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(54) **RADIAL PULSED ARC DISCHARGE GUN FOR SYNTHESIZING NANOPOWDERS**

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See application file for complete search history.

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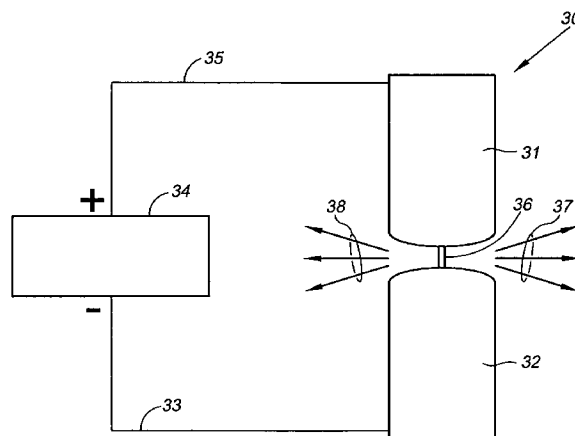
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(57) **ABSTRACT**

A system and method for synthesizing nanopowder which provides for precursor material ablation from two opposing electrodes that are spaced apart within a gaseous atmosphere, where a plasma is created by a high power pulsed electrical discharge between the electrodes, such pulse being of short duration to inertially confine the plasma, thereby creating a high temperature and high density plasma having high quench and/or reaction rates with the gaseous atmosphere for improved nanopowder synthesis.

19 Claims, 2 Drawing Sheets



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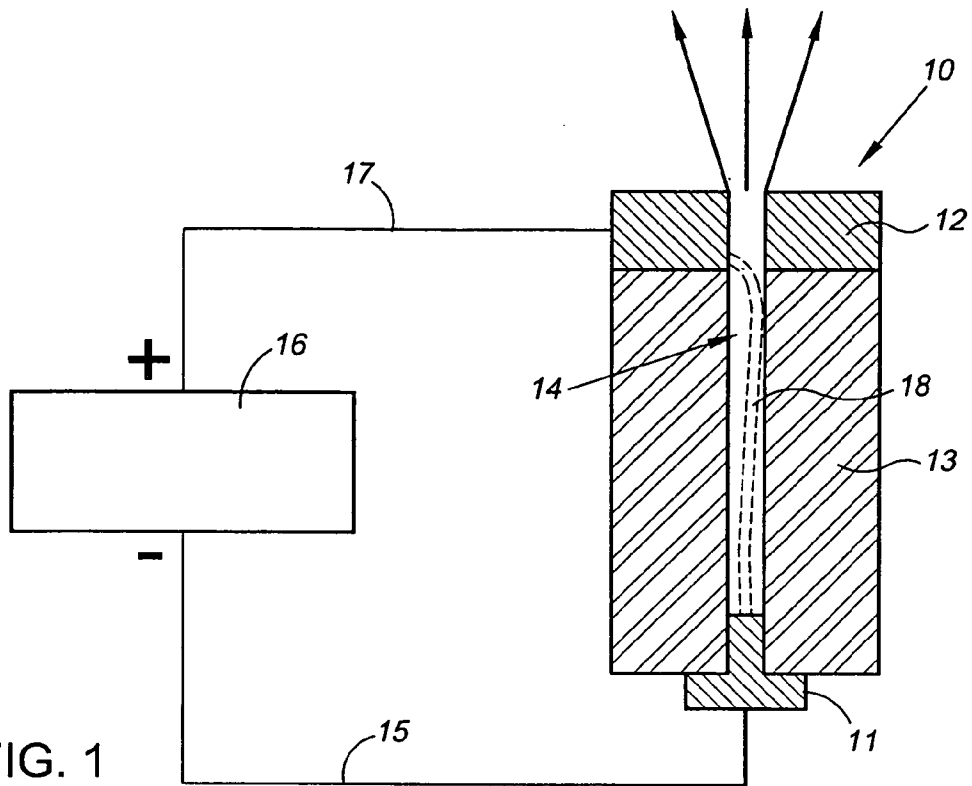


FIG. 1
PRIOR ART

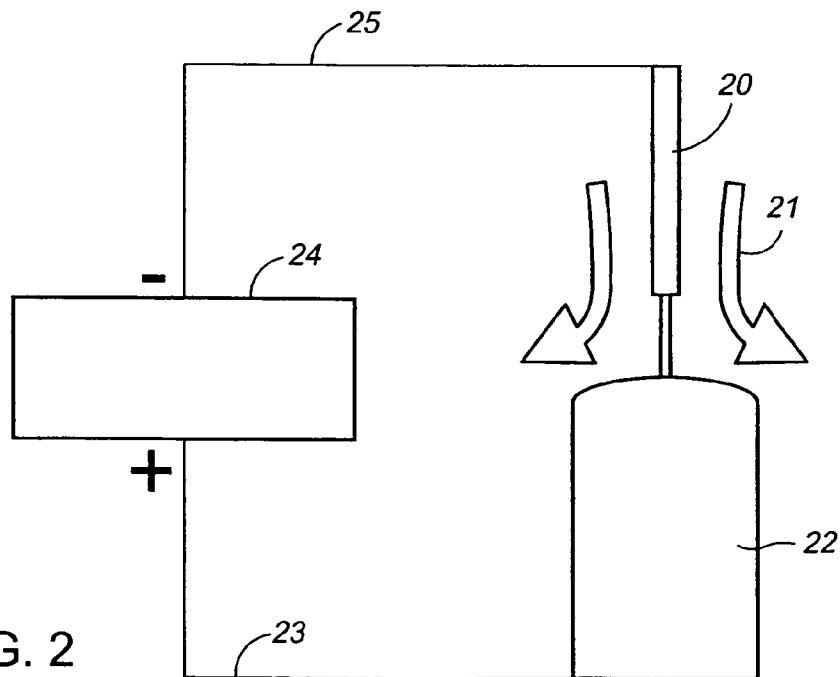
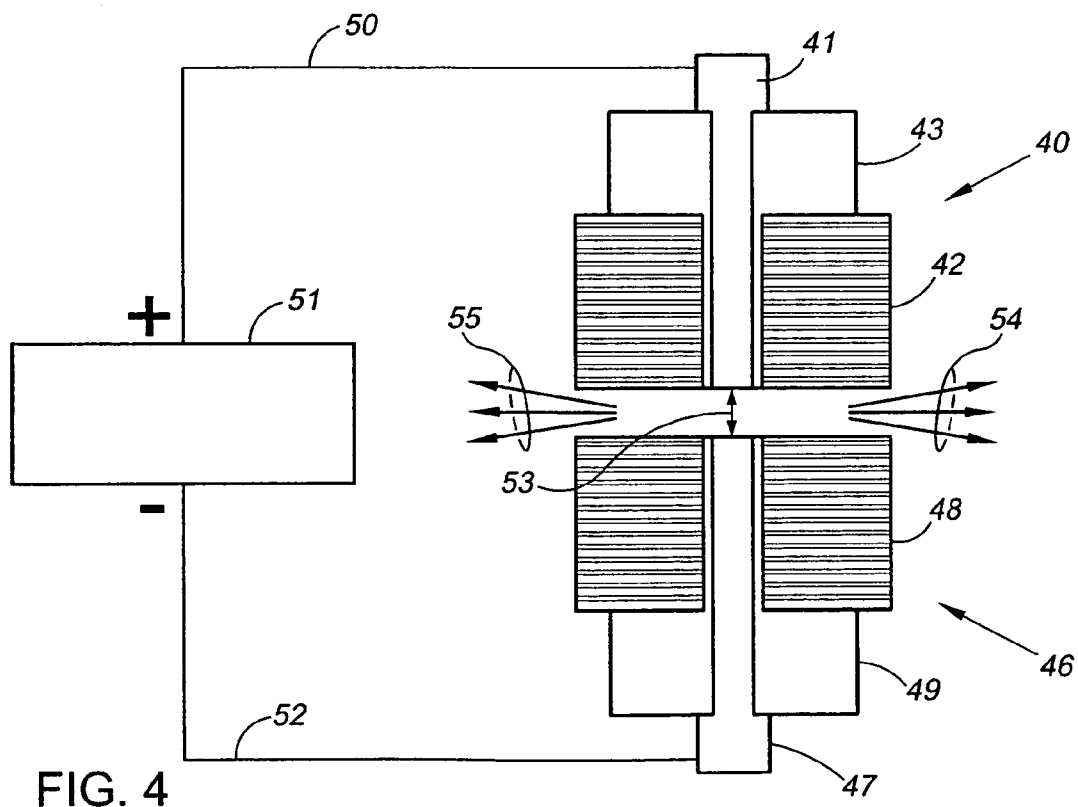
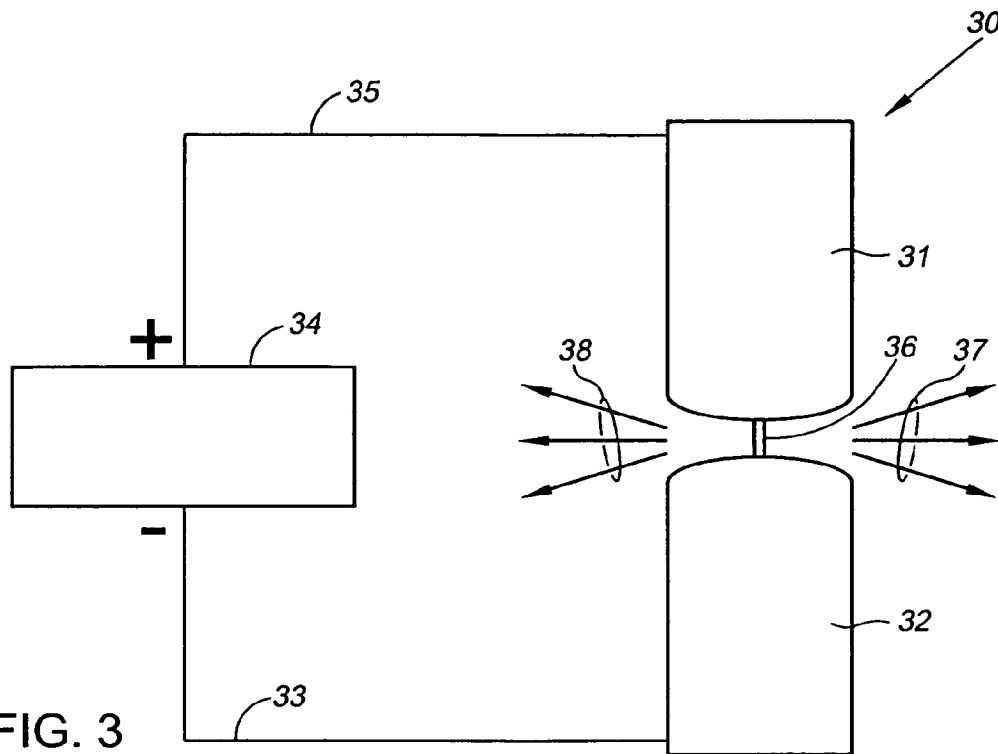


FIG. 2
PRIOR ART



RADIAL PULSED ARC DISCHARGE GUN FOR SYNTHESIZING NANOPOWDERS

PRIORITY DATE CLAIM

This patent application is a continuation of U.S. patent application Ser. No. 10/455,292, "RADIAL PULSED ARC DISCHARGE GUN FOR SYNTHESIZING NANOPOWDERS," with named inventors Kurt Schroder and Doug Jackson, filed Jun. 5, 2003, now U.S. Pat. No. 6,777,639 which claims the benefit of the earlier filing date of the U.S. Provisional Patent Application Ser. No. 60/388,200, filed Jun. 12, 2002, with named inventors Kurt Schroder and Doug Jackson, and all of which are assigned to the assignee of the present invention.

BACKGROUND OF THE INVENTION

Systems for producing nanopowders are known which ionize an inert gas to create a plasma in a reactor vessel that vaporizes a precursor material. A quench and/or reaction gas is injected into the vaporized precursor material to produce nanopowders having a desired composition. U.S. Pat. No. 5,514,349 discloses a transferred arc system similar to a tungsten inert gas (TIG) welder, wherein a single rod of metal precursor material acts as an anode, and is vaporized by feeding the anode past a nonconsummable tungsten cathode to expose the anode to a discharge arc. A gas is then injected into the vaporized material to quench and form the nanopowder. The transferred arc system is designed to avoid erosion of the tungsten electrode.

U.S. Pat. No. 6,472,632 discloses another method to produce nanopowders which uses a prior art axial electrothermal gun, as illustrated in FIG. 1. The axial electrothermal gun or axial gun **10** is shown with a breech electrode **11**, an annular muzzle electrode **12**, and a barrel **13** having a hollow bore **14**. The breech electrode **11** fits into and pneumatically seals one end of the hollow bore **14**. The muzzle electrode **12** is attached to and substantially axially aligned with the barrel **13**. The breech electrode **11** further is connected by way of a conducting wire **15** to the negative terminal of a high-power, pulsed discharge power supply **16**, the positive terminal of which is connected by way of a conducting wire **17** to the muzzle electrode **12**. Unlike the transferred arc system, the polarity of the electrodes in the axial gun **10** is not important, and the device can be operated with the polarity reversed. This axial gun embodiment has been successful in producing moderate volumes of nanopowder in the 10–100 nanometer range.

In operation, the power supply **16** is energized to create an electric field between the breech electrode **11** and the muzzle electrode **12**, and thereby discharge a high power pulsed arc **18** between the electrodes. The discharge of the pulsed arc **18** ablates the muzzle electrode **12**, which is the primary source for plasma. More particularly, the material removal rate from the muzzle electrode **12** is a factor of 10–100 greater than the material removal rate from the breech electrode **11**.

The pulsed arc discharge in the axial gun **10** occurs in an interval of time too short to allow the plasma to fully expand and equilibrate in the surrounding atmosphere (inertial confinement), and in an area physically confined by the walls of the bore **14** (physical confinement). The combined confinement creates a high temperature (~50,000° K.), high density (~10²⁰/cm³) plasma which is not ordinarily obtainable in other plasma based systems such as those disclosed by U.S. Pat. Nos. 5,514,349; 5,874,684; and 5,851,507. The plasma acts to ablate the muzzle electrode **12** by means of the rapidly exiting high-temperature plasma. The plasma together with the ablated material exits the muzzle **12** under

high pressure (~15,000 PSI) and supersonic velocity. The ablated material thereafter may be quenched by and/or reacted with a surrounding gas such as one or more of air, oxygen, nitrogen, or helium to produce a cloud of nanopowder.

Referring to FIG. 2, a prior art transferred-arc-discharge process is illustrated in which a tungsten electrode **20** is shielded in a flow of pure inert gas **21** such as Argon, and is principally aligned with a rod **22** of precursor material. The inert gas shield protects the tungsten electrode **20** from erosion and oxidation. The inert gas ionizes to sustain the arc, but does not act to quench or react with material removed from rod **22**.

Rod **22** is connected by way of a conducting wire **23** to the positive terminal of a DC power supply **24**, the negative terminal of which is connected by way of a conducting wire **25** to the tungsten electrode **20**. The tungsten electrode **20** is charged negatively with respect to the rod **22** to retard the absorption of heat and rate of erosion of the tungsten electrode. With these polarities, the material removal rate from the rod **22** is a factor of 100–1000 times greater than that of the tungsten electrode **20**.

In operation, the DC power supply **24** is energized to effect a continuous DC low power arc discharge between the tungsten electrode **20** and the rod **22**. The arc discharge erodes rather than ablates the rod **22**. The material so produced is conveyed away from the vicinity of the arc discharge by the flow of the pure inert gas **21**, and injected with a quench and/or reaction gas(es) **21**, such as argon, helium and oxygen, to form the nanopowder.

From the above, it should be readily apparent that material removal in the operation of a transferred arc process, or an axial gun process is primarily from a single electrode.

BRIEF DESCRIPTION OF THE DRAWINGS

While the novel aspects and features of the invention are defined in the appended claims, the principles of the invention, illustrative embodiments, and preferred modes of use are best understood by reference to the Description Of Preferred Embodiments in conjunction with the following drawings, in which:

FIG. 1 is a graphic diagram of an axial electrothermal gun in the prior art, which is comprised of a breech electrode, a muzzle electrode, and a hollow bore;

FIG. 2 is a graphic diagram of a transferred-arc-discharge process in the prior art, which is comprised of a tungsten electrode that is charged negative with respect to an anode electrode composed of precursor material;

FIG. 3 is a graphic diagram of a radial electrothermal gun in accordance with the present invention, wherein an anode and a cathode of the gun are each composed of a precursor material, and are substantially axially aligned but spaced apart opposite to each other; and

FIG. 4 is a graphic diagram of an alternative embodiment of the present invention wherein the anode and cathode electrodes are placed within an ablative body, but are separated from the ablative electrodes by an insulator.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

The following terms shall have the definitions given below when used in either lower case or with capitalizations in this specification:

"Ablation" shall mean the removal of material from a body of precursor material due to the combined effects of heat transfer and mechanical shear caused by high fluid velocities.

"Erosion" shall mean the removal of material from a body of precursor material through localized heat transfer, as occurs when an electrical discharge arc attaches to a surface.

"Axial Gun" shall mean a pulsed power electrothermal gun, wherein a breech electrode and a muzzle electrode are composed of a precursor material, and are separated by a barrel with a bore.

"Radial Gun" shall mean a pulsed power electrothermal gun wherein two opposing electrodes of the gun are composed of a precursor material which is to be ablated, and the electrodes are substantially axially aligned opposite to each other within a gaseous atmosphere. The term "radial gun" is used for convenience only, and is not meant to imply any limitation of the present invention.

"Nanopowder" shall mean nanomaterial primarily comprised of nanoparticles which are of a size of 1–500 nanometers (nm).

Referring to FIG. 3, a radial gun 30 in accordance with the present invention is illustrated, wherein an anode electrode 31 and a cathode electrode 32 (each formed of a precursor material and of uniform cross section) are spaced apart opposite to each other, and substantially axially aligned for maximal production of nanopowders. The electrode composition is typically the metal of the nanopowder being produced, such as but not limited to aluminum, tantalum, titanium, or zirconium. The nanopowder itself may be, but is not limited to, a metal such as aluminum or copper; an oxide such as Al_2O_3 or TiO_2 ; a nitride such as TiN, ZrN, or Ta_2N ; or other metals and metal compounds. Please refer to the article, "Pulsed Wire Discharge for Nanosize Powder Synthesis", by Weihua Jiang and Kiyoshi Yatsui, IEEE Transactions On Plasma Science, vol. 26, No. 5, pp. 1498–1501 (October 1998).

The cathode electrode 32 is connected by way of a conducting wire 33 to the negative terminal of a pulsed power supply 34, the positive terminal of which is connected by way of a conducting wire 35 to the anode electrode 31. The pulsed power supply 34 may be of any of plural well known designs (which may vary depending upon the material and material size being produced) that are offered by and ordered from any of numerous well known manufacturers. Except for the pulsed power supply 34, the radial gun 30 may but need not be contained within a chamber or reactor vessel such as that disclosed in U.S. Pat. No. 5,514,349. The reactor vessel is filled with inert and/or reactive gases including but not limited to Argon, Nitrogen, Oxygen, or a combination thereof. The selection of the gaseous atmosphere is based upon the nanopowder that is desired, and the effect of the gas is to control the expansion and quench rate of the plasma that is created by a high-power pulsed electrical discharge between the anode electrode 31 and the cathode electrode 32.

In operation, the pulsed power supply 34 is energized to effect a high-power pulsed electrical discharge 36 between the cathode electrode 32 and anode electrode 31. For the current invention, the connection to the electrodes could easily be reversed in polarity without impacting the process. The energy from the discharge melts, vaporizes and ionizes material from the two electrodes to create a high temperature (of the order of $50,000^\circ K$), high density metal plasma which continues to sustain the electrical discharge from the pulsed power supply. The combined physical axial confinement provided by the electrodes and the inertial confinement resulting from the short term pulse of the electrical discharge, act to increase the temperature and density of the plasma to heights not ordinarily obtainable by other nanopowder synthesizing processes or apparatus. Since the electrodes impede axial expansion of the plasma, expansion occurs primarily radially as indicated by arrows 37 and 38 of FIG. 3. As the plasma expands, additional material is

ablated from the electrodes. This additional material also contributes to the overall plasma and helps sustain the electrical discharge between the electrodes. The expanding plasma is forced out of the confines of the radial gun at supersonic speeds. It subsequently undergoes expansion, mixes with the chamber gases, and quenches at a rate of the order of $10^{6^\circ} K/sec$ to form nanopowder. Depending on the ambient gas, the plasma may also react with the gaseous atmosphere to form compounds as it is being quenched.

After each high power electrical discharge, the pulsed power supply 34 is reenergized. Further, after nanopowder is produced from one or more high power, pulsed electrical discharges between the electrodes, the anode electrode 31 and the cathode electrode 32 are indexed toward each other only as required to maintain the production rate. The gas surrounding the electrodes is replenished as necessary to sustain the atmosphere within the reactor vessel.

It is to be understood that the above process may occur without the benefit of a reactor vessel or chamber, as the plasma created by the present invention need only be quenched by or reacted with a surrounding gaseous atmosphere to produce a desired nanopowder.

TABLE I

SYNTHESIZING DEVICE	ELECTRODE	PRIMARY MATERIAL REMOVAL MECHANISM	RELATIVE AMOUNTS OF MATERIAL REMOVED
Transferred Arc	Tungsten	Arc Erosion (Minimized)	1
	FeedStock	Arc Erosion	100–1000
Axial Gun	Breech	Arc Erosion	1
	Muzzle	Ablation	10–100
Radial Gun	Electrode 1	Ablation	~1
	Electrode 2	Ablation	~1

In Table I above, relative removal rates between electrodes of a same nanopowder synthesis device are shown. Table I is not intended to show comparisons between synthesis devices.

Referring to Table I, it is seen that the two electrodes of the radial gun each serve almost equally as sources of ablative material. By way of contradistinction, both the axial gun and the transferred arc synthesizing devices have only one electrode that contributes to any substantial degree to the production of nanopowder.

Readily apparent differences between the transferred-arc processes and the radial gun are: (1) polarity reversal is of little to no effect in the radial gun, because the electrode material removal caused by erosion is negligible compared to the material removal from ablation; (2) material removal with a radial gun occurs at two electrodes rather than a single electrode; (3) no inert gas shielding is required by the radial gun to protect against electrode erosion; and (4) the power supply of the radial gun is pulsed rather than continuous.

The radial electrothermal gun also provides the following additional advantages over the transferred-arc processes for synthesizing nanopowder:

- i. Increased quench rate of 10^6 – 10^8 degrees Kelvin/sec. with the radial electrothermal gun (as compared to 10^4 degrees Kelvin/sec. with the forced convection quenching used with the transferred arc processes) allows smaller sized nanopowder to be synthesized in higher aerosol densities;
- ii. More uniform time-temperature nanoparticle synthesis, i.e. nucleation and growth, histories are provided with a radial gun which result in more uniform nanoparticle sizes;

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iii. The increased plasma temperature occurring in a radial gun allows the synthesis of nanopowder from material having very high melting and boiling points;

iv. Less energy is used to produce greater quantities of nanopowder because the short duration of the pulsed discharge does not allow sufficient time for thermal diffusion. Consequently, there are fewer thermal (energy) losses to the surrounding environment; and

v. Lower agglomeration of nanopowder occurs with the radial gun.

vi. Since both the cathode and the anode of the radial gun are made out of the precursor or ablative material, no contamination is introduced from the electrodes. Inherent to the transferred arc process is the contamination that comes from the tungsten electrode.

The radial electrothermal gun provides advantages including the following over the axial electrothermal gun in synthesizing nanopowder:

i. No high pressure seals are needed since the only physical confinement comes from the electrodes;

ii. No insulating bore is required. Thus, a source of both major cost, and impurities in the production of nanopowder is removed;

iii. The electrodes can be large diameter rods requiring almost no machining, thus further reducing cost; and

iv. The electrodes are nearly all consumed.

A further comparison among the radial gun of the present invention, and prior art axial guns and transferred arc processes appears, in Table II below.

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for a given arc current. The resulting plasma may be at a lower temperature and/or lower density than otherwise could be achieved, production rates are decreased, and the material produced is of a non-uniform quality. A solution to this problem is provided by the radial gun embodiment of FIG. 4.

Referring to FIG. 4, an alternative embodiment of the present invention is illustrated in which a first composite electrode 40 is comprised of a solid anode electrode 41 which is seated within a wider annular ablative body 42. The anode electrode 41 is electrically isolated from the annular ablative body 42 by an annular insulator body 43. Similarly, a second composite electrode 46 is comprised of a solid cathode electrode 47 which is seated within a wider annular ablative body 48. As before, the cathode electrode 47 is electrically isolated from the annular ablative body 48 by an annular insulator body 49.

The anode electrode 41 is connected electrically by way of a conducting wire 50 to the positive terminal of a high power, pulsed power supply 51, the negative terminal of which is electrically connected by way of a conducting wire 52 to the cathode electrode 47. The composite electrodes 40 and 46 are separated by an axial distance 53.

The anode electrode 41, cathode electrode 47, annular ablative body 42, and annular ablative body 48 may be of the same material. The annular ablative bodies 42 and 48 also may be of materials different from that of the anode electrode 41 and the cathode electrode 47, and even may be nonconductors since their purpose is to provide axial physi-

TABLE II

	RADIAL GUN	AXIAL GUN	TRANSFERRED ARC DISCHARGE
Peak Discharge Power (Watts)	10 ⁸	10 ⁸	10 ⁵
Duty Cycle	Pulse	Pulse	Continuous
Pulse Length (sec.)	10 ⁻³	10 ⁻³	Infinite
Bore	None	Yes	None
Arc Confinement	Physical In Axial Direction, Inertial In Radial Direction	Physical In Radial Direction, Inertial In Axial Direction	None
Arc Temperature °K	50,000	50,000	10,000
Plasma Quench Mechanism	Supersonic Expansion Into Surrounding Atmosphere	Supersonic Expansion Into Surrounding Atmosphere	Externally Forced Convection
Plasma Expansion Between The Electrodes	Primarily Radial	Axial	N/A
Electrode Ablation	Both Electrodes	Primarily Muzzle Electrode, Lesser Extent Breech	None. Tungsten Electrode Erosion Is Minimized. Precursor Material is eroded.
Inert Gas Electrode Shielding	No	No	Yes
Effect Of Electrode Polarity Reversal	Little To No Effect	Little To No Effect	Non-Consumable Electrode Erosion And Nanopowder Contamination.

The radial gun embodiment of FIG. 3 provides a substantial improvement in cost and efficiency. The high power (megawatt), electric discharge of the radial gun, however, may occur from time to time at other than the center of the electrodes. In this event, the resistance of the arc is decreased, and less energy may be added to the arc discharge

cal confinement of the plasma and to be ablated. In the event the annular ablative bodies 42 and 48 are nonconductors, no annular insulator is required between the ablative bodies and the electrodes.

As illustrated in FIG. 4, the solid anode electrode 41 and the solid cathode electrode 47 have round uniform cross-

sections. The electrodes may be composed of metals including, but not limited to, aluminum, copper, or iron. The annular ablative bodies **42** and **48** are hollow, and may be composed of the same materials as the electrodes, as well as nonconductive materials. The annular insulator bodies **43** and **49** respectively electrically isolate the anode electrode **41** and the cathode electrode **47** from the annular ablative bodies **42** and **48**.

In operation, the pulsed power supply **51** is energized to cause a high-power, pulsed electrical discharge between the cathode electrode **47** and the anode electrode **41**. The discharge arc will attach to the cathode and anode because they are conductors. As the cathode electrode **47** and anode electrode **41** are respectively located in the center of the composite electrodes **46** and **40**, which are characterized by diameters that are larger than those of the cathode and anode, the discharge arc can be said to be physically confined to the center of the composite electrodes to a greater extent than provided by the anode and cathode electrodes of the same diameter in the radial gun embodiment of FIG. 3. The construction of the composite electrodes **40** and **46** thus dramatically increase the probability of the discharge arc attaching to the tip areas of both the anode electrode **41** and the cathode electrode **47**. The added confinement of the electrical discharge increases its energy to melt, vaporize and ionize material ablated from the anode electrode **41**, the cathode electrode **47**, and the two annular ablative bodies **42** and **48**. Further, the composite electrodes **40** and **46** prevent the axial expansion of the plasma, and the inertial confinement afforded by the pulsed electrical discharge deters expansion of the plasma radially outward as indicated by arrows **54** and **55** of FIG. 4. A high temperature, high density plasma thus is created. The ablation of the anode electrode **41**, the cathode electrode **47**, and the annular ablative bodies **42** and **48**, by the high velocity exit of the plasma radially outward, produces nanopowder at an even higher rate per discharge than that of the invention embodiment illustrated in FIG. 3 above. Further, the ablated material is forced out of the radial gun by the plasma at supersonic speed to be quickly quenched by and/or quickly react with the surrounding gas within the reactor vessel to produce nanopowder.

Although the preferred embodiments of the invention have been disclosed in detail, various substitutions, modifications, and alterations can be made without departing from the spirit and scope of the invention as defined in the claims.

What is claimed is:

1. An apparatus for synthesizing nanopowders, which comprises:

a first hollow ablative body composed of a first precursor material and having a first outer diameter, and further having a first axial bore of a second diameter;

an anode electrode of a second precursor material having a third diameter smaller than said second diameter, and fitted within said first axial bore but electrically isolated from said first hollow ablative body;

a second hollow ablative body composed of a third precursor material and having a fourth outer diameter, and further having a second axial bore of a fifth diameter;

a cathode electrode of a fourth precursor material having a sixth diameter and fitted within said second axial bore but electrically isolated from said second hollow ablative body, and wherein said first hollow ablative body and said second hollow ablative body are spaced apart in a gaseous atmosphere; and

a power supply which is in electrical communication with said anode electrode and said cathode electrode, and which effects a high power pulsed electrical discharge to ablate said first hollow ablative body and said second hollow ablative body to produce said nanopowders.

2. The apparatus of claim **1**, wherein said gaseous atmosphere consists of one of an inert gas, a reactive gas, and a mixture of inert and reactive gases.

3. The apparatus of claim **1**, wherein said gaseous atmosphere is a mixture of inert gases.

4. The apparatus of claim **1**, wherein said anode electrode, said cathode electrode, said first hollow ablative body, and said second hollow ablative body are composed of a same material.

5. The apparatus of claim **1**, wherein said first hollow ablative body and said second hollow ablative body are composed of a first material, said anode electrode and said cathode electrode are composed of a second material, and said first material is different from said second material.

6. The apparatus of claim **1**, wherein said first hollow ablative body and said second hollow ablative body are made of nonconducting material, and are without electrical insulation.

7. The apparatus of claim **1**, wherein electrical insulation is provided by a first annular insulator placed between said anode electrode and said first hollow ablative body, and by a second annular insulator placed between said cathode electrode and said second hollow ablative body.

8. The apparatus of claim **1**, wherein said first hollow ablative body and said anode electrode form a first composite electrode, said second hollow ablative body and said cathode electrode form a second composite electrode, and said first composite electrode and said second composite electrode radially center said pulsed electrical discharge and substantially prevent axial expansion of a plasma created from ablation of said precursor material.

9. The apparatus of claim **8**, wherein said pulsed electrical discharge is of a duration short enough to inertially confine said plasma.

10. The apparatus of claim **8**, wherein said plasma is of a high temperature and high density to accommodate more rapid quenching by and more rapid reaction with said gaseous atmosphere for improved nanopowder production rates.

11. The apparatus of claim **10**, wherein said high temperature is of the order of 50,000° K.

12. The apparatus of claim **1**, wherein said nanopowders are comprised substantially of unagglomerated nanopowder.

13. The apparatus of claim **1**, wherein said first hollow ablative body, and said second hollow ablative body, are both one of conductor, semiconductor, and nonconductor.

14. The method of claim **1**, wherein the first outer diameter is the same as the fourth outer diameter.

15. The method of claim **1**, wherein the second diameter is the same as the fifth diameter.

16. The method of claim **1**, wherein the third diameter is the same as the sixth diameter.

17. The method of claim **1**, wherein the first precursor material is the same as the third precursor material.

18. The method of claim **1**, wherein the second precursor material is the same as the fourth precursor material.

19. The method of claim **1**, wherein:
the first outer diameter is the same as the fourth outer diameter;

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the second diameter is the same as the fifth diameter;
the third diameter is the same as the sixth diameter;
the first precursor material is the same as the third
precursor material; and

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the second precursor material is the same as the fourth
precursor material.

* * * * *