Effects of Feed Gas Composition and Catalyst Thickness on Carbon Nanotube and Nanofiber Synthesis by Plasma Enhanced Chemical Vapor Deposition

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Effects of Feed Gas Composition and Catalyst Thickness on Carbon Nanotube and Nanofiber Synthesis by Plasma Enhanced Chemical Vapor Deposition

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Many engineering applications require carbon nanotubes with specific characteristics such as wall structure, chirality and alignment. However, precise control of nanotube properties grown to application specifications remains a significant challenge. Plasma-enhanced chemical vapor deposition (PECVD) offers a variety of advantages in the synthesis of carbon nanotubes in that several important synthesis parameters can be controlled independently. This paper reports an experimental study of the effects of reacting gas composition (percentage methane in hydrogen) and catalyst film thickness on carbon nanotube (CNT) growth and a computational study of gas-phase composition for the inlet conditions of experimentally observed carbon nanotube growth using different chemical reaction mechanisms. The simulations seek to explain the observed effects of reacting gas composition and to identify the precursors for CNT formation. The experimental results indicate that gas-phase composition significantly affects the synthesized material, which is shown to be randomly aligned nanotube and nanofiber mats for relatively methane-rich inlet gas mixtures and non-tubular carbon for methane-lean incoming mixtures. The simulation results suggest that inlet methane-hydrogen mixture coverts to an acetylene-methane-hydrogen mixture with minor amounts of ethylene, hydrogen atom, and methyl radical. Acetylene appears to be the indicator species for solid carbon formation. The simulations also show that inlet methane-hydrogen mixture does not produce enough gas-phase precursors needed to form quality CNTs below 5% CH₄ concentrations in the inlet stream.

Keywords: Carbon Nanotubes, Carbon Nanofibers, Plasma Enhanced Chemical Vapor Deposition, Reaction Mechanisms.

1. INTRODUCTION

Carbon nanotubes (CNTs) possess extraordinary properties that can be applied to a number of fields in engineering and science,¹ and improved understanding of CNT formation would enable many future nanotechnology areas.² Formation of ‘filamentous carbon’ was reported in literature by Baker et al.³ in early 1970s and by Oberlin et al.⁴ in the mid-1970s. At the time, these reports did not stimulate the interest of the research community compared to recent interest generated by the report of CNTs grown by Iijima.⁵ CNTs have attracted much attention because of their unique mechanical, electrical, and thermal properties. Much current research focuses on characterizing these properties more fully, and researchers are now beginning to develop engineering applications in a wide variety of fields, such as field emission displays,⁶–⁸ interconnects for nanoscale circuits,⁹,¹⁰ thermal interfaces,¹¹–¹⁵ and biological sensors.¹⁶,¹⁷ CNT length, diameter, chirality, alignment, and density can strongly influence the efficacy of nanotubes in such devices. However, CNT growth mechanisms are not yet clearly understood, and precise control of their properties grown to prescribed specifications with good repeatability remains a significant challenge.

The first step in achieving control of CNT synthesis involves finding the range of conditions needed for their formation. Most prior studies have reported the conditions under which growth occurred successfully; however, these reports often do not provide insights into the sensitivity of synthesis to relevant operating parameters, such as inlet gas composition and catalyst thickness. The present work reports the results of a systematic study of the ranges of relevant parameters in which nanotubes and nanofibers do and do not grow in the PECVD process.

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Footnotes:

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Plasma-enhanced chemical vapor deposition (PECVD) enables independent control of synthesis variables such as substrate temperature, substrate bias, operating pressure and inlet feed gas composition. The dissociation of hydrocarbons in plasma permits synthesis of CNTs and carbon nanofibers (CNFs) to proceed at temperatures lower than those encountered in other techniques and allows for controllable gas chemistry near the growth substrate. In addition, vertical alignment of CNTs grown by PECVD has been well documented and could aid in development of applications requiring high spatial density of devices, such as ultra dense digital memory.

Until recently, CNTs grown by PECVD had been limited to those with the multi-walled structure, and the abundance of carbon produced by hydrocarbon dissociation in plasma was speculated to inhibit the growth of single-walled CNTs (SWCNTs). Even catalysts known to produce SWCNTs by thermal CVD produced only multi-walled CNTs (MWCNTs) in PECVD synthesis, with Delzeit reporting as small as double walled CNTs amongst larger multi-walled varieties. Recent reports by Kato et al., Li et al., and Maschmann et al., however, have demonstrated successful synthesis of SWCNTs by PECVD from pre-prepared particle catalysts. Significantly, Li et al. reported an unexpectedly high preferential growth (~90%) of semi-conducting SWCNTs with radio frequency PECVD. While the present work employs film-deposited catalysts that produce multi-walled CNTs, the results of the parametric study are likely to be relevant to all forms of CNTs produced by PECVD.

Of the many parameters that can be varied, inlet gas composition and catalyst thickness are the two that are studied here. Feedstock gas compositions range from 3% to 20% methane in hydrogen, and catalyst thicknesses vary from 5 nm to 20 nm. The effects of each parameter are characterized through interpretation of scanning electron micrographs and Raman spectra. The gas-phase environment in the PECVD reactor has been simulated to enhance the fundamental understanding of CNT growth mechanisms and to aid in controlling the synthesis process.

2. EXPERIMENTAL DETAILS

50 nm diameter silicon wafers served as substrates for growth, and wafers were used with the native oxide present. A thin 20 nm film of chromium was first evaporated in a Varian electron beam evaporator, followed by a thin film of nickel catalyst. The chromium layer was deposited under the nickel catalyst layer to promote adhesion of the nickel layer to the silicon wafer and to block silicides from contaminating the nickel catalyst. The objective of using this chromium underlayer was to achieve good anchoring of carbon nanotubes so that the nanotubes could be used for applications requiring such bonding. The thickness of the chromium layer was held constant while three different nickel catalyst thicknesses were studied: 20 nm, 10 nm and 5 nm. The deposition of both metals occurred at 7 × 10⁻⁷ torr, and the deposition rate was held constant at approximately 0.1 nm/sec by controlling the electron beam current and by using an Inficon XMS-3 thickness monitor.

Carbon nanotube synthesis occurred in a SEKI AX5200s microwave plasma-enhanced chemical vapor deposition (PECVD) reactor system. The system allows independent control of substrate temperature, substrate position, gas flow rates, chamber pressure, and microwave power. H₂ (Praxair 4.5 grade) and CH₄ (Praxair ultra high purity) were supplied to the chamber via separate mass flow controllers with full scale capacities of 1000 standard cubic centimeters per minute (scm) and 10 sccm, respectively. N₂ gas (Praxair 4.8 grade) was supplied to the chamber for purposes of venting and purging the reactor through a 10 sccm mass flow controller. Susceptor temperature was controlled using a 5 kW RF induction heating source (Advanced Energy PE-II) with a feedback thermocouple embedded in the center of the susceptor at a depth of 2.54 mm from the susceptor surface. The susceptor surface temperature inside of the chamber was monitored using a dual-wavelength pyrometer (Williamson Model 90) aimed near the center of the test substrate. The reactor plasma was powered by a 1.5 kW, 2.45 GHz microwave generator (ASTeX AX2100). The main control panel monitored the net microwave energy deposition rate by measuring both supplied and reflected microwave power.

The metal-coated silicon substrates were placed on a 50 mm diameter molybdenum puck and inserted into the reactor. The molybdenum puck serves to concentrate the plasma above the sample wafer. For each growth run, the chamber was evacuated to 0.7 torr for 5 minutes to remove contaminant species and was then pressurized with H₂ at 40 standard cubic centimeters per minute (scm) to 10 torr for 5 minutes. The susceptor temperature was set to 700 °C, and the system remained at this temperature for 5 minutes after steady state was achieved. The plasma was ignited at 500 W power for 2 minutes for pretreatment of the catalyzed wafer to promote the aggregation of catalytic metal particles from the film. Lastly, CH₄ was introduced for 10 minutes. Growth runs were conducted with CH₄ flow rates of 10 sccm (20% CH₄), 5 sccm (11.1% CH₄), 2.5 sccm (5.9% CH₄) and 1.25 sccm (3% CH₄). After each growth run, the system was returned to its initial state, and the chamber was left to cool to room temperature. All other parameters except catalyst film thickness and CH₄ ratio were held constant for all experiments.

3. SIMULATION METHODOLOGY

The zero-dimensional SAMP (Simple Analysis of Materials Processing Reactors) code was used for the present simulations of methane-hydrogen mixtures. The
code solves volume-averaged species mass, gas energy and plasma energy conservation equations. The SAMPR reaction mechanism used in the present study involves reactions among neutral species through recombination, abstraction, neutral-ion charge exchange reactions, ion-ion recombination reactions, electron-ionic recombinations, and electron impact dissociation, ionization and excitation reactions.\textsuperscript{27} The governing gas energy equation is

\[
\rho V \frac{dT_e}{dt} = \left( \sum_i \frac{m_i}{n_i} \frac{\partial}{\partial t} \rho V n_i \right) - \dot{m}_e T_e + 3 \sum_i \frac{\rho V n_i}{m_i} \sum_j R_{ij} k(T_e - T_i) + \frac{3}{2} \frac{\rho V n_i}{m_i} k(T_e - T_i) + V \sum_i h_i M_i \sum_j R_{ij} - h_i A(T_e - T_i) \quad (1)
\]

The terms on the right side of Eq. (1) represent the inlet mixture’s sensible heat, outlet mixture’s sensible heat, gained from elastic collisions between electrons and neutral species, heat gained from charge-exchange collisions of electrons and neutral species with ions, heat due to chemical energy and heat loss to ambient, respectively.\textsuperscript{27,28} This energy equation essentially provides a balance equation for the equilibrium gas temperature \(T_e\). The ionic temperature \(T_{i,\text{ion}}\) is assumed to be constant (1160 K) and variation of this parameter had a negligible effect on the results.\textsuperscript{27,28}

The coupling between the plasma energy source and the plasma species is described in the plasma energy equation, which takes the form

\[
\frac{3}{2} \frac{\rho V n_i}{m_i} \frac{\partial}{\partial t} \rho V n_i = P_{in} - \dot{Q}_{in} n_i - \dot{Q}_{out} n_i - \sum_j R_{ij} k(T_e - T_i) - 3 \sum_i \frac{\rho V n_i}{m_i} \sum_j R_{ij} k(T_e - T_i) - A_{\text{el}} k(T_e - T_i) + A_{\text{i}} \sum_j \sum_{\text{nucl.}} m_{\text{nucl}} n_{\text{nucl}} k(T_e - T_i) - \ln \left( \frac{(A_{\text{el}}/A) \sum_{\text{nucl.}} m_{\text{nucl}} n_{\text{nucl}} k(T_e - T_i)}{(n_{\text{el}}/4) V_{\text{el}}} \right) \quad (2)
\]

The terms on the right side of Eq. (2) represent input power from an external source (one of the main parameters in the present study), inflow of electron energy, outflow of plasma power density to initiate reactions, and the role of plasma (ionic species and electrons) to initiate reactions in the inlet mixture. Because the reduced GRI 2.11, GRI 3.0, HACA 1997, and HACA 2000 reaction mechanisms include only neutral species and the SAMPR reaction mechanism includes electron, neutral, and ionic species, the present simulations with reduced GRI 2.11, GRI 3.0, HACA 1997, and HACA 2000, relevant ionic species, electron and elementary ion-containing reactions were included from the SAMPR reaction mechanism to initiate reactions in the inlet methane-hydrogen gas mixture.

Simulations assume uniform plasma power deposition from an external source in the plasma of a PECVD reactor, as shown in Figure 1, to represent the energy deposition by microwave power in the PECVD reactor. The plasma volume used in the present simulations was estimated from visual observations under experimental growth conditions in a PECVD reactor. The corresponding calculated volumetric plasma power densities decrease with increased input plasma power and increase with increased reactor pressure, consistent with observations in the literature.\textsuperscript{36,37} At 10 torr reactor pressure, the volumetric plasma power density decreases from 5.7 W cm\(^{-3}\) to 4.8 W cm\(^{-3}\) with

\[
\rho V \frac{\partial}{\partial t} \rho V n_i = \dot{m}_e n_i - \dot{m}_i + \sum_j R_{ij} \quad (3)
\]
an increase in input plasma power from 200 to 500 W. In the present work, the internal heat transfer coefficient, $h_{int}$, of the PECVD reactor is used as a free parameter that is adjusted to match the simulated plasma gas temperature with experimentally measured H-atom temperatures in a similar reactor. For the gas composition study, the plasma gas temperature at 10 torr and 500 W input plasma power is found to be approximately 1700 K. The inlet gas compositions were the same as described in the previous section. In addition to the experimental inlet gas composition, a simulation representative of diamond growth conditions was also performed at 0.82 sccm inlet methane flow rate, which corresponds to 2% of the total flow rate.

4. RESULTS AND DISCUSSION

Each experiment yielded a black film over regions of the substrate where the catalyst metal was originally deposited. The chromium/nickel bi-layer used in this study proved to be successful, not only in the growth of carbon film, but in the growth of film that was well adhered on to the substrate surface. A stream of dry nitrogen was blown over each sample to assess qualitatively the degree of the adhesion, and no flaking of the carbon film was observed. In contrast, preliminary synthesis experiments with only a nickel catalyst layer yielded carbon film that did not adhere well to the substrate surface in the presence of a nitrogen gas stream. Following this initial examination for adhesion, the samples were observed in a scanning electron microscope (FESEM, Hitachi S-4700).

Low-magnification FESEM images were recorded to assess overall characteristics, and high-magnification images were used to measure the approximate diameter distributions of the nanotubes and nanofibers. Diameters were measured using conventional digital imaging software by calculating the number of pixels across a nanotube or nanofiber and converting the result into an approximate dimension via the scale bar associated with each image.

The quality of growth deteriorated as the CH$_4$ flow rate was decreased. For carbon-rich mixtures, definitive, long and straight carbon nanotubes were observed, and for carbon-lean mixtures, non-tubular carbonaceous materials were predominantly observed. Figure 2 shows high-magnification SEM images of the resulting structures for four different gas compositions, ranging from methane concentrations of 20% to 3%. All images correspond to nickel catalyst thicknesses of 20 nm. Importantly, the images illustrate a strong effect of gas-phase composition, with richer mixtures producing predominantly tubular and/or fibrous structures and leaner mixtures producing predominantly non-fibrous media. This result suggests that gas-phase composition profoundly influences the type and purity of carbon produced. A similar trend was observed for other catalyst thicknesses.

Figure 3 shows the resulting diameter measurements from all cases. The results indicate that, for 20 nm nickel catalyst layers, the average diameters fall in the range of 35 nm to 55 nm for all gas-phase conditions. Considering that the diameters of single-walled carbon nanotubes are typically small (1 to 10 nm), any carbon nanotubes observed in the present study were multi-walled. These measurements agree with other published results, which also used 20 nm nickel catalyst layers with a chromium underlayer.

Figure 3 also illustrates an inverse relationship between diameter and CH$_4$ flow rate over the ranges considered. The diameter generally increased as the CH$_4$ concentration decreased from 20% to 5.9%. For all thicknesses of nickel catalyst, nanotube growth was almost non-existent in the leanest mixture, and mostly non-tubular carbonaceous materials were present.

The diameters exhibited a general decreasing trend as the thickness of the nickel catalyst thickness decreased (see Fig. 4). This behavior agrees with prior results and tends to confirm the dependence of nanotube diameter on the size of the catalyst metal. A positive correlation between film thickness and diameter is most prevalent at 20% CH$_4$, for which diameter decreased consistently as the nickel film thickness decreased. For the cases of 5.9% and 11.1% CH$_4$, a slight increase in diameter was observed at the minimum nickel film thickness of 5 nm, compared to the 10 nm case.

To assess the composition of the non-tubular content, Raman spectrographs were recorded for the samples. Figure 5 shows the resulting spectrum for a film that contained primarily non-tubular content (20 nm nickel, 3% methane). The figure shows two distinct peaks above 1000 cm$^{-1}$. The first peak, at 1330 cm$^{-1}$, is indicative of diamond (sp$^3$) or disordered bonding (D band). The
second, at 1580 cm\(^{-1}\), indicates graphitic (sp\(^2\)) bonding (G band). The presence of diamond bonding is not surprising, given that the same PECVD process is used extensively to grow diamond films under similar carbon-lean gas conditions.\(^{31}\) The graphitic peak is likely from both non-tubular and tubular content in the sample. We also note that Raman spectra from richer mixtures also showed strong D bands, and this result suggests that even in the samples with relatively dense nanotube and nanofiber mats, much non-graphitic content exists.

The present simulations focused on the effects of inlet methane concentration on the presence of various reactants in the volume-averaged mixture. Figure 6 shows the mole fractions of major species formed in the plasma at different inlet CH\(_4\) concentrations for different reaction mechanisms. The major species formed in the plasma are H, H\(_2\), CH\(_2\), CH\(_3\), C\(_2\)H\(_2\), and C\(_2\)H\(_4\). The sum of major species concentrations is at least 99.89% for HACA 1997, HACA 2000, and SAMPR reaction mechanisms and 99.99% for GRI 2.11 and GRI 3.0 reaction mechanisms for all inlet

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**Fig. 2.** SEM images of carbon nanotubes grown on 20 nm nickel catalyst films under different CH\(_4\) flow rates: 20% (top left), 11.1% (top right), 5.9% (lower left), 3% (lower right).

**Fig. 3.** Diameter dependence on CH\(_4\) flow rate for various nickel catalyst film thicknesses.

**Fig. 4.** Diameter dependence on nickel catalyst film thickness for various CH\(_4\) flow rates.
concentrations considered. This result indicates that all major species formed in the plasma are very similar for different reaction mechanisms. Figure 6 also shows that HACA 1997 and HACA 2000 reaction mechanisms estimate almost identical species concentrations formed in the plasma. The same is true for GRI 2.11 and GRI 3.0 reaction mechanisms. This result suggests that effects of changes in the rate parameters between reaction mechanisms (GRI 2.11 vs. GRI 3.0 and HACA 1997 vs. 2000) is insignificant under the present simulation conditions. Hereafter, ‘HACA’ will refer to both HACA 1997 and HACA 2000 and ‘GRI’ will refer to both GRI 2.11 and GRI 3.0 reaction mechanisms.

The results indicate that inlet CH₄ converts to mainly C₂H₂ and H₂, as C₂H₂ concentrations are the highest among carbon-containing species in the plasma, and that H₂ mole fractions are higher than the corresponding mole fractions in the inlet stream for all reaction mechanisms studied here. The CH₄ mole fractions are the lowest for the SAMPR reaction mechanism as compared to the HACA and GRI mechanisms, as inlet methane-hydrogen mixture dissociation is the highest for the SAMPR mechanism. H, CH₃ and C₂H₄ are also formed in the plasma, but their mole fractions are at least an order of magnitude lower than that of C₂H₂. Formation of H and C₂H₄ are favored by the SAMPR mechanism over HACA and GRI mechanisms, while CH₃ formation is favored by the HACA and GRI mechanisms over SAMPR at all levels of inlet CH₄ concentration. The mole fractions of CH₃, C₂H₂, and
C\textsubscript{2}H\textsubscript{2} increase with increasing inlet CH\textsubscript{4} for all reaction mechanisms. Even though all reaction mechanisms contain neutral and ionic species, under the present simulation conditions, only neutral species are formed to a significant degree in the plasma. This result indicates that gas phase chemistry in the plasma is dominated by the neutral species.

The above experimental results show (see Fig. 2) that CNTs of reasonable quality are formed at 5.9% CH\textsubscript{4} concentration in the inlet stream, and the quality of the growth product generally increases with increasing inlet CH\textsubscript{4}. At 3% inlet CH\textsubscript{4}, formation of non-tubular carbon was observed. The present simulations show that the gas mixture reactivity in the plasma increases significantly above 5% inlet CH\textsubscript{4} concentration and increases further with an increase in the inlet CH\textsubscript{4} concentration. The CH\textsubscript{4} concentrations in Figure 6 suggest that its dissociation is insignificant below 5% CH\textsubscript{4} concentrations in the inlet stream; hence the inlet methane-hydrogen mixture is not reactive enough to produce gas phase precursors needed to form quality CNTs below 5% CH\textsubscript{4} concentrations.

At 2% inlet CH\textsubscript{4} concentration, diamond is typically formed. Petherbridge et al.\textsuperscript{42} conducted simulations of a similar reactor at 2000 K gas temperature, and observed that if the [H]/[C\textsubscript{2}H\textsubscript{2}] ratio is greater than 0.2 and if the CH\textsubscript{3} mole fraction is greater than 10\textsuperscript{-7}, then diamond growth occurs. Conversely, if the [H]/[C\textsubscript{2}H\textsubscript{2}] ratio is less than 0.2 but [CH\textsubscript{4}] is greater than 10\textsuperscript{-7}, then non-diamond growth appears. Under the present simulation conditions, CH\textsubscript{3} mole fractions are greater than 10\textsuperscript{-7} in all cases. Figure 7 shows the change in [H]/[C\textsubscript{2}H\textsubscript{2}] ratio with an increase in the CH\textsubscript{4} mole fraction in the inlet stream. Significantly, simulations with all mechanisms show that the [H]/[C\textsubscript{2}H\textsubscript{2}] ratio is higher than 0.2 for 2% inlet CH\textsubscript{4} concentration (suggestive of diamond growth according to the Petherbridge et al. criterion), and is much less than 0.2 for 5.9% and higher inlet CH\textsubscript{4} mole fractions (indicative of non-diamond carbon). Again for all mechanisms, the [H]/[C\textsubscript{2}H\textsubscript{2}] ratio is nearly 0.2 at the 3% inlet CH\textsubscript{4} mole fraction in the inlet stream. We note that the results from present study are consistent with the Petherbridge et al. criteria in that almost no tubular carbon content was observed at 3% inlet CH\textsubscript{4} mole fraction, while carbon nanotubes and/or nanofibers were observed for all higher inlet methane concentrations. The correlations between the present experimental study and the numerical simulations, interpreted through the Petherbridge et al. criteria, appear to be sound and provide a rational basis for predicting the occurrence of carbon nanotube synthesis under the set of conditions considered herein.

5. CONCLUSIONS

The reported results indicate that inlet gas-phase composition can profoundly affect the synthesis of carbon products produced by PECVD. The effects of variations in catalyst film thickness and methane flow rate on the resulting nanotube diameters were studied from FESEM images. The nanotube diameters showed a general decreasing trend in size as the thickness decreased, while the diameters showed a general increasing trend as the methane flow rate was decreased. Reasonable quality CNTs form at 5.9% CH\textsubscript{4} concentration in the inlet stream, and the quality of CNTs generally increases with increasing inlet CH\textsubscript{4}. Simulations indicate that the inlet methane-hydrogen gas mixture converts to an acetylene-hydrogen gas mixture, and C\textsubscript{2}H\textsubscript{2}, H, H\textsubscript{2}, and CH\textsubscript{3} are the main precursors formed in the plasma for both diamond and CNT growth conditions. The trends predicted by the present simulations for CNT growth conditions are similar to those estimated by experimental data for diamond growth conditions. The simulations confirm that the atomic hydrogen to acetylene ratio ([H]/[C\textsubscript{2}H\textsubscript{2}]) demarcates a change in growth products, as suggested by Petherbridge et al.\textsuperscript{42} This study concludes that changes in catalyst thickness and gas-phase composition exhibited strong effects on the diameters of the nanotubes and nanofibers and the quality of growth. Future planned studies using in situ
Effects of Feed Gas Composition and Catalyst Thickness on CNT and Nanofiber Synthesis by PECVD

NOMENCLATURE

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<th>Symbol</th>
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<td>A</td>
<td>reactor surface area, m²</td>
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<td>Aeff</td>
<td>effective area to account for the deviation of sheath edge ion density from that at the center, m²</td>
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<td>c_e</td>
<td>mixture specific heat at constant pressure, J kg⁻¹ K⁻¹</td>
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<td>E_e</td>
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<td>H_j</td>
<td>threshold energy for electron impact reaction j, J</td>
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<tr>
<td>y_{in}</td>
<td>mass fraction of species i at inlet</td>
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Greek symbols

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<tr>
<td>\lambda</td>
<td>mean free path of gas molecules, m</td>
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<tr>
<td>\rho</td>
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<td>\nu_{e,el}</td>
<td>charge exchange collision frequency, s⁻¹</td>
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<td>\nu_{e,el}</td>
<td>elastic collision frequency between electrons and species i, s⁻¹</td>
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<td>electron thermal velocity, m s⁻¹</td>
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Subscripts

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References and Notes

11. J. Li, Q. Ye, A. Cunill, H. T. Nguyen, R. Steven, J. Han, and M. Meyyappan, Appl. Phys. Lett. 82, 2491 (2003).

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