Advances in Materials and Processing Technologies XVI

Edited by
Zone-Ching Lin, You-Min Huang and Liang-Kuang Chen

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Preface

The present volume contains selected papers from the 16th International Conference on Advanced Materials and Processing Technologies (AMPT 2013) held in Taipei, Taiwan. This conference was co-organized by the SME (Society of Manufacturing Engineers) Taipei Chapter, and the National Taiwan University of Science and Technology (NTUST).

AMPT 2013 is the 16th event in the series, and furthermore, is the 20th anniversary since its inauguration. With two decades of glorious and well-recognized experiences, AMPT 2013 is a major global event in the manufacturing research community and industry. This event has been a good opportunity for expert engineers and researchers to share their new ideas and progresses in manufacturing engineering. This conference covers a wide range of fields including manufacturing processes, materials, automation and control, mechanical design, manufacturing management, and bio-medical manufacturing. This year, the theme of the conference is set as "Advanced Materials Processing and Manufacturing Technologies through Multi-disciplinary Integration." This volume presents a selection of papers submitted to AMPT2013 from universities and industries all over the world. All of the papers were rigorously reviewed by at least two expert referees. Selection of papers for this volume is primarily based on the paper quality and relevancy to the theme. The volume aims to present to the readers the necessity of integration in the field of materials and processing technologies including the forming, machining, automation, manufacturing systems, metrology, precision engineering, computer-aided engineering, and micro/nano technology. It can be regarded as a highlight of recent multi-disciplinary integrations in the materials and processing technologies.

The organizing committee is grateful to all of the contributors who made this volume possible. As the guest editors of the volume, we wish to acknowledge all of those who have reviewed and revised the papers for this volume. Thanks are also due to Miss Wei-Jen Wang at the National Taiwan University of Science and Technology for her clerical and administrative work; and Trans Tech Publications for producing the volume.

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Table of Contents

II. Advanced Machining

III. Geometry Analysis and Forming

IV. Forming Processes

V. Forming with Temperature Influence

VI. Surface and Precision Engineering

VII. Computer Aided Engineering

VIII. Automation and Manufacturing Systems

IX. Micro/Nano Technology

I. Material Science and Processing

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A STRUCTURE AND MORPHOLOGY OF NANOCOMPOSITES COMPOSED OF CARBON NANOTUBES WITH A VARYING FRACTION OF PLATINUM NANOPARTICLES

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Abstract. A structure of nanocomposite materials consisting of carbon nanotubes with a varying fraction of platinum nanoparticles (5, 10 and 20 wt %) is compared in the paper. High-quality CNTs obtained in the CVD process, 100-200 μm long with a standard deviation of below 20% and with a diameter of 10-20 nm, with a standard deviation of below 30%, were used in the research. Raw CNTs did not contain metallic impurities or amorphous carbon deposits. An indirect method of bonding the earlier produced platinum nanoparticles to the surface of functionalised carbon nanotubes was employed to deposit platinum nanoparticles onto the surface of carbon nanotubes. A full array of changes in the loading of carbon nanotubes' surface with platinum nanoparticles was achieved as a result of the experiments performed, starting with homogenous deposition to the clearly developed large agglomerations of platinum nanoparticles. The studies carried out using scanning electron microscopy, transmission electron microscopy, scanning transmission electron microscopy and X-ray structural analysis have confirmed differences in the morphology, homogeneity and density of coating the carbon nanotubes' surface with variedly concentrated platinum nanoparticles. Differences were also revealed in the structure of the newly formed nanocomposites. A nanocomposite with a 5% fraction of platinum nanoparticles demonstrates the best structure-related properties for the materials obtained.

Introduction

Numerous research institutions around the world have been interested in the recent years in interdisciplinary knowledge about nanostructures and their manufacturing technologies and this is accompanied by rapid development in research in this field. Carbon Nanotubes (CNT), being objects built of rolled graphene planes with the diameter of a fraction of nanometre to several dozen nanometres with their length reaching up to several micrometers, have enjoyed strong interest of scientists [1-4]. Due to their unique electrical, mechanical, chemical, magnetic and optical properties, are currently a subject of extensive research from laboratories around the globe [2, 5]. A prerequisite for the practical exploitation of carbon nanotubes' potential application opportunities on a wide scale is an ability to build larger structures and combine them with other materials in a planned and controlled manner. Carbon nanotubes deposition methods using various types of nanoparticles, in particular SiO_2, TiO_2, Ti, Pd, Ag, Pt, Au, Cu, CdS, CdSe, CdTe, are described in the literature [5-10]. The results presented in scientific publications confirm that CNT-NPs (Carbon NanoTubes-NanoParticles) nanocomposites can be fabricated this way, however, the efficiency of the methods described is not always satisfactory on a practical basis. Further research is still needed to optimise the solutions proposed. It is in particular clearly necessary to develop more effective, repeatable technologies, better suited to specific applications and permitting to deposit nanocrystals possessing complex morphologies and dispersion.

The carbon nanotubes-nanoparticles-type nanocomposites represent a valuable material due to a combination of unique physical and chemical properties of their components [11]. The both components feature a large specific surface area and high electrical conductivity. Carbon nanotubes

with the density of approx. 1.33-1.40 g/cm^3 are light, flexible and resistant to bending, stretching and torsion. The bonds between the atoms of carbon in carbon nanotubes are stronger than in diamond, besides, their area is chemically and thermally stable. It has been confirmed that the conductivity of carbon nanotubes changes as a result of interaction with the molecules of multiple chemical substances, both fluids and gases [12, 13], and the effect is even stronger after depositing the nanoparticles of precious metals onto their surface (e.g.: Au, Ag, Pt, Pd, Rh) [14-16]. A major issue relating to the use of CNT-NPs nanocomposites is to control the efficiency of uniformity of nanoparticles deposition onto nanotubes' surface. In order to resolve this issue, it is of essence to explain a formation mechanism of the nanostructures developed and determine the type and quantity of defects occurring in the deposition process. Significant progress can be achieved in such areas as optoelectronics, medicine, energy conversion and storage, with practical applications of such materials in those areas being very wide, by popularising effective procedures of deposition of metal nanoparticles onto carbon nanotubes' surface [17-20].

A structure and morphology of the fabricated nanocomposite materials composed of carbon nanotubes and platinum nanoparticles, with a varying weight fraction (5, 10 and 20 wt %), is compared in this paper. An indirect method of bonding the earlier produced platinum nanoparticles to the surface of functionalised carbon nanotubes was employed to deposit platinum nanoparticles onto the surface of carbon nanotubes. A varying volume fraction of platinum is affecting the structure and morphology of the nanocomposites manufactured. The stage of carbon nanotubes functionalisation plays an essential role in the nanocomposite manufacturing process as such process is conditioning the number of function groups produced (-COOH, -COH, -CO) [21, 22], corresponding to the number of free spaces to which platinum nanoparticles can be connected. A yet unconfirmed hypothesis argues that an unsatisfactory number of function groups developed on the surface of nanotubes may be one of the reasons for agglomeration of platinum nanoparticles. The investigations of the structure and morphology of the materials obtained have been carried out with Electron Transmission Microscopy (TEM), Scanning Transmission Electron Microscopy (STEM) and Scanning Electron Microscopy (SEM). The phase and chemical composition of the nanocomposites manufactured was determined in an X-ray qualitative and quantitative phase analysis.

Research materials and methodology

High quality carbon nanotubes with the length of 100-200 μm and diameter of 10-20 nm obtained in Chemical Vapour Deposition (CVD) were used for the investigations (Fig. 1). The CNTs produced did not contain metallic impurities or amorphous carbon deposits. A commercially available 8% chloroplatinic acid H_2PtCl_6 was used for synthesising platinum nanoparticles.

a) b)

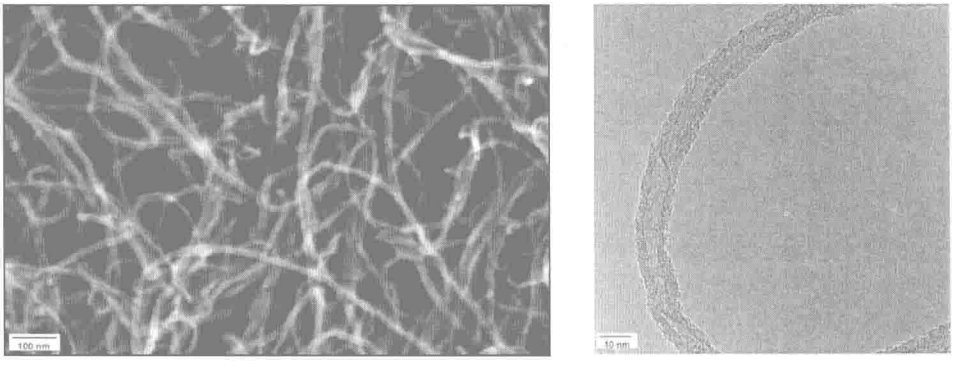

Fig. 1 Unmodified carbon nanotubes: a) SEM image, b) TEM image

Carbon nanotubes possess hydrophobic properties standing for low wettability by fluids, which directly means that their surface can be coated with nanoparticles of metals to a limited extent. To improve efficiency of the process of depositing platinum nanoparticles onto the surface of carbon nanotubes, their functionalisation is necessary. Carbon nanotubes are immersed in a mixture of concentrated HNO_3-H_2SO_4 acids at a rate of 1:3. To dispergate carbon nanotubes in a mixture of acids, a beaker was placed for 30 minutes in an ultrasound scrubber, and then a solution with nanotubes was left covered for 24 hrs. The same nanotubes treatment procedure with, respectively, submerging, ultrasound dispergation and leaving under cover, has been made using a 30% H_2O_2 solution. Carbon nanotubes were permeated following the functionalisation process, washed 5 times in deionised water and dried for 12h at 120°C.

Pt was precipitated as a result of the reduction reaction of a chloroplatinic acid mixture H_2PtCl_6 with ethylene glycol. The functionalised carbon nanotubes, with 15 ml ethylene glycol added, were dispergated in an ultrasound washer in a process lasting 30 minutes. 5 ml of acetone was added to the suspension obtained during constant mixing with a magnetic stirrer. Depending on the planned loading of nanotubes with platinum nanoparticles (5, 10 and 20 wt %) a suitably measured amount of chloroplatinic acid H_2PtCl_6 was added to the suspension using a pipette. All this was heated under a reflux condenser for 8 h at 140°C. The nanotubes produced were then filtered and washed 5 times in deionised water. The nanotubes were dried for 12 hours at 120°C following decoration.

The nanostructures obtained were observed using electron microscopes: a scanning and transmission microscope. SEM images were made using a scanning electron microscope SEM Supra 35 by Carl Zeiss equipped with the X radiation spectrometers: an energy dispersion EDS and wavelength WDS spectrometer and a system for analysing diffraction of back scattered electrons EBSD by EDAX. High resolution and the precision imaging of the preparations viewed was achieved by applying high-performance In-lens SE detector working with low beam voltage and with a very small distance of the preparation tested to the electron gun (working distance). The specimens viewed of the carbon nanotubes decorated with Pt nanoparticles were imaged without depositing conductive layers. TEM and STEM images were performed using a transmission electron microscope STEM TITAN 80-300 by FEI fitted with an electron gun with FEG field emission, a condenser spherical aberration corrector, STEM scanning system, light and dark field detectors, HAADF (High Angle Annular Dark Field), Electron Energy-Loss Spectroscopy EELS, Energy-filtering transmission electron microscope EFTEM, EDS spectrometer as well as equipment and software permitting to implement the electron tomography technique. The exact imaging of the materials examined was possible by applying an HAADF detector (Z contrast). Platinum nanoparticles, due to the different numbers of carbon and platinum atoms: C (Z=6), Pt (Z=78), were seen as lightly illuminating precipitates on the surface of carbon nanotubes. The materials for transmission electron microscopy investigations were prepared by dispergating the nanocomposites obtained in ethanol using an ultrasound washer, and then by depositing them using a pipette with beads onto a TEM mesh. A copper grids coated with a carbon film was used. The material deposited as beads was dried with free air at room temperature. A phase and chemical composition analysis of the examined materials was carried out by means of an X-ray diffraction diagram X´Pert Pro by Panalytical (Cu K_α radiation, $\lambda = 1.54050$ Å) fitted with a semiconductor (band) X'Celerator detector.

Experiment results

Fig. 2 presents the results of observations of carbon nanotubes with a weight volume 5 % of platinum nanoparticles made with a scanning electron microscope SEM. Platinum nanoparticles arranged uniformly on the surface of carbon nanotubes are seen on the photographs as light smudges visible on the twisted nanotubes.

The images of carbon nanotubes decorated with platinum nanoparticles with a varying weight fraction of nanoparticles, i.e.: 5, 10 and 20 %, representing the results of own observations made with a transmission electron microscope, are presented in respectively Fig. 3, Fig.4 and Fig.5. A full array of changes in the loading of carbon nanotubes' surface with platinum nanoparticles was