Detailed Numerical Simulation of Single-Walled Carbon Nanotube Synthesis in a Radio-Frequency Induction Thermal Plasma System

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Abstract. 2D axisymmetric numerical calculations are conducted to model the thermo-fluid fields and chemical reactions leading to the formation of SWCNTs in an RF plasma system. A modified version of the SWCNT “reduced” chemical model is used to estimate the formation of SWCNT in an RF plasma system for the first time. The “reduced” model incorporates 14 species and 36 chemical reactions to predict the formation of metal and carbon clusters and SWCNTs. By combing the chemistry model into the RF plasma CFD code, the formation and development of carbon and metal catalyst clusters and their reactions which produce SWCNTs are shown. The chemistry model is shown to under-predict the yield rate of SWCNT. In order to better predict the yield rate, a sensitivity analysis is performed to modify the dominant reaction rates. The modified model predicts the yield of SWCNTs correctly within the range reported experimentally. However, more studies should be conducted to validate the accuracy of the model for different operating conditions.

1. Introduction

Carbon nanotubes have been at the forefront of nanotechnology since their discovery in 1991. These nanomaterials are expected to make a major impact particularly in the composite material fields such as aerospace, automobile, and personal protective industries, where strength and weight of materials are important factors. A new method for the production of SWCNTs at large-scale that is receiving much attention is the induction thermal plasma process, developed initially for fullerene synthesis [1-3].

Figure 1 shows a schematic of an induction thermal plasma system for the continuous production of SWCNTs. The synthesis system mainly consists of a plasma torch, a reaction chamber and a filtration system. Three different gas streams of powder, central, and sheath gases are introduced into the
induction plasma torch. The central gas has a swirl component to stabilize the plasma. The mixture of carbon black and metallic catalyst powder (nickel) is carried into the plasma region by the powder gas. The reaction chamber includes a graphite liner, heating elements, and a thermal insulator, which are employed for an active control of maximum achievable temperature, background temperature, and cooling rate. The final products are then collected in a filtration chamber, which consists of three porous metallic filters.

A detailed kinetic mechanism for SWCNT production in numerical simulations of an RF plasma system is required for understanding the process and identifying the parameters that mainly influence nanotube production. In the present study, a “reduced” form of a fullerene/soot/metal cluster model developed for the arc discharge and laser ablation processes [4] has been employed and modified to describe the SWCNT formation kinetics in this RF plasma process, and applied to a CFD code which models the 2-D thermo-fluid field of an RF plasma system with chemical reactions [5, 6].

Figure 1. Schematic layout of an induction thermal plasma processing system for the continuous production of high quality single-walled carbon nanotubes (SWNT) at large-scale

2. Model Description

In the present work, the models of the RF plasma generation, and the interaction between the plasma and injected feedstock material. i.e., carbon and nickel particles are considered in a 2D axisymmetric numerical framework. The kinetic mechanism of the formation of SWCNTs and several intermediate species was approximated by using the carbon/metal cluster model (see section 2.6) developed by Scott [4]. In order to integrate the kinetic model into the reaction chamber’s thermo-flow field model, a separate in-house gas phase parallel computational fluid dynamics code [7-8] was used. The parallel computing code was primarily developed for computing the detailed chemical kinetic mechanisms of soot formation in flames [7-8], and can perform numerous fast computations on multiple computer processing units (CPUs).

2.1. Radio Frequency Plasma Model

A 2-dimensional axi-symmetric model for simulations of plasma generation by an RF induction plasma torch has been developed based on the procedure described by Mostaghimi et al. [9] and Proulx et al. [10]. The following assumptions were considered in modeling the induction plasma: (i) two-dimensional, axisymmetric and steady-state flow; (ii) quasi-neutral and optically thin plasma in a local thermodynamic equilibrium (LTE) state; (iii) negligible displacement current and viscous dissipation; (iv) planar coils; (v) turbulent flow; (vi) consideration of the momentum and energy source terms due to the particle loading. Based on these assumptions, the plasma equations are described by steady-state conservation equations for the transport of mass, momentum, energy, concentration of species and the k-ε turbulence model coupled with electromagnetic field equations.
The axisymmetric cylindrical coordinate system \((r, z)\) was adopted in this model. The governing equations are similar to those in previous works \([11, 12]\).

### 2.2. Plasma-Injected Particle Interaction Model

In the induction thermal plasma process, the carbon precursor material is directly evaporated in the plasma plume, and therefore, the yield rate of SWCNT in the reactor can be affected by the evaporation efficiency of the injected particles. The Lagrangian approach was used to describe the injected particles inside the plasma \([13, 14]\). The following simplifications were assumed in this work: (i) spherical particles were assumed; (ii) a uniform temperature distribution inside a particle was assumed; (iii) particle sizes were assumed to follow the Rosin-Rammler distribution function; (iv) the rarefaction effect (Knudsen effect) was not taken into account \([15]\). The detailed formulations can be found in previous works \([11, 12]\).

### 2.3. Computational domain and operating condition

The computational domain in the present work, figure 2, consists of two zones; an RF plasma torch, and a reaction chamber. A commercial RF plasma torch considered in this work (PS-50, TEKNA Plasma Systems, Inc.), is driven by a plate power of 45 kW with the coupling efficiency of about 70% which is operated at an oscillator frequency of 3 MHz.

The detailed dimensions of the reactor system can be found in the previous studies \([11,16]\). In this work, the length of the graphite liner and insulator (wool) in the reaction and quenching chamber was extended from 50 cm to 1 m in accordance with the new experimental setup being used at NRC. A typical operating condition used in this numerical study is summarized in table 1.

![Figure 2. The computational domain including RF plasma torch and the reaction chamber](image)

| Net plasma power (kW) | Pressure (kPa) | Carrier gas (slpm) | Central gas (slpm) | Sheath gas (slpm) | Feed rate (g/min) |
|-----------------------|----------------|--------------------|--------------------|------------------|-------------------|
| 31.5                  | 66             | 3 (Ar)             | 23 (Ar)            | 135 (He)         | 1.5 (CB/Ni: 91/9 wt.%) |

A mixture of carbon black (CB), and nickel was considered as a feedstock material, with a ratio of CB/Ni = 91.0 wt%/9.0 wt%, mean diameter of 2 µm, and minimum and maximum diameters of 1 and 3 µm, respectively.

### 2.4. Chemistry model

The fullerene/soot/metal clusters model was developed by Scott \([4]\) in predicting the production of nanotubes in arc vaporization and laser ablation systems \([18-20]\). Scott’s proposed model is based on the fullerene model of Krestinin et al. \([20]\) with a modification on the soot formation component. Their
model is based on the formation of clusters of carbon that are built up from atoms to carbon chains, rings and polycycles, fullerene shells, C_{60} and C_{70}, and then soot. They give detailed gas-phase reactions for producing carbon clusters and fullerenes as well as for fragmentation and evaporation of clusters. The final production of carbon cluster growth is soot that grows via heterogeneous reactions according to its surface area. Scott [4] added combined carbon-nickel clusters to the fullerene model of Krestinin et al. [20].

In Scott’s carbon-nickel clusters model, the soot-nickel clusters (ZNiC) are nuclei for SWCNT formation and growth. In the model, the formation of SWCNTs was assumed to occur from Ni_{1024} clusters (NiC) reacting with soot (Z) and fullerene (C_{60F}) as feedstocks. The size of the nickel cluster was approximately chosen based on the diameter of SWCNT. After the formation, the clusters are assumed to combine with large carbon clusters, namely soot or fullerene, to form SWCNTs. The model cannot represent the real number of nanotubes of different lengths, but the total amount of carbon in SWCNTs (the mass fraction) can be meaningful in the post-processing analysis.

The “reduced” model includes 14 species and 35 reactions. The reduced kinetics model has been previously used in CFD simulations of a multi-cycle laser ablation method in the production of nanotubes and the model was shown to be computationally inexpensive and robust compared to the full CNT model [4]. The reactions in this work are from “reduced” chemistry model by Scott [4], reproduced in table 2. The model, as described by Scott [4], is phenomenological and assumes constant reactions rates of formation of nickel-carbon clusters, approximating their kinetic rates, since there was no available data on temperature-dependency. All SWCNTs in the model are assumed to consist of 999 carbon atoms. This assumption means the model cannot represent the real number of nanotubes of different lengths, but the total amount of carbon in SWCNTs (the mass fraction) can be meaningful in the post-processing analysis.

| Reactions | A [mole-cm-sec-K] | b | E [K] |
|-----------|------------------|---|-------|
| C + C = C₂ | 2.00E+14 | 0 | 0 |
| C + C₂ = C₃ | 2.00E+14 | 0 | 0 |
| C₂ + C₂ = C₃ + C | 2.00E+15 | 9040 |
| C₁ + C₁ = 0.15CC | 2.00E+14 | 0 | 0 |
| CC+CC=1.025CC | 2.30E+14 | 0 | 0 |
| CC + C₂ = 1.05CC | 2.30E+14 | 0 | 0 |
| CC + C₃ = 1.075CC | 2.30E+14 | 0 | 0 |
| CC = 0.95CC + C₂ | 3.20E+13 | 0 | 61900 |
| CC + C₃ = 0.70C₆₀F + C | 2.00E+13 | 0 | 0 |
| CC + C₂ = 0.70C₆₀F | 4.00E+09 | 0 | -30196 |
| CC + C = 0.6833333C₆₀F | 2.00E+13 | 0 | 0 |
| C₆₀F = 1.45CC + C₂ | 8.00E+13 | 0 | 61900 |
| C₆₀F + C₁ = 0.7875Z | 2.00E+13 | 0 | 10065 |
| CC + CC = Z | 4.00E+13 | 0 | 0 |
| Z + C₁ = 1.0375Z | 4.00E+12 | 0 | 0 |
| Z + C₂ = 1.0250Z | 4.00E+12 | 0 | 0 |
| Z + C = 1.0125Z | 4.00E+12 | 0 | 0 |
| Z = DZ | 1.00E+02 | 0 | 0 |
| Ni + Ni = Ni₂ | 3.70E+12 | 0.5 | 0 |
| Ni₂ + Ni = Ni₃ | 4.10E+12 | 0.5 | 0 |
| Ni₃ + Ni = 0.039062NiC | 4.40E+12 | 0.5 | 0 |
| Ni₃ + Ni = 0.0039062NiC | 4.51E+12 | 0.5 | 0 |
| Ni₃ + Ni = 0.0048828NiC | 4.37E+12 | 0.5 | 0 |
| Ni₃ + Ni = 0.0058593NiC | 4.45E+12 | 0.5 | 0 |
| Ni + NiC = 1.0009766NiC | 8.04E+13 | 0.5 | 0 |
26  Ni$_2^+$ + NiC  1.0019531NiC  5.96E+13  0.5  0
27  Ni$_3^+$ + NiC  1.0029297NiC  5.03E+13  0.5  0
28  Ni$_3^+$ + Ni  4.30E+17  0.5  24506.6
29  Ni$_3^+$ + Ni$_1^+$ + Ni  3.37E+17  0.5  28145.3
30  Z + NiC  0.9990234ZNiC + Ni+ 0.0009766Z  6.61E+18  0.5  44780
32  Z + NiC  ZNiC  1.00E+16  0.5  0
33  DZNiC  ZNiC  1.00E+04  0  0
34  C$_{60F}$ + ZNiC  0.08008008008CNT + ZNiC  1.00E+12  0.5  0
35  C$_{60F}$ + ZNiC  0.06006006006CNT + ZNiC  1.00E+12  0.5  0
36  C$_{60F}$ + ZNiC  0.002002002002CNT + ZNiC  1.00E+12  0.5  0

Table 2-1. Nomenclature and definitions of species in reaction mechanisms [4]

| Species       | Definition                                         |
|---------------|----------------------------------------------------|
| NiC           | Nickel cluster (Ni$_{1024}$)                       |
| Z             | soot ($C_{80}$)                                    |
| DZ            | dead soot ($C_{80}$)                               |
| C$_{60F}$     | fullerene                                         |
| ZNiC          | soot-nickel cluster ($C_{80Ni_{1024}}$)            |
| DZNiC         | dead soot-nickel cluster ($C_{80Ni_{1024}}$)       |
| CNT           | carbon nanotube ($C_{999}$)                        |

2.5. Computational Scheme and Boundary Conditions

In the computational domain shown in figure 3, the no-slip boundary conditions are applied to the torch and reaction chamber walls. The temperature on the outside of the reaction chamber is set to room temperature, i.e., 300K. At the torch and reactor axes, the radial velocity and the radial gradients of all other variables were set zero, while axial gradients of the variables were assumed zero at the reactor exit. Lastly, the boundary conditions needed for the electromagnetic field calculation inside the torch were based on Xue’s approach [6].

First, the 2D axisymmetric thermo-fluid fields inside the plasma torch and reaction chamber were modeled by the commercial code FLUENT [21]. The thermo-flow field equations, solved inside the reactor, were continuity, conservation of momentum, conservation of energy, and conservation of species. The k-ε turbulent model was implemented for the flow inside the reactor. FLUENT solves the discretized forms of the governing equations by a SIMPLE-like algorithm proposed by Patankar [22]. The vector potential equation for the electromagnetic field was solved by using the User Defined Functions (UDFs) approach. The Discrete Phase Module (DPM) of FLUENT was used to consider the interaction between plasma and the injected particles. In total, 70 particles of each nickel and carbon black were released from the probe located at the center of the torch and the injection velocity of the particles was assumed to be equal to the axial velocity of the carrier gas. For the given operating conditions in table 1, all the injected particles were completely evaporated before leaving the reactor. In FLUENT the thermodynamic and transport data for the plasma gases (i.e., argon and helium) were obtained from the tabulated data [23], which were calculated with the assumption of LTE. The effects of the metallic and carbon vapors on the plasma properties - except on the radiation and electrical properties - were taken into account using the available data for carbon, and nickel vapors [24]. In the second part of the simulations, only the thermo-flow field results from FLUENT simulations for the reaction chamber is interpolated into the mesh generated by the parallel computing code [7] where the chemical kinetic mechanism, described in section 2.4, for the formation of SWCNT is included. In the parallel computing code, the finite volume method is used to discretize the equations of conservation of species mass and energy. A staggered mesh is used with a semi-implicit scheme to solve the discretized equations [22]. The diffusive terms are discretized using a second-order central difference scheme while the convective terms are discretized using a power law scheme [22]. At each time step, all the gaseous species including those generated from the reactions in the chemical kinetic model
were solved in a coupled way at each control volume to handle the stiffness of the system. In order to speed up convergence, only the species equations were iterated and after every few thousands iterations, the temperature field was updated. In the parallel computing code, the thermal properties of the gaseous species and chemical reaction rates are obtained using CHEMKIN subroutines. Distributed-memory parallelization with strip-domain decomposition is employed. The computational domain is divided uniformly into 64 subdomains with 3 columns of control volumes per CPU as shown in figure 3. The algorithm uses the Message Passing Interface (MPI) library to parallelize the code. The computations are performed on the General Purpose Cluster (GPC) systems of SciNet [26], on eight 8-processor nodes on the infiniband network with 2.53 GHz chip speeds.

Figure 3. The computational domain of the reaction chamber and the mesh in the parallel computing code

The results are divided into two main sections; in the first section the simulations of the thermo-fluid filed inside the RF plasma torch and the reaction-quenching chamber are shown and the results from the implementation of the chemical kinetics model into the CFD code for predicting the formation of carbon-nickel clusters and carbon nanotube (specifically SWCNT) are presented. A sensitivity analysis on the chemistry model was conducted to adjust (within the uncertainty range) the rate reactions of the most dominant reactions in order to compare the SWCNT yield with the experimental data. In the second section (sec 3.3), the CFD simulations were repeated using the modified chemistry model and the results were compared with the experimental data.

3. Results and Discussion

3.1. CFD results with original chemistry model

The distributions of the temperature and species concentration fields are shown in figures 4. The off-axis profiles of the high temperature zones are clearly observed for both cases due to the skin effect. The central region is also cooled down by the presence of injected powders. Once the plasma flow leaves the torch it expands into the reaction chamber and enhances the mixing phenomena inside the reaction chamber, the off-axis temperature peak begins to disappear. The presence of the graphite liners throughout the entire length of the chamber helps to maintain the higher temperature region (>1400 K) inside the reactor longer and therefore lengthen the favorable temperature window for the growth of SWCNTs. Figure 4b shows the distribution of the carbon and nickel mass fractions inside the reactor system. The carbon and nickel vapours produced in the hot regions are transported to the cooler regions via diffusion and convection, and the carbon and nickel atoms start to form various carbon clusters, nickel clusters, nickel-carbon composite clusters, and SWCNTs.
In the next step in the modeling, the calculated thermo-fluid fields inside the reaction chamber are used as initial conditions and interpolated over the grid generated by the parallel computing code (see figure 3). Similarly, the data extracted at the exit of the torch are used as the boundary condition at the reaction chamber’s inlet in the parallel computing code and the chemical kinetic model for the formation of intermediate species and SWCNT, table 2, was added. Then, the thermo-fluid fields with the chemical reaction mechanism were iterated until a converged solution was obtained.

Figure 5 illustrates the mass fraction of carbon nanotubes (specifically SWCNTs) generated inside the reaction chamber. The main concentration is located away from the centreline and close to the reactor’s wall where the temperature gradient is steep. The production of SWCNTs in the RF plasma processing system is estimated about 10~20 wt% of the injected carbon black material in the experiments. According to operating conditions of this work, with a feed rate of 81.9 g/h of carbon black, the SWCNT mass flow rate obtained from the experiments is between 8.6-17.2 g/h. To evaluate the predicted results from the chemistry model, the mass flow rate of SWCNTs at the exit of the reaction chamber was calculated as follows:

$$\dot{n}_{\text{CNT}} = \frac{3600 \times \frac{f}{h}}{\sum_j \left( A_j \times U_j \times \rho_j \times Y_{\text{CNT},j} \right)}$$

where $A_j$ is the cross section area of cell $j$ at the exit of the reactor; $Y_{\text{CNT},j}$ is the mass fraction of carbon nanotubes in the cell $j$ at the exit of the reactor; $U_j$ and $\rho_j$ are the axial velocity and density of the mixture in cell $j$. The yield rate of SWCNT predicted by the model was 0.548 g/h. It is assumed that this under-prediction of yield relates to the chemistry model and the wide uncertainty ranges of reaction rates. In order to better predict the yield of SWCNTs, in the next section a sensitivity analysis
was performed on the chemistry model to identify the dominant reactions and their role in production of carbon nanotubes in the simulations.

![SWCNT mass concentration in the reaction chamber](image)

**Figure 5.** SWCNT mass concentration in the reaction chamber

### 3.2. Sensitivity Analysis of the chemistry model

In sensitivity analysis, the sensitivity of the model’s results to a small perturbation of a single input is studied. Sensitivity analysis and absolute rate of production parameters provide useful tools to assess the importance of different reactions and pathways on the formation of desired final products in the system. A zero-dimensional test calculation and a sensitivity analysis on the original reduced chemistry model of Scott [4] were performed in CHEMKIN-PRO [27] to identify the most important reactions in the chemistry model that influence the yield rate of CNTs. The chemical rate equations given in table 2 were calculated using the Perfectly Stirred Reactor (PSR) model in CHEMKIN-PRO. The mass-weighted average of the temperature and species concentrations and the area-weighted average of the axial velocity profiles at the inlet of the reaction chamber in an RF plasma processing system were calculated and employed as the inlet boundary conditions in the zero-dimensional. A steady state condition was assumed to analyze the reaction rates in different temperature regimes, and the temperature inside the PSR model was reduced from 4000 K to 1200 K. The residence time in each case was set to 30 seconds. Table 3 shows the sensitivity of SWCNT formation to the most three dominant reactions in different temperature regimes. It can be seen that at temperatures higher than 2000 K, the most dominant mechanisms are the formation of Ni$_2$ from nickel atoms and the reverse reaction of evaporation of Ni$_2$ to nickel atoms. However, since the production rate of nanotubes at this temperature range is negligible, the sensitivity to the reaction Ni$_2$ → 2Ni is less important. As the temperature drops under 2000 K, the dominant mechanisms are the formation of CNT from fullerene (C$_{60}$) and nickel-carbon cluster (ZNiC), i.e., C$_{60}$ + ZNiC → 0.06 CNT + ZNiC, and the nickel-carbon clusters inerting, i.e., ZNiC → DZNiC. The latter reaction has an adverse effect on the formation rate of CNTs by deactivating nickel-carbon clusters. Based on the present analysis, one may conclude that the nanotube formation in the chemistry model is mainly dominated by the one-way reaction: C$_{60}$ + ZNiC → 0.06 CNT + ZNiC and the limiting parameter in the formation rate of nanotubes is the concentration of nickel- carbon clusters (ZNiC).

**Table 3. The dominant reactions and the sensitivity analysis results**

| Reactions / T (K) | 1200   | 1250   | 1300   | 1400   | 1500   | 2000   | 2500   | 3000   | 3500   | 4000   |
|-----------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| Ni + Ni         | -1.18  | 2.5    | 8.7    | 0.3    | 98     |
| Ni$_2$ → Ni + Ni| 1.18   | -2.5   | -8.7   | -0.3   | -98    |
| ZNiC            | 0.945  | 0.95   | 0.956  | 0.968  | 0.979  | 0.998  |
| C$_{60}$ + ZNiC | -0.945 | 0.95   | 0.956  | 0.968  | 0.979  | 0       |
The actual reaction rate coefficients for SWCNT formation and for the rate at which carbon and carbon clusters become over coated with carbon and become poisoned are not accurately known. Therefore in the following test cases the reaction rates of those mechanisms believed to be the most important, were adjusted within their range of uncertainty to gain more realistic production of SWCNT. The most noticeable change in SWCNT mass fraction at temperatures lower than 2000 K was by decreasing the Arrhenius coefficient by one order of magnitude from $10^4$ to $10^3$ in the rate reaction of ZNiC inerting. This change resulted in higher concentration of active carbon-nickel clusters (ZNiC) that can participate in the reaction mechanisms for producing SWCNTs.

3.3. CFD results with the modified chemistry model

To assess the effect of the change in the reaction rate of carbon-nickel clusters deactivation on SWCNT yield, the CFD calculations in the reaction chamber were repeated with the modified reaction rate. Figure 6 shows the mass concentration of SWCNTs in the reaction chamber with the modified rate. The maximum concentration of nanotubes was increased compared to the previous simulation as there are more active carbon-nickel clusters present in the system.

Figure 6. SWCNT mass concentration by the modified chemistry model in the reaction chamber

As can be seen from figure 6, SWCNTs start to form in a small area near the reactor’s inlet and close to the graphite liners. The lower temperature region generated in this area, due to the expansion of the flow entering the reaction chamber, promotes the formation of carbon-nickel clusters (ZNiC) as shown in figure 7. The calculated value of the mass flow rate of SWCNTs at the exit of the reactor in the new simulation is about 12 (gr/min) which is in the range of 8.6-17.2 g/h of the experiments.

Figure 7. Carbon-nickel clusters mass concentration in different temperature regions in the reaction chamber

4. Conclusion

In the present study, a chemistry model of SWCNT formation from the intermediate species developed for the arc discharge and laser ablation processes [4, 17, 18] was used for the computation of SWCNTs yield in an RF plasma processing system. The principle contribution of the present work
is in the adaptation of the chemistry model to a CFD code coupled with an electromagnetic model in order to numerically describe the entire process, in macroscopic scales, from the injection of the feedstock material into the plasma, to the final products containing the SWCNTs. A sensitivity analysis was performed which demonstrated that reactions forming ZNiC were the current bottleneck in SWCNT formation. When key reaction rates were adjusted within their known uncertainty, CNT yield could be well predicted for a single isolated case. Currently, the model, as explained in the previous sections, has deficiencies in accuracy and further work will be done to investigate accurate chemical the reaction rates in different operating conditions.

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