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SYNTHESIS OF CONDUCTING GRAPHITE-LIKE NANOMETER WIRES VIA SOLUBLE PRECURSORS.

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ABSTRACT

Graphite-like conducting materials were encapsulated in the channels of new mesoporous MCM-41 materials with typical channel diameters of 30-40 Å. Acrylonitrile was introduced into the hosts via vapor transport, then polymerized with external radical initiators, $K_2S_2O_8$ and $HNaSO_3$. The polymers in the host cavities were further pyrolyzed at different temperatures under vacuum or nitrogen atmosphere. The properties of the polymer systems were studied while encapsulated or after dissolution of the host. The crystallinity of the hosts is intact after insertion of the polymer (even after pyrolysis at 800°C). The formation of conducting graphite-like materials inside the hosts was demonstrated with Raman and UV spectra. The nitrogen to carbon ratio of the pyrolyzed polymers depends on the pyrolysis temperature and the polymer environment. Most interestingly, the normalized AC absorption of pyrolyzed polyacrylonitrile in MCM-41 (at 800°C) is comparable to graphite.

INTRODUCTION

The possibility of using nanometer conducting wires in future molecular electronic devices[1] has stimulated studies of electronic conductivity in nanometer dimensions. Strategies for stabilizing conjugated polymers in crystalline hosts were developed in our laboratory several years ago[2]. Zeolites with various defined, insulating cavities are the ideal hosts for isolating conducting wires at nanometer dimensions. Following the studies of pseudo one-dimensional conjugated polymers, such as polypyrrole, polythiophene and polyaniline, we have expanded our studies to two-dimensional graphite-like materials, such as pyrolyzed polyacrylonitrile and polyfurfuryl alcohol.

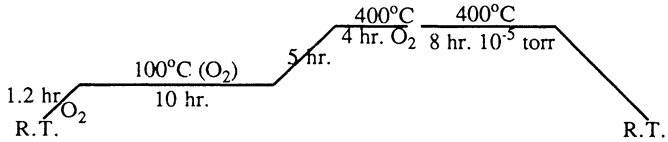
Polyacrylonitrile is a well known carbon fiber precursor[3] polymer which consists of a carbon backbone with a nitrile group on alternating carbon atoms. It can produce insulating, semiconducting or metal-like materials depending on the pyrolysis temperature[4]. Previous studies aimed at the preparation of nanometer conducting wires were limited by the size of zeolite cavities (< 15Å). Recently scientists at Mobil Co. have discovered a new family of aluminosilicates with pore sizes ranging from 20 to 100Å[5]. These materials are excellent hosts for isolating nanometer wires. MCM-41 with regular 34Å hexagonal channels was the major host for this study.

EXPERIMENTAL SECTION

Reagents: $K_2S_2O_8$, $NaHSO_3$ and inhibitor remover were purchased from commercial sources and used without further treatment. Acrylonitrile was passed through the column of inhibitor remover, stored over molecular sieves and distilled before use. MCM-41 was prepared as reported[5] .

Characterization: Fourier transform infrared spectra were measured as KBr pellet or nujol mull using a Mattson Research Series FTIR at 4 cm^{-1} resolution. X-ray powder diffraction data were obtained on a Philips diffractometer using Cu-Kα radiation. Electronic absorption spectra were recorded on a Hitachi U-3501 spectrophotometer with an integrating sphere. The sample was packed in a sample cell which has a $BaSO_4$ background and a quartz window. Nitrogen absorption measurements were conducted on a Coulter Omnisorp 100 physisorption apparatus. The samples were dehydrated under vacuum at 100°C for 4 hours before measurement. The AC (microwave) absorption measurement was performed with an HP8753C network analyzer with maximum frequency of 3G Hz and a home-made cavity. The quality factors (Q) of the empty cavity and cavity with sample in a quartz tube were recorded. From Q, the dielectric constant and conductivity of the sample can be calculated[6].

Dehydration of MCM-41 host: MCM-41 was heated as follows:



Loading acrylonitrile in MCM-41 via vapor transfer:

MCM-41 was contacted with degassed (three cycles of freeze-pump-thaw) acrylonitrile vapor at room temperature for 4 hours. The acrylonitrile loaded MCM-41 (**ANZ**) was evacuated to remove the molecules absorbed on the surface of the host.

Intrazeolite polymerization of acrylonitrile: Under nitrogen, ANZ was mixed with distilled H_2O (typically 1g of ANZ with 20 ml of water). The temperature was raised to 40°C, then $K_2S_2O_8$(aq) and $NaHSO_3$(aq) was added. The mixture was stirred at 40°C under nitrogen for 20 hours, filtered off, washed with water and dried under vacuum. The resulting white solid (**PANZ**) was pyrolyzed under nitrogen at 650°C for 12 hours or at 800°C for 24 hours (**PPANZ**).

RESULTS AND DISCUSSION

Acrylonitrile is a volatile liquid, thus the vapor transfer into the host can be easily achieved at room temperature. The terminal hydroxyl groups of MCM-41 are still observed after inserting acrylonitrile, but at the same time a significant peak at 2240 cm^{-1}, which is the characteristic vibration of CN, appears as shown in Figure 1.

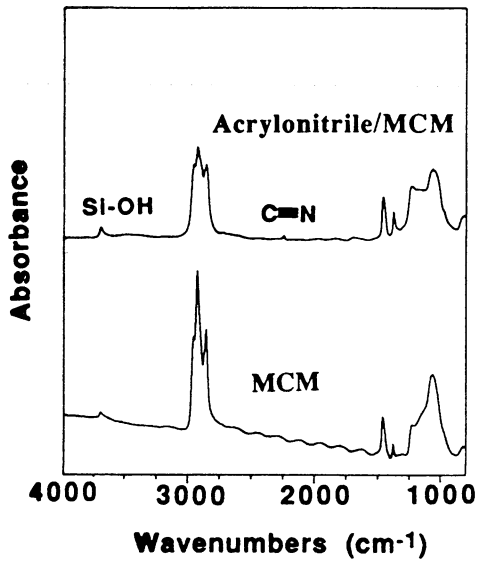


Figure 1. The IR spectra (nujol) of MCM and acrylonitrile/MCM.

An acid-base interaction between guest/host as observed in the aniline/MCM-41 system was not detected here. This probably is due to the weaker basicity of acrylonitrile compared to aniline. X-ray diffraction data show that the crystallinity of the host is intact after insertion of polyacrylonitrile and pyrolysis at 800°C.

The IR spectrum of polyacrylonitrile pyrolyzed at 800°C is featureless, however the Raman spectrum shows two distinct peaks at relative wavenumbers of 1580 cm^{-1} and 1356 cm^{-1} which are similar to the characteristic vibrations of graphite[3]. The other evidence for the formation of graphite-like material comes from the diffuse reflectance UV spectra as shown in Figure 2.

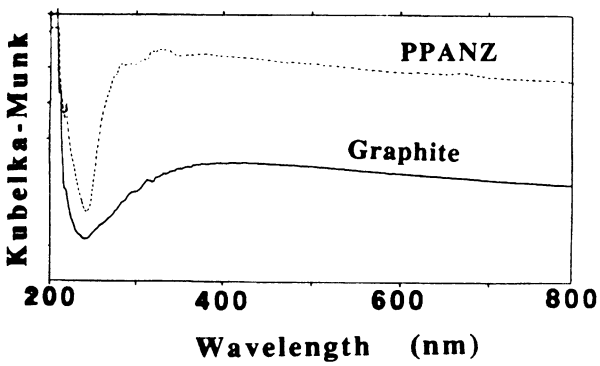


Figure 2. The electronic spectra of PPANZ and graphite.

The spectrum of pyrolyzed polyacrylonitrile/MCM is similar to that of graphite between 200 and 800 nm.

The nitrogen content of pyrolyzed polyacrylonitrile not only depends on the pyrolysis temperature but also on the polymer environment. In general, for the same pyrolyzing temperature polyacrylonitrile in MCM-41 has a higher C/N ratio compared to bulk polymer. The microwave absorption also depends on the C/N ratios and the polymer environments as listed in Table 1.

Table 1: The C/N ratio and AC conductivity at 2.6 GHz of bulk polyacrylonitrile and polyacrylonitrile/MCM pyrolyzed at different temperatures.

Sample	Pyrolysis condition	C/N ratio	AC (S/m) Conductivity
Polymer/MCM	800°C 24hr.	9.86	7.2x10-2
Bulk polymer	800°C 24hr.	8.87	2.4x10-3
Polymer/MCM	650°C 12hr.	6.65	8.2x10-3
Bulk polymer	650°C 12hr.	5.90	2.1x10-3
Graphite		∝	7.2x10-2

The normalized conductivity of PPANZ (with 12% of CH_x) is as high as that of graphite. This is consistent with the formation of graphite-like material in the polyacrylonitrile/MCM system. The formation of graphite-like material inside the channels of the MCM-41 host is supported by the nitrogen absorption measurement as shown in Figure 3.

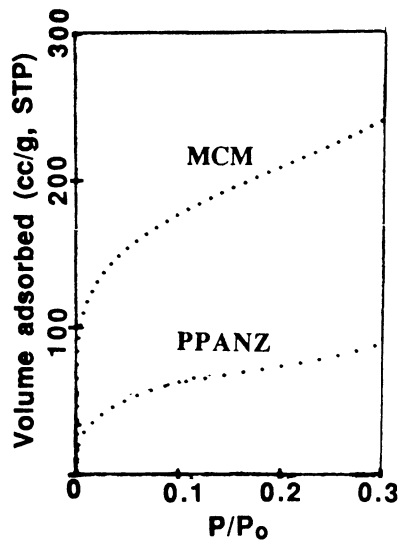


Figure 3. The nitrogen absorption isotherms of PPANZ and MCM.

The pore volume of MCM-41 has significantly decreased after insertion of pyrolyzed polyacrylonitrile. Further characterization of these and related novel nanometer conducting structures will be reported in the near future.

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