

Copper and Nickel Supported FSM-16 Molecular Sieves for Carbon Nanotube Production

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Abstract— FSM-16, Cu-FSM-16 and Ni-FSM-16 type folded sheet mesoporous materials has been synthesized by using kanemite and hexadecyltrimethylammonium bromide as a template. 1, 5, 10 wt % Cu and Ni were loaded by simple impregnation method. The X-ray diffraction and N₂ sorption characteristics show that the resultant materials has uniform pore structure with hexagonal well ordered arrangement. BET surface area, pore volume and pore diameters were decreased as the metal loading increased. Carbon nanotubes (CNTs) have been synthesized within the metal trapped channels of the FSM-16 via chemical vapor deposition using acetylene as the hydrocarbon source. The resultant nanotubes were compared under similar reaction conditions and they were characterized by scanning electron microscopy (SEM), Raman Spectroscopy, AFM and TGA.

Keywords: FSM-16, Metal, CVD, Carbon Nanotube

Introduction

Since the discovery of the CNTs, they have received major attention due to their specific mechanical, physical, electrical properties and technological applications. CNTs can be synthesized by various methods such as laser ablation, arc discharge and chemical vapor deposition (CVD) [1]. Among these methods, CVD is a promising and cost effective process for the large scale production [2]. CVD process is based on the catalytic decomposition of hydrocarbon source on the metal loaded substrate. Mesoporous materials, specifically FSM-16, are favorable candidates for the CNTs production with their unique properties such as large surface areas and high thermal stability [3].

Approaching the desired electronic properties of CNTs depends on certain parameters; catalyst, carbon precursor, synthesis temperature, reaction time, and atmosphere. In present study, we focused on the investigation of CNT growth on the FSM-16 and the metal loaded FSM-16 under various synthesis temperatures.

Experimental

FSM-16 synthesized via intercalating the silicate layers of kanemite NaHSi₂O₅·3(H₂O) with very low concentration of surfactant. N-hexadecyltrimethylammonium bromide and sodium silicate solution used as template for the formation of the pores in FSM-16. Mesoporous FSM-16 silica was modified by impregnation with 1, 5 and 10 wt % Cu and Ni.

The resultant materials were characterized by the powder X-ray diffraction for crystal structure analysis, N₂ sorption for pore size and surface area analysis, Si-NMR and FT-IR for molecular structure analysis, and SEM for morphological analysis.

Prior to CVD, Cu and Ni loaded FSM-16 was treated with H₂ to enhance the catalytic activity. The CVD was carried out in a tube furnace at 873-1073K under argon and acetylene for 30 minutes.

The effect of metal treatment and synthesis parameters on the CNT crystalline organization were examined by X-ray diffraction. The formation of CNT formation was revealed via Raman spectroscopy analysis. The thermogravimetry was implemented in order to examine the ignition temperature and kinetics of CNTs. Also, the morphology of CNTs were visually monitored by SEM and AFM.

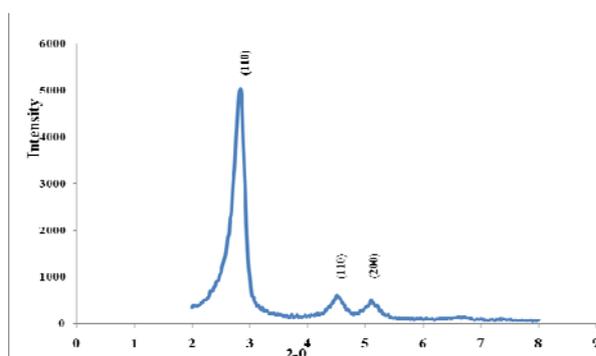


Figure 1. XRD pattern of FSM-16

Results and Discussion

The intensity of the XRD peaks decreased by the metal loading, but the framework of the material was not affected by this treatment. The average BET surface area was found 1100 m²g⁻¹ and BET surface areas and pore diameters decreased as the metal loading increased. In infrared spectra, bands were in agreement with the literature at 2356, 1637, 958, 800, 665 and 620 cm⁻¹ as were observed for FSM-16 [4].

Raman spectra revealed that the reaction temperature effect the quality of and the diameter of the CNTs. Moreover the highest yield was obtained for the Ni loaded FSM-16.

Conclusions

In summary, influence of the catalyst and nanotube synthesis temperature on the characteristics of the CNTs structure and yield in FSM-16, Cu-FSM-16, and Ni-FSM-16 were carried out. The results obtained by TGA, Raman spectroscopy, SEM and X-ray diffractometry suggested that Ni-FSM-16 is a good candidate under these reaction conditions.

References

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