Journal of Colloid and Interface Science 491 (2017) 375–389

Contents lists available at ScienceDirect

Journal of Colloid and Interface Science

journal homepage: www.elsevier.com/locate/jcis

Size- and shape-controlled synthesis of well-organised carbon nanotubes using nanoporous anodic alumina with different pore diameters

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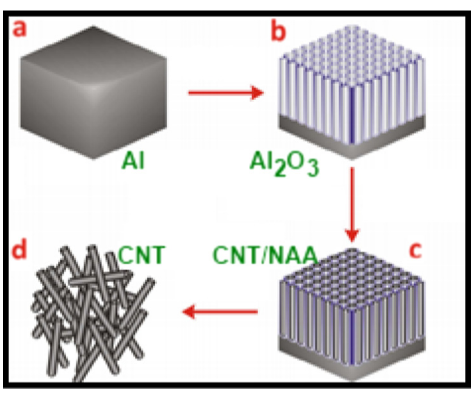
GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 1 April 2016
Revised 11 December 2016
Accepted 13 December 2016
Available online 19 December 2016

Keywords:
Carbon nanotubes
Nanoporous anodic alumina
CVD approach
Composites membrane
Catalyst-free

ABSTRACT

This work aims at introducing the synthesis process of carbon nanotubes (CNTs) inside nanoporous anodic alumina (NAA) templates adopting a catalyst-free chemical vapor deposition (CVD) approach under different conditions. The nanotubular structure of NAA is prepared according to tow-step anodization process. This provides a unique platform to grow CNTs with precisely controlled geometric features. The structural features, crystalline structures and chemical composition of the resulting CNTs-NAA composites were systematically characterized using Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), High Resolution Transmission Electron Microscopy (HRTEM), X-ray Diffraction (XRD), X-ray Photoelectron Spectroscopy (XPS), Energy-dispersive X-ray spectroscopy (EDX), Fourier Transform Infrared Spectroscopy (FTIR) as well as Raman spectroscopy. Preparing the CNTs according to this template technique allows us to obtain nanotubes which are open at one/both end(s) with a uniform diameter (10–200 nm) along the pore length (1–100 μm) without using any metal catalyst.

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http://dx.doi.org/10.1016/j.jcis.2016.12.035
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1. Introduction

The last decades witnessed great advances in the field of nanotechnology. These advances can be exploited to create diverse state-of-the-art materials, nowadays [1–4]. Obviously, carbon-based nanomaterials are among the major driving forces behind the afore mentioned developments [5–9]. Due to their reported unique structural and physical properties (mechanical strength, high electrical and thermal conductivities) [10], carbon nanotubes (CNTs) are in the forefront of nanotechnological research. They have attracted significant interest for industrial applications and received extensive attention in a plethora of areas [11–20]. Before these applications become feasible, however, the manufacturing process of CNTs must be entirely understood in order to produce nanostructures with thoroughly controlled dimensions and properties. The diameter along with the morphology of CNTs manifests a key factor that governs their properties. In fact, extensive research has been directed towards growth of CNTs with controllable dimensions [21]. This gave birth to some approaches which have been used to synthesise CNTs, the most representative of which are arc discharge, laser ablation and catalytic chemical vapor deposition (C-CVD). However, these manufacturing methods present many fundamental disadvantages such as the expensive equipment needed, the synthesis's high temperature, the use of toxic and hazardous materials, impurities/contaminations, etc.). Therefore, the physical and chemical properties of the resulting CNTs depend on both the manufacturing method and the manufacturer, thus preventing the production of standardized CNTs. Generally, CNTs are graphitized but their structure is closed at the tips with a tangled and bundled nature and most importantly there is very little control over their formation [22,23]. Even though previous studies have shown that CNTs synthesis with controlled geometries can be achieved by controlling the catalyst size and the interaction strength between the catalyst and the support, these results are far from satisfactory. On the other hand, nanoporous anodic alumina membranes (NAAMs) are considered excellent inorganic templates to synthesise CNTs membranes featuring vertically aligned nanotubes because their unique set of properties (e.g. hexagonally arranged cylindrical pores, tunable nanometric scale, chemical, mechanical and thermal stability, cost-competitive and scalable fabrication process, etc.) [24]. In addition, liberated CNTs with well-defined geometric characteristics and narrow size distribution can be obtained simply by removing the porous alumina matrix by a conventional wet chemical etching. Herein, we put forward an innovative process to make CNTs membranes with totally controlled properties (e.g. geometry, shape, chemical composition, surface chemistry, etc.). This fabrication allows to feature hexagonally arranged nanotubes with a vertical alignment and well-defined geometry. The afore motioned process is based on a catalyst-free CVD approach, in which NAAMs act as templates. This makes it possible to synthesise CNTs-NAAMs and liberated CNTs for chemical separations. The simplicity, versatility, scalability and cost-competitive fabrication process, allow this approach to be envisaged for producing CNTs featuring

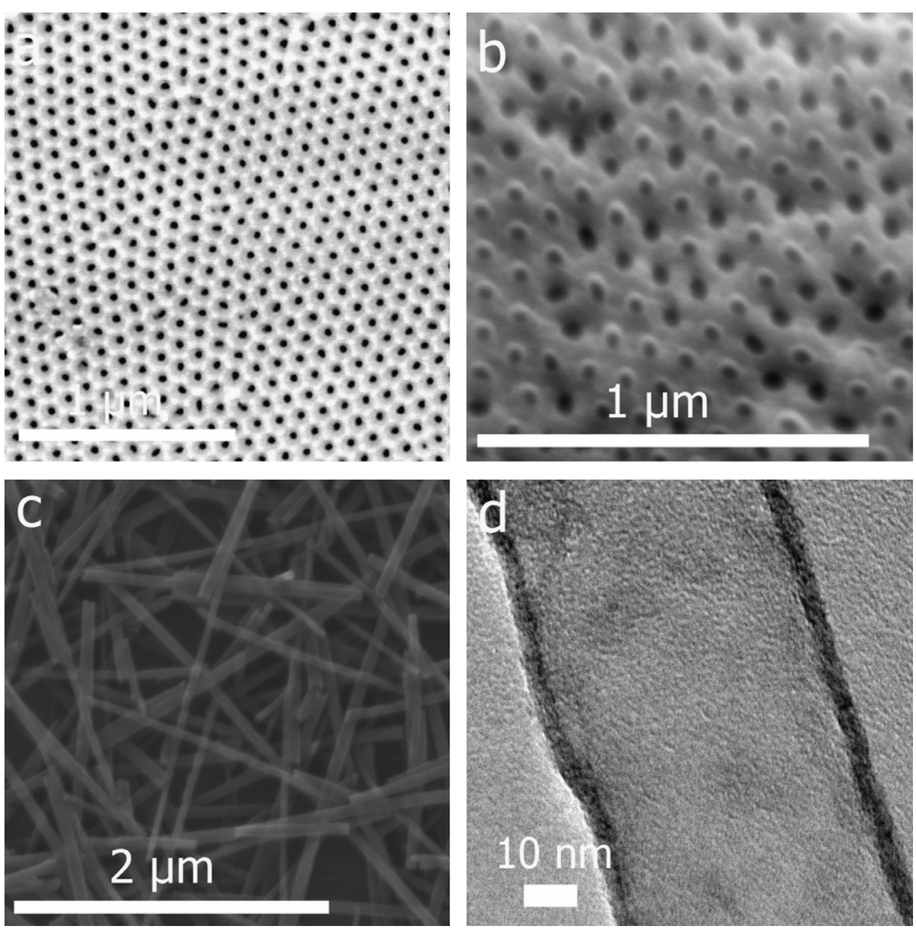

Fig. 1. NAA prepared by electrochemical anodization of Al foils in oxalic electrolyte under *soft/mild anodization* conditions and CNTs/NAA composites prepared by catalyst-free carbon precursor (toluene/ethanol). (a) SEM image of the surface of NAA pores before CNTs deposition. (b) CNTs structure inside NAA pores. (c) Liberated CNTs after removal of NAA template. (d) HRTEM image of liberated CNTs grown inside NAA pores.

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