Cold Wall Reactor for Continuous Production of Carbon Nanomaterials

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Abstract

Current lab-scale carbon nanotube (CNT) production methods have processing times in excess of one hour, but the actual growth of CNTs only takes a few minutes. The additional processing time is due in part to the hot-wall batch design of existing reactors, where the entire body of the reactor has to be heated and cooled for each production cycle. Heating the entire reactor is inefficient and research has shown that it is only necessary to heat the substrate on which the CNTs are grown. Current research demonstrates the feasibility of producing CNTs continuously, but a complete working reactor has yet to be realized. The design of the cold wall reactor for the continuous production of CNTs features a heating plate and a ‘reel-to-reel’ substrate feeding system housed in an insulated reactor vessel. The feeding system passes the thin flexible substrate across the heating element, raising it to the required temperature. Meanwhile, hydrocarbon gas is impinged upon the substrate, depositing the carbon required for CNT growth. This final design was developed and built into a prototype which will be tested by researchers at Northeastern University in a second phase of this project.

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The Need for Project

Current lab-scale carbon nanotube (CNT) production methods have processing times in excess of one hour, but the actual growth of CNTs only takes a few minutes. Heating the large thermal mass of the reactor is inefficient; it requires a large amount of power and a significant amount of time to heat and cool for each production cycle. Current research has shown that it is only necessary to heat the substrate on which the CNTs are grown. Past experiments elsewhere have demonstrated the feasibility of producing CNTs continuously, but a completely functional cold-gas reactor has yet to be realized.

The Design Project Objectives and Requirements

**Design Objectives**

The overall objective of the project is to design and build a cold wall reactor that will achieve and maintain conditions favorable to CNT growth. The reactor must be low cost, easy to implement, and able to operate continuously.

**Design Requirements**

The reactor needs to heat the substrate, a stainless steel mesh, up to 1000°C, which is the maximum ideal CNT growth temperature reported in previous literature. When the reactor is used to produce CNTs, it will be flooded with hydrocarbon gas, so the reactor needs to be airtight at atmospheric pressure and use non-corroding materials. The reactor also needs to operate continuously, moving the substrate at an adjustable but controllable speed around 1 mm/s. The reactor should significantly reduce overall processing time of CNTs as compared with traditional hot wall reactors.
Design Concepts Considered

Several design concepts were considered for the two main system functions, heating and substrate feeding. For the feeding system, a cartridge, magazine, and reel-to-reel design were proposed. For the heating system, applying current directly to the substrate, a heating plate, and a toaster oven style system were considered.

Feeding System

The feeding system introduces the substrate into the heating zone of the reactor, where CNTs are grown. Three main design concepts were considered. The first, the cartridge, would use a stack of substrates loaded into a cartridge, which would then be placed into the reactor and allowed to react. However, this would be more of a batch process rather than a continuous process. The ‘magazine’ method involves feeding an individual piece of substrate into the heating zone, allowing it to react, and then removing it and feeding the next piece into the heating zone. The ‘reel-to-reel’ method uses a design similar to a cassette tape to feed a flexible substrate continuously through the reaction zone.

Heating System

The heating system is the second primary system of the reactor; it is responsible for bringing the substrate to a set temperature between 700 and 1000°C. The first concept was to apply an electrical current directly to the substrate. Substrates used will be metallic foils or meshes, which have low resistivity and would allow for effective joule heating. One drawback would be that this method is not applicable to other substrates, such as powders. Using resistive heating, the substrate could be heated through the use of a hot plate. The substrate could then rest on the hot plate and heat would conduct to the substrate, heating it effectively and uniformly. This would also be applicable to other types of substrates. Similar to the idea of the hot plate, but without direct contact, the substrate could be placed between two hot plates in a ‘toaster oven’ configuration, which would also effectively heat the substrate.

Magazine feeding system concept

Heating element

Substrate

Toaster oven heating system concept

Heating elements

Substrate
**Recommended Design Concept**

The final reactor design is a stainless steel box which contains a hot plate embedded in insulation. A reel-to-reel system is used to feed the substrate over the hot plate with a gas feeding system diffusing gas over the surface of the substrate.

**Design Description**

The overall reactor design includes substrate feeding and heating systems as well as a gas feeding system. The substrate heating system is a nichrome heating element embedded in alumina-silica fiber insulation directly below the substrate feeding system. The substrate feeding system features a removable reel-to-reel cassette in which the roll of substrate is passed over the heating element to bring it to the desired temperature for CNT growth. The carbon bearing gas is impinged over the substrate and it diffuses on the substrate’s surface as the substrate moves across the heating element.

**Analytical Investigations**

The effects of heat transfer in the reactor were studied in several different ways during the system design. To be conservative, the heat transfer from the heating element to the insulation and walls was modeled using a one term approximation for transient conduction. The model considered that all of the heat from the heating element went into heating the walls and insulation. By varying parameters of thermal conductivity, $k$, it was found that a maximum acceptable thermal conductivity for the insulation was $0.2 \text{ W m}^{-1} \text{ K}^{-1}$. Also, a simplified radiative heat transfer model was implemented to determine if there was a minimum distance the walls must be from the heating element. Since the distance from the heating element to wall is in the macro-scale, a far field condition is used. The far field condition implies that the distance from the heating element to the walls is not critical. Furthermore, the effects of radiative heat transfer can be reduced using low emissivity materials on the walls. An ANSYS FEA model of the system was also run to gain a better understanding of the temperature gradient inside the reactor.

**Key Advantages**

The recommended design uses a reel-to-reel feeding system with a flat plate heating element. The reel-to-reel system is robust, controllable, and user-friendly. The design allows for maximum contact between the substrate and the heating element, heating it effectively and uniformly. Diffusing the gas perpendicular to the substrate allows for more of the gas to be utilized in the production of CNTs.
Financial Issues

Lab scale reactors are usually custom made by research groups. It is unlikely that this prototype design will reach mass production. To date, $1,400 has been spent on materials for the prototype of the reactor. The majority of the cost came from the need for specialized materials to withstand the high temperature corrosive environment inside the reactor. Lab scale reactors are usually custom built by research groups, and often cost at least two to five times this amount. It is unlikely that this prototype design would be commercially produced. However, the reactor was designed with future scalability of the process in mind.

Recommended Improvements

A second phase of this project should experimentally determine if CNTs can actually be grown in the prototyped reactor. The team designed the reactor to achieve conditions favorable to CNT growth. However, due to time constraints and health and safety concerns, it was not possible to test if CNTs can be grown in the prototyped cold wall reactor at this time. The second phase of this project will be to use the designed reactor to actually grow CNTs. In attempting to grow CNTs, it is likely that potential improvements to the system will be identified. One such improvement already identified is to find a heating element with a higher power density to reduce the ramp up time of the system.