Bull. Mater. Sci., Vol. 31, No. 4, August 2008, pp. 609–611. © Indian Academy of Sciences.

Synthesis of single wall carbon nanotubes from a lamellar type aluminophosphate ($AlPO_4$-L)

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MS received 31 October 2007; revised 29 February 2008

Abstract. **Single wall carbon nanotubes are synthesized from a lamellar type aluminophosphate, $AlPO_4$-L. The lamellar aluminophosphate was synthesized from hexamethyleneimine template. The latter was calcined at argon atmosphere for 12 h at 600°C. The resulting carbonaceous material was treated with 1 N H_2SO_4 to remove the aluminophosphate skeleton. Characterization of the resulting carbon revealed to contain single walled nanotubes. These nanotubes are applicable to store more hydrogen.**

Keywords. **$AlPO_4$-L; single wall carbon nanotubes.**

1. Introduction

Carbon nanotubes (Iijima 1991) are nano-scale structures formed by self assembly. They possess excellent chemical and physical properties (Rodney and Donald 1995; Chen *et al* 1998) that make them promising candidates for various applications ranging from electronics (Chauvet *et al* 1997) to drug delivery (Smart *et al* 2006). Carbon nanotubes synthesis has been carried out by three methods viz. arc discharge (Iijima and Ichiashi 1993), laser ablation (Guo *et al* 2003) and chemical vapour deposition (Kong *et al* 1998).

There are certain steps in nanotube formation that require the catalytic role of the metal (Homma *et al* 2003). Metals like Fe, Co and Ni which form eutectic compound with carbon (Massalki *et al* 1986) are widely used in preference to other metals. The support used for the metals will influence the dispersion and electronic state of the metal and thereby the concentration and the types of nanotubes formed. In this communication, the influence of Al metal in aluminophosphate on carbon nanotubes formation is reported.

2. Experimental

Single wall carbon nanotubes were synthesized from a lamellar type aluminophosphate, $AlPO_4$-L. The lamellar aluminophosphate was synthesized from hexamethyleneimine template, pseudoboehmite, orthophosphoric acid and water mixture. The synthesis was as follows: 3·58 g of pseudoboehmite (74·2%, Vista chemicals, USA) was mixed well with 10 ml of distilled water. 5·75 g of orthophosphoric acid (85%, s.d. fine, India) was added slowly to make a white thick paste. This paste was aged for overnight at room temperature. 5·82 g of hexamethyleneimine (98%, Aldrich, USA) along with 10 ml of distilled water was added slowly to the paste and stirred well. The final active gel was charged into a Teflon lined stainless steel autoclave. Crystallization was carried out for 2 days at 473 K. The product was separated and washed with distilled water and dried at 383 K for 8 h and subjected to physicochemical characterization.

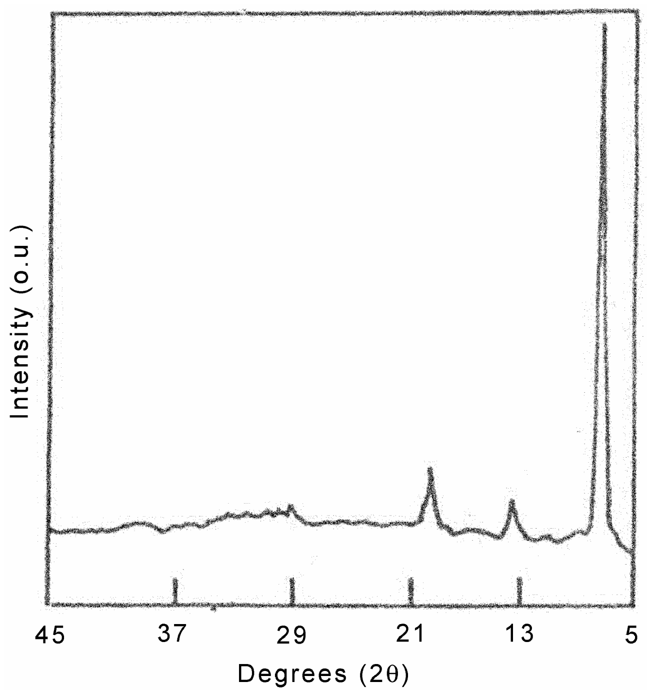

Figure 1. X-ray diffraction pattern of $AlPO_4$-L.

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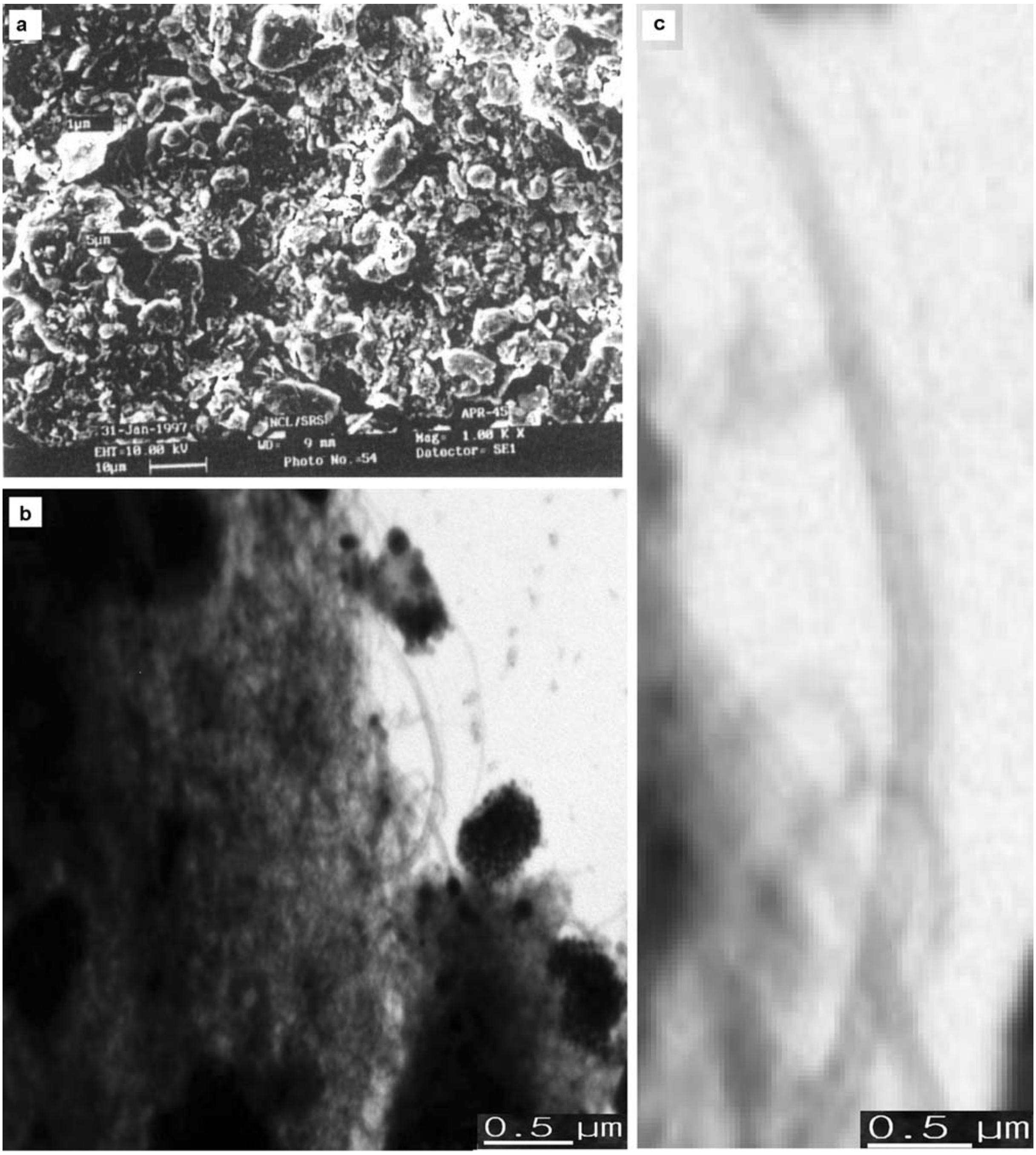

Figure 2. (**a**) Scanning electron micrograph of $AlPO_4$-L, (**b**) transmission electron micrograph and (**c**) magnified transmission electron micrograph of carbon nanotubes.

The synthesized $AlPO_4$-L was calcined in argon atmosphere for 12 h at 600°C. The resulting carbonaceous material was treated with 1N H_2SO_4 to remove the aluminophosphate skeleton.

X-ray diffractograms were recorded on Rigaku Multiplex diffractometer using CuKα radiation and a proportional counter as detector. The scanning electron micrographs were taken using a Topcon, SM-300. The samples were deposited on a sample holder with an adhesive carbon foil and sputtered with gold. From the transmission electron microscopy, the samples were ground and deposited on a circular disc of fine copper mesh covered with collodion. Images were taken using a JEOL JSM-2000 EX electron microscope operated at an acceleration voltage of 200 kV.

3. Results and discussion

Figure 1 shows the as-synthesized $AlPO_4$-L which was found to be of short range ordered lamellar type molecular sieve. It is assumed that the template (hexamethyleneimine) molecules are sandwiched in between the aluminophosphate layers. The scanning electron microscopic analysis shows (figure 2a) that the particles are flag shaped with 5–10 μm particle size.

It is found that the Al present in AlPO framework catalysed the carbon nanotubes formation. Unlike siliceous material the aluminophosphate can be washed out by treating with concentrated sulphuric acid.

The carbon nanotubes formed from the present synthesis are around 50 nm sized and single walled (figure 2b). The thermogravimetric analysis of the carbon nanotubes reveals that the sample loses most of the weight at 600°C at oxygen atmosphere.

This synthesis reveals a simple and environmentally safe and simple process for carbon nanotubes production. Further the fact that aluminophosphate can be used as skeleton in carbon nanotubes formation is established.

4. Conclusions

Single wall carbon nanotubes are synthesized from a lamellar type aluminophosphate, $AlPO_4$-L. The lamellar aluminophosphate was synthesized from hexamethyleneimine template, pseudoboehmite, orthophosphoric acid and water mixture. The latter was calcined in argon atmosphere for 12 h at 600°C. The resulting carbonaceous material was treated with 1N H_2SO_4 to remove the aluminophosphate skeleton. Characterization of the resulting carbon revealed to contain single walled nanotubes.

Acknowledgement

The author thanks the Council of Scientific and Industrial Research, New Delhi, for a fellowship.

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