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Douglas et al.

[11] **3,887,722**

[45] **June 3, 1975**

[54] **METHOD FOR PRODUCING A PLURALITY OF FILAMENTS IN A RADIO FREQUENCY REACTOR**

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[73] Assignee: **United Aircraft Corporation,** East Hartford, Conn.

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[52] **U.S. Cl.** **427/46;** 219/10.55; 219/10.61

[51] **Int. Cl.** **C23c 11/00**

[58] **Field of Search**..... 117/93, 93.2, 106 R, 107.1, 117/228, 231; 219/10.55, 10.61

[56] **References Cited**

UNITED STATES PATENTS

3,471,321	12/1969	Qualline et al.	117/107.1
3,572,286	3/1971	Forney	117/93.2

Primary Examiner—William D. Martin
Assistant Examiner—John H. Newsome
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[57] **ABSTRACT**

A process for producing, simultaneously, a plurality of low density, high modulus filaments by vapor deposition on a moving wire substrate heated by electrical power dissipation involves passing all the wires through a single reactor simultaneously and precisely positioning the wires within the reactor and maintaining their position during deposition.

10 Claims, 3 Drawing Figures

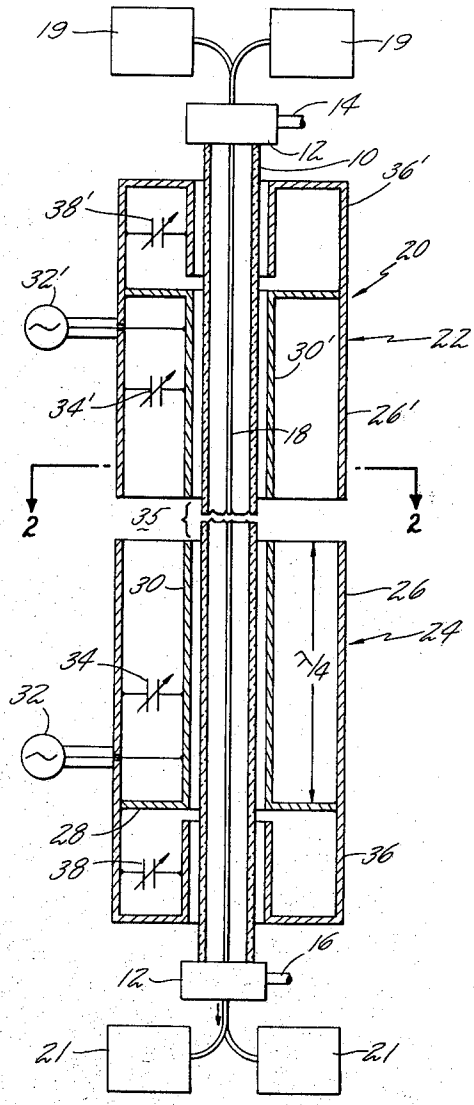


FIG. 1

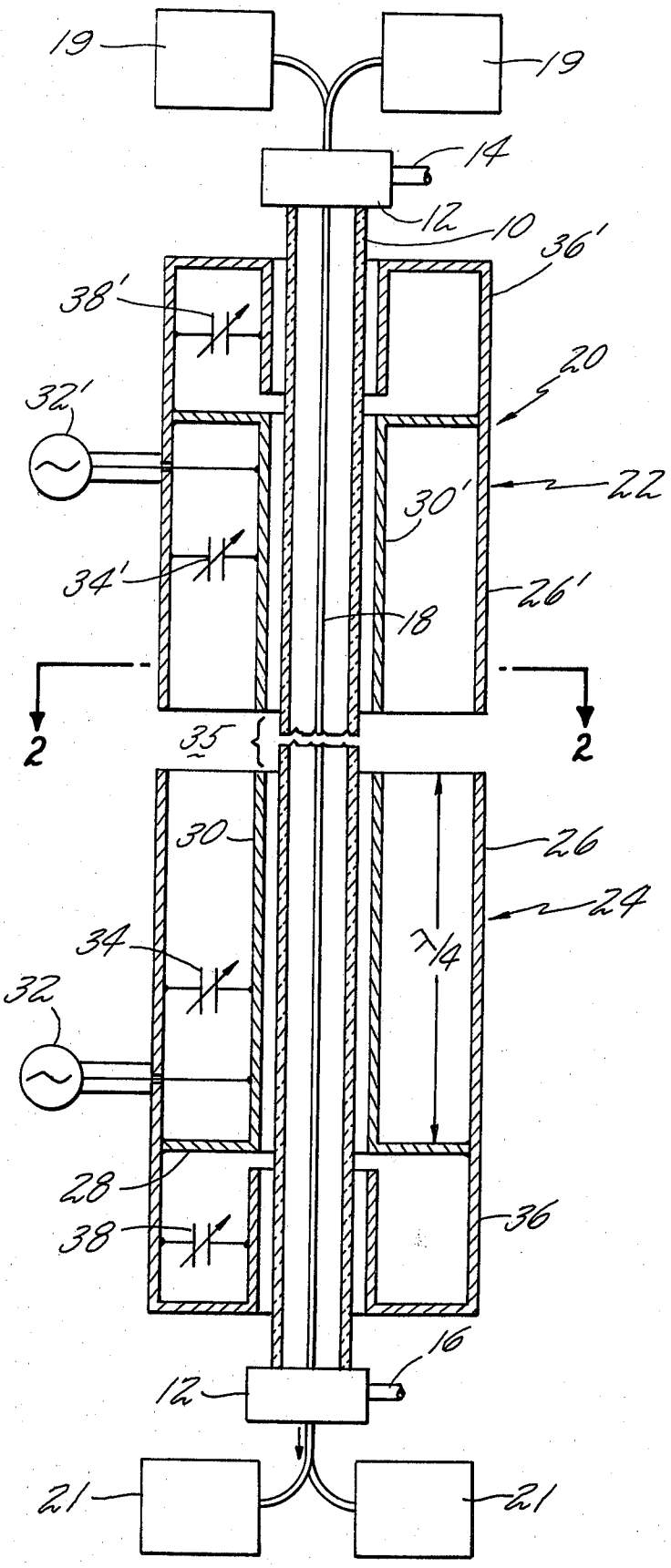

19
19
14
12
10
36'
38'
20
32'
22
30'
34'
26'
18
2
2
35
30
26
24
34
λ/4
32
28
38
36
16
12
21
21

FIG. 2

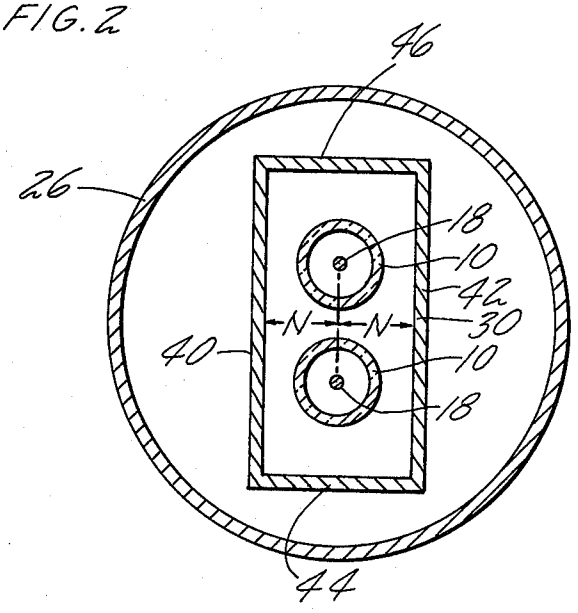
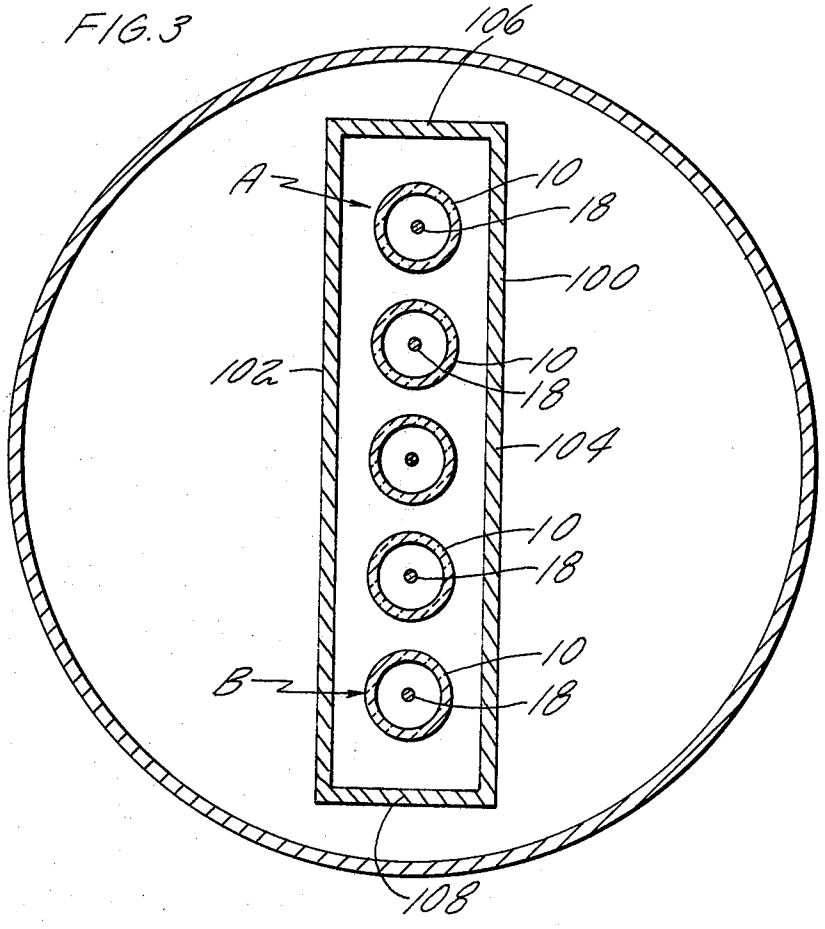

46
26
18
10
42
N
N
30
40
10
18
44

FIG. 3


106
A
10
18
100
102
10
18
104
10
18
B
10
18
108

METHOD FOR PRODUCING A PLURALITY OF FILAMENTS IN A RADIO FREQUENCY REACTOR

BACKGROUND OF THE INVENTION

This invention relates to a process and apparatus for simultaneously depositing a coating on a plurality of moving wire filaments, the moving filaments being heated by radio frequency energy.

The use of low density, high modulus filaments as a reinforcement material in lightweight composite structures has become more and more important in recent years. In particular, filaments of boron, produced in a reactor by a method which involves the chemical reduction of a boron halide onto a direct current resistively heated tungsten wire substrate, have found wide application in meeting the stringent demands of the aerospace industry. Such a method is shown, for example, in U.S. Pat. No. 3,549,424 to Rice and sharing the same assignee as the present application. An alternate method for producing such filaments has come into favor recently wherein the substrate wire is heated by energy in the radio frequency range rather than being resistively heated by a direct current. The radio frequency energy is transmitted to the fiber by a radio frequency energy coupler. Details of such a process are found in copending U.S. application Ser. No. 225,350 filed Feb. 10, 1972 now U.S. Pat. No. 3,811,940 by Douglas, Gregory and Stielau, which is a continuation of abandoned U.S. application Ser. No. 865,157 filed Oct. 9, 1969 now abandoned.

As the demand for these reinforcing filaments increases it is desirable to find techniques for reducing the cost and increasing the speed at which these filaments are produced. One technique which has been suggested is to simultaneously produce a plurality of filaments using a single reactor. For example, this suggestion was made on page 11 lines 10–12 of the aforementioned Douglas et al copending application (column 5, lines 59–64 of the patent). While this suggestion is no doubt an obvious one, it is difficult to put into practice, and, as far as the Applicants know, it has not been successfully accomplished at this time by anyone other than themselves.

SUMMARY OF THE INVENTION

An object of the present invention is a process for simultaneously producing a plurality of low density, high modulus filaments using a single reactor.

A further object of the present invention is a process for simultaneously producing a plurality of low density, high modulus filaments having substantially the same diameter and physical properties using a single reactor.

Accordingly, in one aspect of this invention a plurality of suitable wire substrates, such as tungsten, are simultaneously drawn through a radio frequency reactor which includes coupler means having wall means forming a channel aligned with the axis of the wires; the wires are precisely positioned as they move through the channel to assure that each of the finished wires exiting from the reactor are substantially the same in diameter and physical properties.

Initial attempts to produce a plurality of identical filaments simultaneously using a single reactor were not particularly successful. Additional experimentation lead to the discovery that the positions of the wires with respect to the radio frequency energy coupler and with respect to neighboring wires was critical and had to be precisely controlled. It was also discovered that certain channel shapes give better results.

The foregoing and other objects, features and advantages of the present invention will become more apparent in the light of the following detailed description of preferred embodiments thereof as illustrated in the accompanying drawing.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is an elevational view, in section, of a radio frequency reactor for producing a plurality of filaments simultaneously;

FIG. 2 is a cross sectional view taken along the line 2—2 of FIG. 1 showing the preferred arrangement for two wire substrates; and

FIG. 3 is a cross sectional view taken along the line 2—2 of FIG. 1 illustrating the preferred arrangement of more than two wire substrates.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring now to the drawing, where similar numerals indicate similar parts, an apparatus suitable for carrying out the process is schematically shown in FIGS. 1 and 2. The apparatus as shown is designed for producing only two filaments for the purposes of clarity; however, it will become apparent that this apparatus may readily be modified to handle many more filaments as hereinafter further discussed.

The apparatus comprises a pair of vertical reaction chambers **10** (FIG. 2) which may be made of glass or quartz, fitted at both ends with appropriate closure means **12** through which suitable gas inlets **14** and outlets **16** pass for communication with the chambers **10.** In this embodiment each chamber **10** has its own inlet **14** and outlet **16.** The closure means **12** permit axial passage of each wire substrate **18** through the reactor, the closure means containing suitable fluids, such as an inert gas or liquid mercury or the like to seal the chambers **10** from the atmosphere. Although in this embodiment the wire travels downward in the drawings (from the top toward the bottom of the page), direction of movement is not critical and upward movement will produce satisfactory results.

Each wire substrate **18** is fed into its respective reaction chamber from its own payoff unit **19,** which maintains a constant tension on the wire. After passing through the reaction chamber **10,** each finished filament is received onto take-up means **21.**

The reaction chambers **10** are received within a quarterwave phased, tuned coaxial cavity coupler generally represented by the numeral **20** which comprises upper and lower coaxial cavities **22, 24** respectively in symmetrical relation. Each coupler **20** includes an outer electrically conductive enclosure **26,** which supports, by annular flange **28,** an inner inductor channel **30.** As shown in the drawing, power is fed in by a coaxial lead from a radio frequency power source **32.** A variable capacitor **34** is provided between the enclosure **26** and channel **30** for adjusting (i.e. tuning) purposes. By placing two such configurations in axial alignment with the proper phasing to obtain a phased, coaxial cavity, there is created between the couplers **20** an oscillating axial electric field of high density. This field induces an alternating electric current in the wires **18** in the area **35** between the upper and lower coaxial cavities **22, 24.** This area **35** is known as the hot zone. Escaping energy

is reflected back into the hot zone **35** by a resonant energy trap **36,** tunable by a variable capacitor **38.**

The basic goal of an apparatus such as the one as hereinabove described is to produce a plurality of filaments having substantially identical diameters and strength. During experimental runs of the above-described apparatus and other apparatus of a similar nature several parameters were discovered which had a significant effect on the similarity between filaments being produced simultaneously in the same reactor. It was determined, for example, that the alternating current flowing through one wire could induce a current in an adjacent wire and thereby affect the temperature of the adjacent wire resulting in variations in the final diameter and strength of the filaments. The amount of power or current induced into an adjacent wire depends on several factors such as how well each wire is coupled to the main array, and the diameter of the substrate and its proximity to the adjacent wire. In order that this interaction between fibers results in negligible diameter and strength differences, the wires in the apparatus of the preferred embodiment are spaced apart such that the power induced in an adjacent wire is less than 5 percent of the total power in the wire.

It is also known that the quality and speed of the deposition process is effected by the proximity of the wire substrate to the inductor channel **30.** Of course, the farther away the wire substrate **18** is from the wall of the channel **30** the lower the power in the wire. While all the walls of the channel **30** will have some effect on the power in the wire **18,** some of the walls and portions thereof will have a negligible effect, relative to the effect of the nearest walls. In this specification and in the claims appended hereto a wall or portion thereof of the channel **30** which has an effect on the power induced in the wire which is not negligible is hereinafter referred to as an "affecting wall." In order that the plurality of filaments produced simultaneously within a single channel **30** are substantially the same diameter and the same strength, each wire is positioned, relative to affecting walls, in the same manner as every other wire within the channel **30;** also, each wire of this preferred embodiment is spaced the same distance from adjacent wires to nullify possible effects caused by adjacent wires as hereinabove discussed. In the preferred embodiment shown in FIG. **2** each wire **18** is positioned the same distance from the side wall **40** as from the side wall **42,** as well as being positioned the same distance from the end wall **44** or from the end wall **46.** Thus each wire **18** sees an identical environment. The more nearly identical the placement of each fiber is relative to its neighboring fiber and to the coupler **20,** the greater will be the similarity between the fibers produced.

Initially it was not felt that precise positioning of each wire **18** would be necessary. However, initial unsatisfactory test results proved this to be incorrect. Assuming, in this preferred embodiment, that the nominal distance of each wire **18** from a particular adjacent affecting wall, such as the wall **42,** is N then the position of each wire **18** should be maintained within **2** percent of N from that wall to ensure reasonable identity of environment.

Although the channel **30** of the preferred embodiment is rectangular in cross section, it is contemplated that other shapes may be used. For example, the cross section of the channel **30** may be circular; in that instance all of the foregoing criteria are still valid. In other words the spacing between wires should be the same and they should not be too close to each other such that relatively large currents are induced therein by adjacent wires; also, each wire should be maintained within **2** percent of a nominal distance N from the nearest wall. If only two wires are run they should be 180° apart (i.e. on a diameter).

A further problem which is felt to have an effect on the production of multiple fibers from a single reactor is the fact that the wires **18** vibrate as they move through the chambers **10,** thus changing the wires position relative to the walls of the channel **30** and to other fibers within the channel. While it is virtually impossible to eliminate all vibrations, the constant tension units **19** are beneficial in this regard. While some satisfactory experimental runs were made without constant tension units, the best results were obtained with these units. With respect to the problem of vibration the rectangular shape of the channel **30** is beneficial and is the preferred shape. That is because a wire **18** vibrating from left to right within the channel **30** continuously crosses an imaginary plane positioned midway between the walls **40, 42.** Thus, each swing to the left is theoretically nullified in its effect on the deposition process by a corresponding swing to the right, wherein the average position of the wire is precisely in the imaginary plane.

While the preferred embodiment shows each wire **18** as being within a separate reaction chamber **10,** satisfactory results may be obtained by using a single reaction chamber surrounding both wires. However, there are advantages to having individual reaction chambers surrounding each fiber. One advantage is that if a wire breaks it remains segregated from other wires within the channel **30** and can do no additional harm, such as by coming into contact with the other wires necessitating a shutdown of the entire reactor. Another advantage is that the gas supplies to each wire **18** can be individually controlled and may be varied to compensate for other variations within the system thereby counteracting them so that all the wires produced within a single reactor are the same size and strength.

The following table gives boron fiber test data from runs of the apparatus of the preferred embodiment hereinabove described:

TABLE I

Run No.	Avg. Fiber Dia. (Mils)		Avg. Fiber Strength (KPSI)	
	Fil. No.1	Fil. No.2	Fil. No.1	Fil. No.2
1	2.5	2.6	414	329
2	4.35	4.15	556	460
3	4.1	4.1	546	464
4	4.1	4.0	453	420
5	5.4	5.4	436	563

The average diameter of the fibers in Table I is given in mils while the average ultimate tensile strength is given in thousands of pounds per square inch. It should be noted from this table that in every run except one the diameters of the pair of fibers produced did not differ by more than .0001 inch. Although better correspondence between the ultimate tensile strength of the fibers produced in these tests is desirable, the results shown in Table I are considered to be quite good and the strength of the fibers produced, except in one in-

stance, was always greater than 400,000 pounds per square inch.

FIG. 3 is illustrative of how more than two wire substrates **18** should be arranged within a reactor having a rectangular channel **100.** Five wires **18** are shown by way of example. All are positioned within an imaginary plane spaced equidistant from the side walls **102, 104** of the channel **100,** each wire being spaced the same distance from the next adjacent wire. It is recognized that the end wires designated A and B do not have wires on each side thereof, but rather are adjacent an end wall **106, 108** respectively. Variations in the diameter and strength of filaments from wires A and B (if any) due to their end positions may be corrected by modifying the flow of gases through their respective chambers **10.** It may also be desirable to space the end wires A, B a fair distance from the end walls **106, 108** so that these end walls are not affecting walls as hereinabove defined. If adjacent wires are spaced sufficiently far apart such that their effect on each other is very small then the fact that the end wires do not have a wire on either side thereof should not be cause for concern.

Although in the embodiments of FIGS. 2 and 3 the channels **30, 100,** respectively, are rectangular. Their advantage lies in the fact that the side walls **40, 42** and **102, 104** of the channels have their surfaces parallel, thereby making it easier to position each wire such that it sees the same environment as each of the other wires.

Although the invention has been shown and described with respect to preferred embodiments thereof, it should be understood by those skilled in the art that various changes and omissions in the form and detail thereof may be made therein without departing from the spirit and the scope of the invention.

Having thus described typical embodiments of our invention, that which we claim as new and desire to secure by Letters Patent of the United States is:

1. A process for simultaneously producing a plurality of filaments using a single electrical heating unit by vapor depositing material from a material containing decomposable gas onto a plurality of moving wires heated by electrical power dissipation therein wherein the impedance of the wire changes during deposition, comprising the steps of:

passing said wires through a single coaxial cavity coupler means to electromagnetically couple said wires to radio frequency source means thereby exposing said wires to an axially oscillating electric field having a density sufficiently high to heat the wires and cause deposition of said material thereon, said coupler means including wall means forming a channel aligned with the axis of said wires;

positioning each of said wires within said channel at the same nominal distance from adjacent affecting walls of said channel, and;

maintaining the position of each wire within **2** percent of said nominal distance from said affecting walls.

2. The process according to claim **1** including the step of spacing said wires from each other such that the power induced in each wire by adjacent wires is less than 5 percent of the total power in said wire.

3. The process according to claim **2** wherein the step of spacing said wires includes locating each wire equidistant from adjacent wires.

4. The process according to claim **3** wherein the step of maintaining the position of each wire includes maintaining constant and equal tension on each of said wires to minimize vibration of the wires.

5. The process according to claim **3** including the step of enclosing each of said wires within its own individual reaction chamber.

6. The process according to claim **5** including the step of varying the gas flows within said individual reaction chambers to compensate for uncontrollable variations in the deposition buildup on each wire.

7. The process according to claim **1** wherein said affecting walls include spaced apart parallel wall surfaces, and the step of positioning said wires includes locating each wire equidistant from each of said surfaces.

8. A process for simultaneously producing a plurality of filaments using a single electrical heating unit by vapor depositing material from a material containing decomposable gas onto a plurality of moving wires heated by electrical power dissipation therein wherein the impedance of the wire changes during deposition, comprising the steps of:

passing said wires through a single coaxial cavity coupler means to electromagnetically couple said wires to radio frequency source means thereby exposing said wires to an axially oscillating electric field having a density sufficiently high to heat the wires and cause deposition of said material thereon, said coupler means including wall means forming a channel aligned with the axis of said wires, said wall means including spaced apart parallel wall surfaces;

enclosing each of said wires within its own individual reaction chamber within said coaxial cavity coupler;

maintaining constant and equal tension on each of said wires;

positioning each of said wires equidistant from adjacent wires; and

positioning each of said wires equidistant from each of said wall surfaces.

9. The process according to claim **8** wherein said wires are positioned a nominal distance N from each of said wall surfaces including the step of maintaining the position of each wire within two percent of N from said wall surfaces.

10. The process according to claim **9** including the step of varying the gas flows within said individual reaction chambers to compensate for uncontrollable variations in the deposition buildup on each wire.

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