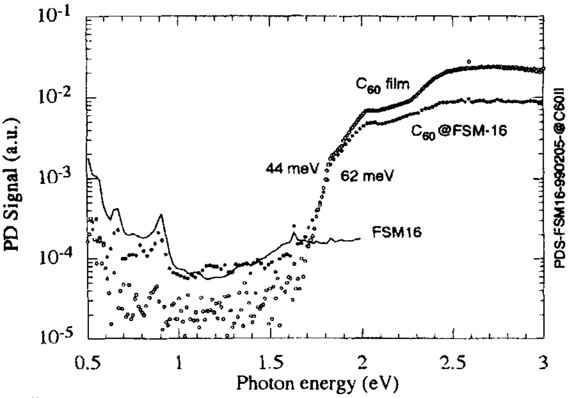

FIGURE 4. PD spectra of photo-themal deflection spectra, PDS, of FSM-16, a C_{60} film and C_{60}@FSM-16.

CONCLUSIONS

Nanostructured composites of C_{60} as a guest material and FSM-16 for the host material were prepared by using a liquid phase method for embedding C_{60} with a help of toluene into the pores of FSM-16. The C_{60} embedded in the pores of FSM-16, which was written in a simplified form as C_{60}@FSM-16 in this paper, was studied by photoluminescence spectra (PL) at 77 K mainly. The shifts of peaks were observed in PL spectrum which was explained by the quantum size confinement of electrons in the C_{60} solid. X ray diffraction , SAXS and ESR spectra were used to characterize C_{60}@FSM-16. PDS was used to obtain the optical absorption characteristics which indicates an interaction of C_{60} molecules with the walls of pores of FSM-16.

Acknowledgments

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