# Optimization of the Ferromagnetic Nanoparticles Fabrication for Medical Applications

**DOI**
https://doi.org/10.18690/um.feri.4.2025.42

**ISBN**
978-961-286-986-1

KATARZYNA WOJTERA, KRZYSZTOF SMOLKA,
LUKASZ SZYMANSKI

Lodz University of Technology, Institute of Mechatronics and Information System, Lodz, Poland
katarzyna.wojtera@p.lodz.pl, krzysztof.smolka@p.lodz.pl, lukasz.szymanski@p.lodz.pl

Carbon nanotubes possess remarkable properties, rendering them applicable across various fields, including medicine, particularly in hyperthermia treatments when filled with ferromagnetic material. This study focuses on refining the synthesis process of iron-filled multi-walled carbon nanotubes (Fe-MWCNTs) to produce high-purity nanocontainers with elevated iron content. Fe-MWCNTs were synthesized via catalytic chemical vapor deposition (CCVD), with temperature identified as a crucial determinant of synthesis efficiency. The paper also outlines the characterization of the resultant material. To gain insights into the underlying phenomena, computer simulations were conducted using COMSOL software.

**Keywords:**
multi-walled carbon nanotubes filled with iron (Fe-MWCNTs), catalytic chemical vapor deposition (CCVD), COMSOL Multiphysics simulations, ferromagnetic materials, hyperthermia treatments

University of Maribor Press

## I Introduction

Since their discovery by Iijima in 1991 [1], carbon nanotubes have captured the attention of researchers globally due to their remarkable properties, including electrical conductivity [2], mechanical strength [3], high temperature stability, and chemical resistance [4]. These properties enable diverse industrial applications, such as electronics, textiles, supercapacitors, field emitters [1, 5], and fillers in conductive polymer composites [6]. Moreover, carbon nanotubes hold potential in biomedicine, serving as drug carriers or for hyperthermia-induced cancer cell destruction. However, realizing these applications requires efficient synthesis processes to tailor nanotube properties.

Presently, popular synthesis methods include Arc Discharge (AD) synthesis [8], Laser Ablation (LA) [9], and Chemical Vapor Deposition (CVD) [10, 11]. In the described experiments, catalytic chemical vapor deposition (CCVD) with continuous catalyst delivery via solution was employed.

The experiments aimed to optimize the evaporation temperature and dosing rate of ferrocene solution, as well as carrier gas flow rate, and determine the deposition zone temperature conducive to maximal carbon nanotube growth (referred to as the highest "forest" of nanotubes) with encapsulated iron. Encapsulation is crucial for using synthesized Fe-MWCNTs in hyperthermia, ensuring precise localization and heating within tumor tissue [12, 13]. Functionalizing Fe-MWCNT surfaces is also essential, involving iron nanoparticle removal to enhance heating efficiency [14, 15]. Maintaining low amorphous carbon impurities is imperative.

Initially, ferromagnetic-filled CNT synthesis was conducted electrothermally to establish synthesis parameters. General principles of the CCVD process were simulated, creating a heat distribution model using COMSOL Multiphysics software. This computational tool, employing the finite element method, offers high precision in analyzing various phenomena, ranging from optics to heat transfer [16].

## II Materials and methods

### A. Synthesis of Fe-MWCNTs

Carbon nanotubes were produced using catalytic chemical vapor deposition (CCVD) methodology [17, 18]. The synthesis setup utilized a horizontal furnace with three distinct temperature zones for precise thermal management. Zone I served as the catalyst solution evaporation area at 573K, Zone II maintained a temperature of 573K, while Zone III, the deposition zone, operated within the temperature range of 1020K to 1120K. Argon was employed as the carrier gas throughout the process. Ferrocene (Fe(C5H5)2), dissolved in xylene, functioned as the catalyst, also contributing as an additional carbon source for nanotube growth. This method was chosen due to its flexibility in adjusting parameters such as catalyst dosage, temperature in both evaporation and deposition zones, carrier gas flow rate, and synthesis duration. External injection of the mixture occurred at varying inlet velocities ranging from 10 ml/h to 18 ml/h to analyze material characteristics concerning infusion speed.

### B. Modelling using Comsol Multiphysics

The next stage of the work was to simulate the general principles of the CCVD process and create a heat distribution model, which was prepared using COMSOL software. The simulation provided valuable information regarding the choice of parameters for the synthesis of Fe-MWCNTs.

The Navier-Stockes equation was used to describe the gas flow in the reactor chamber (1), (2) and (3). The flow of carrier gas (Ar2) in the quartz reactor was assumed to be laminar, so the Navier-Stockes equation for an incompressible and viscous fluid was used [19].

## III Results

Fig. 1 shows the simulation of heat distribution during the CCVD carbon nanotubes synthesis process. One important part of the process optimization was the proper placement of the silicon wafer, which is the substrate for nanotube growth. The wafer should be placed so as to achieve the highest temperature stability. As mentioned earlier, the synthesis of carbon nanotubes takes place in a furnace divided into three temperature zones. In the first and second the temperature is 570 K, in the third 1070 K. The third zone in the graph below (Figure 1) starts at 60cm of the furnace. The obtained plot shows the results of simulating the temperature distribution in the furnace with the placement of silicon wafers in different places of the third temperature zone.

The results of this simulation show that the temperature on the wafer is most stable when the wafer is placed evenly in the middle of the third temperature zone or in the second half of this zone. Furthermore, when examining the process carried out in the laboratory, it was observed that less material was formed at the end of the third temperature zone than at the beginning. Hence the conclusion that the silicon substrate should be placed evenly in the middle of the third zone to ensure both better temperature stability and process efficiency.

Another aspect investigated during the computer simulations in the COMSOL environment was the determination of the heating time of the quartz reactor before the start of the synthesis process.

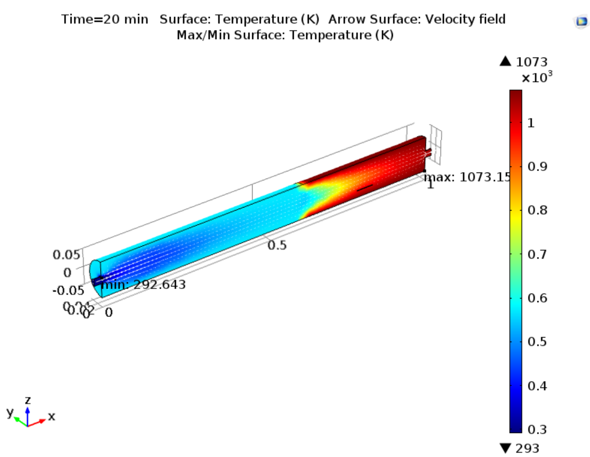


**Figure 1: Heat distribution model during the synthesis process [own elaboration].**

The next step of the study was to optimise the synthesis of Fe-CNTs by CCVD with respect to iron content. Since one of the objectives of the work was to obtain the highest possible iron content in the nanotubes, the results of different syntheses, in which the concentration of ferrocene-xylene mixture was changed, were compared. The iron content was examined by thermogravimetric analysis (TGA).

In the trials carried out for the synthesis of carbon nanotubes, a number of dependances have been demonstrated that define the temperature limits at which successful nanoscale processes occur. In order to present these dependency, only SEM images allowing observation of the process products have been selected and presented.

## IV Conclusions

In the optimization of carbon nanotube synthesis via catalytic chemical vapor deposition (CCVD), various studies were conducted involving COMSOL simulations and experimental trials to identify optimal parameters. It was found that placing the silicon substrate in the middle of the third temperature zone yields the best results. Pre-synthesis preparation time of at least 10 minutes is necessary for the wafer temperature to reach the deposition zone temperature, aligning with existing literature. The ideal substrate thickness ranges from 0.5mm to 2.5mm, ensuring proper temperature distribution. The carrier gas flow rate should be kept below 1250 SCCM for efficient substrate transfer. Optimizing the CCVD process led to the discovery that a substrate solution concentration of 200mg/ml yields the highest quality nanotubes. Further analysis using electron microscopy and thermogravimetry revealed temperature-dependent growth rates and filling degree conditions for Fe-MWCNTs. Ongoing research involves XRD analysis to identify iron allotropic varieties and determine deposition zone temperatures for strictly ferromagnetic materials. Additionally, efforts are directed towards maximizing alpha-Fe nanotube filling in MWCNTs.

**References**

[1] Iijima S.: Helical microtubules of graphitic carbon, Nature 354, 1991, 56–58, [doi: 10.1038/354056a0].
[2] Filleter T., Bernal R., Li S., Espinosa H.D.: Ultrahigh Strength and Stiffness in Cross-Linked Hierarchical Carbon Nanotube Bundles, Advanced Materials 23 (25), 2011, 2855–2860 [doi: 10.1002/adma.201100547]
[3] de Heer W.A., Chatelain A., Ugarte D.: A carbon nanotube field-emission electron source, Science 270, 1995, 1179–1180.

[4] Thostenson E., Chunyu Li, Tsu-Wei Chou: Nanocomposites in context, Composites Science and Technology, 65(3–4), 2005, 491–516, [doi: 10.1016/j.compscitech.2004.11.003].

[5] Hong S., Myung S.: Nanotube Electronics: A flexible approach to mobility. Nature Nanotechnology 2 (4), 2007, 207–208 [doi: 10.1038/nnano.2007.89].

[6] Hafner J.H., Bronikowski M.J., Azamian B.R., Nikolaev P., Rinzler A.G., Colbert D.T., Smith K.A., Smalley R.E.: Catalytic growth of single-wall carbon nanotubes from metal particles, Chem. Phys. Lett. 296, 1998, 195–202

[7] Jeszka J.K., Pietrzak L.: Polylactide/Multiwalled Carbon Nanotube Composites – Synthesis and Electrical Properties. Polimery, 55(7-8), 2010, 524–528.

[8] L. Szymanski, Z. Kolacinski, G. Raniszewski, and E. Gryska, "CNTs synthesis on steel strip a in microwave plasma reactor for medical application," IEEE-NANO 2015 - 15th Int. Conf. Nanotechnol., pp. 1062–1065, 2015.

[9] J. Chrzanowska et al., "Synthesis of carbon nanotubes by the laser ablation method: Effect of laser wavelength," Phys. Status Solidi Basic Res., vol. 252, no. 8, pp. 1860–1867, 2015.

[10] K. Wojtera, M. Walczak, L. Pietrzak, J. Fraczyk, L. Szymanski, and A. Sobczyk-Guzenda, "Synthesis of functionalized carbon nanotubes for fluorescent biosensors," Nanotechnology Reviews, vol. 9, no. 1, pp. 1237–1244, Jan. 2020, doi: 10.1515/ntrev-2020-0096.

[11] G. Raniszewski, Z. Kolacinski, and L. Szymanski, "Ferromagnetic nanoparticles synthesis in local thermodynamic equilibrium conditions," IEEE-NANO 2015 - 15th Int. Conf. Nanotechnol., pp. 323–326, 2015.

[12] G. Raniszewski, A. Miaskowski, and S. Wiak, "The Application of Carbon Nanotubes in Magnetic Fluid Hyperthermia," J. Nanomater., vol. 2015, 2015.

[13] A. A. Bhirde et al., "Targeted Killing of Cancer Cells in Vivo and in Vitro with EGF-Directed Carbon Nanotube-Based Drug Delivery," ACS Nano, vol. 3, no. 2, pp. 307–316, Feb. 2009, doi: 10.1021/nn800551s.

[14] D. Chen et al., "Photoacoustic Imaging Guided Near-Infrared Photothermal Therapy Using Highly Water-Dispersible Sin-gle-Walled Carbon Nanohorns as Theranostic Agents," Advanced Functional Materials, vol. 24, no. 42, pp. 6621–6628, Nov. 2014, doi: 10.1002/adfm.201401560.

[15] A. L. Antaris et al., "Ultra-low doses of chirality sorted (6,5) carbon nanotubes for simultaneous tumor imaging and photo-thermal therapy," ACS Nano, vol. 7, no. 4, pp. 3644–3652, Apr. 2013, doi: 10.1021/nn4006472.

[16] "COMSOL Multiphysics 5.5 Reference Manual," COMSOL AB, 2019.

[17] E. Kowalska et al., "Morphology and electronic properties of carbon nanotubes grown with Fe catalyst," Journal of Materials Research, vol. 18, no. 10, pp. 2451–2458, 2003, doi: 10.1557/JMR.2003.0341.

[18] L. Szymanski, Z. Kolacinski, S. Wiak, G. Raniszewski, and L. Pietrzak, "Synthesis of Carbon Nanotubes in Thermal Plasma Reactor at Atmospheric Pressure," Nanomaterials, vol. 7, no. 2, p. 45, Feb. 2017, doi: 10.3390/nano7020045.

[19] N. S. R. et al. . Nishant Sharma. R et al., "The Simulation of Three-Dimensional Heat Transfer Through an Insulated Pipe by Variation of Solver Parameters," Int. J. Automob. Eng. Res. Dev., 2019.