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(54) Title: ENERGY GENERATION METHOD AND APPARATUS



(57) Abstract: An energy generation method includes irradiating a first material containing nanoscale sized space therein with microwave or radio frequency radiation having a first energy to generate a second energy greater than the first energy.

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## ENERGY GENERATION METHOD AND APPARATUS

## BACKGROUND OF THE INVENTION

[0001] The present application claims benefit of priority of U.S. application serial number 10/846,045 filed May 14, 2004 (published as US published application 2005/0007001 A1) and U.S. provisional applications serial numbers 60/584,486 and 60/609,276, filed on July 2, 2004 and September 14, 2004, respectively, wherein all three are incorporated herein by reference in their entirety. The present invention is generally directed to an energy generation method and apparatus and more specifically to energy generation from carbon nanotubes and other nanostructured materials irradiated by long wavelength electromagnetic radiation.

[0002] Since the discovery of carbon nanotubes (CNTs) and the advancement of their synthesis techniques interest in these structures has expanded into a wide range of applications and properties. One article reported that nanotubes combusted in air when exposed to the radiation from the flash of an ordinary camera. However, other electromagnetic responses from nanotubes than mere combustion are desirable.

## BRIEF SUMMARY OF THE INVENTION

[0003] One preferred aspect of the present invention provides an energy generation method which includes irradiating a first material containing nanoscale sized space therein with microwave or radio frequency radiation having a first energy to generate a second energy greater than the first energy.

## BRIEF DESCRIPTION OF THE DRAWINGS

[0004] Figure 1 is a photograph of light and plasma emission from a SWNT sample.

[0005] Figure 2 is a photograph of the melted tube connected to a vacuum system after irradiation of a SWNT sample with microwaves.

[0006] Figures 3A and 3B are micrographs of pre and post microwave irradiated nanotubes, respectively, according to specific examples of the present invention.

[0007] Figures 4A, 4B and 4C are Raman spectra of SWNTs according to specific examples of the present invention.

[0008] Figure 5 is a light emission spectra of SWNTs according to specific examples of the present invention.

[0009] Figure 6 is a temperature versus time plot of SWNTs according to specific examples of the present invention.

[0010] Figure 7 is a plot of light emission spectra of 25 mg hydrogen degassed BuckyPearl® SWNTs overlaid on standard optical source spectra corresponding to two temperatures, according to specific examples of the present invention. Figures 8A-8G are schematic illustrations of the microwave source and reflector of the specific examples.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0011] The present inventors have realized that energy may be generated by irradiating a material containing a nanoscale sized space therein with microwave or radio frequency radiation to generate energy from the material. The energy generated from the material preferably has a magnitude that is greater than the energy of the applied microwave or radio frequency radiation (i.e., an energy gain). The generated energy preferably comprises energy that is released in whole or in part from the material and/or from interaction(s) between the radiation and the material. Thus, efficient long wavelength to thermal energy conversion, preferably with an energy gain or release results by irradiating the material with long wavelength radiation, such as radiation having a frequency of between about 1 gigahertz and about 1 terahertz, for example.

**[0012]** The nanoscale sized space in the material preferably comprises one or more filled or unfilled cavities or voids in the material having a smallest dimension of less than 1.2 nanometers. Preferably, the nanoscale sized space is a substantially cylindrical enclosed cavity in the material having a diameter of less than 1.2 nanometers. For example, a substantially cylindrical cavity has a shape that is or resembles a cylinder, but may not necessarily have a straight sidewall and/or may not necessarily have flat upper and lower bases. The cavity or void may be filled with atoms or molecules that are not part of the lattice of the material or the cavity or void may be left unfilled.

**[0013]** The material is preferably a carbon material, but may comprise other non-carbon materials as well. Preferably, the material comprises carbon nanotubes and the nanoscale sized space comprises the internal space that is surrounded by the nanotube wall or walls. Preferably, the carbon nanotubes have an internal diameter of 1.2 nanometers or smaller, more preferably 1.1 nanometers or smaller. The nanotubes may comprise single walled carbon nanotubes or multi-walled carbon nanotubes, such as two or more walled carbon nanotubes, having an innermost diameter of 1.1 nanometers or smaller. Alternatively, the nanotubes may have an internal diameter larger than 1.1 nm. For example, multi-walled nanotubes may have an internal diameter of greater than 1.1 nm, but a space between the walls of 1.1 nm or less. The internal space in the nanotubes is substantially cylindrical because it has curved rather than flat bases or ends. This internal space may be filled with hydrogen, oxygen, deuterium, tritium, lithium, or other atoms or molecules as will be described in more detail below.

**[0014]** It should be noted that non-carbon nanostructured materials, such as metal oxide hollow nanohorns or hollow nanowires having an internal diameter of 1.1 nm or less may also be used. Likewise, carbon and non-carbon bulk material with nanoscale sized space therein may also be used. Furthermore, it is possible that carbon tips in the carbon nanotube act as pin point electron field emitters which contribute to the energy generation and conversion effect. Thus, any suitable material

that contains carbon or other suitable tips which act as pin point electron field emitters may also be used.

**[0015]** Preferably, the carbon nanotubes comprise purified, single walled carbon nanotubes. A higher or greater purity nanotubes are preferred. The nanotubes may be purified by any suitable purification method. Without wishing to be bound by a particular theory, it is believed that purification removes amorphous catalyst and unprocessed carbon precursor material from exterior of the nanotubes. It is believed that the purification increases the amount of pure nanotubes per unit volume, which increases the energy gain. Without wishing to be bound by a particular theory, it is believed that the energy gain is caused by a nanostructured material, such as carbon nanotubes. Thus, an increase in the amount of pure nanostructure material per unit volume increases the energy gain. Furthermore, preferably, the nanotubes comprise highly dense carbon nanotubes. An example of highly dense nanotubes are BuckyPearl® nanotubes available from Carbon Nanotechnologies, Inc. (CNI) of Texas. Typical carbon nanotubes have a density of about 15 mg/m<sup>3</sup>, while BuckyPearl® nanotubes have a density of about 600 mg/m<sup>3</sup>. Thus, a density of above 100 mg/m<sup>3</sup>, such as 100 mg/m<sup>3</sup> to 600 mg/m<sup>3</sup> is preferred. Without wishing to be bound by a particular theory, it is believed that an increase in the amount of pure nanostructure material per unit volume (i.e., density) increases the energy gain.

**[0016]** The material may be irradiated with any suitable long wavelength radiation that produces an energy gain. For example, the frequency of the long wavelength radiation may range from about 1 GHz to about 1 terahertz, including from 1 to 90 GHz, preferably from 2.4 to 12 GHz. Depending on the definition of the exact location of the imaginary boundary between radio frequency and microwave bands, the lower end of the 1 to 90 GHz range is either in or borders the radio frequency range, while the middle portion and upper end of this range are in the microwave range. Preferably, microwave radiation is used. Any suitable microwave radiation power may be used, such as a power of between 30 watts and 100 kW, preferably between 30 watts and 1 kW.

[0017] Preferably, the material, such as carbon nanotubes, is irradiated with long wavelength radiation, such as with pulsed microwave radiation to provide an electric field of at least about 10,000 V/cm in the material. The provided electric field in the material is preferably 10,000 V/cm to 1.2 million V/cm, such as between 15,000 and 100,000 V/cm. A sufficient electric field is provided in the carbon nanotubes for a sufficient time to generate at least one of the energy gain and a plasma. The energy gain and/or plasma may be generated almost instantaneously, such as in a fraction of a second after the application of the electric field or after a longer electric field application, such as an electric field application of at least 1 to 2 seconds, for example of at least 1 to 20 seconds.

[0018] For example, the electric field of at least about 10,000 V/cm may be provided in the carbon nanotubes for a sufficient time to generate the energy gain by several different methods. The carbon nanotubes tend to move around upon the irradiation with microwave radiation and thus may move out of the zone where sufficient microwave radiation exists to produce the sufficient electric field for the energy gain. Thus, the sufficient electric field is provided in the carbon nanotubes for a sufficient time to generate the energy gain either by restraining the carbon nanotubes from moving during the irradiation with the microwaves pulses and/or by configuring the incident microwave radiation such that it covers a sufficient area in which the radiation is able to generate a sufficient electric field in the nanotubes for the energy gain. The carbon nanotubes may be restrained by being placed in a container between microwave transparent packing material. The packing material keeps the nanotubes in place during the irradiation. The packing material and radiation configurations will be described in more detail below.

[0019] The material, such as carbon nanotubes, is located in a non-oxidizing ambient while it is irradiated microwave pulses. For example, the material may be located in a high or low vacuum or in an inert ambient. An inert ambient includes a nitrogen or an inert gas, such as argon or helium.

[0020] The material, such as carbon nanotubes, emits thermal energy (i.e., heat) as well as at least one, and preferably all three, of visible, infrared and

ultraviolet radiation when it is irradiated with long wavelength radiation. The details of the emitted thermal and radiation energy will be described in more detail below. Preferably, the magnitude of the thermal energy emitted by the material is greater than the magnitude of the energy of the microwave radiation. Preferably, the magnitude of the thermal energy emitted by the material is at least 2 to 1,000 times, such as 10 to 100 times greater than the magnitude of the energy of the microwave radiation. For example, the magnitude of the thermal energy emitted by the material may be at least 10 times greater than the magnitude of the energy of the microwave radiation.

[0021] Preferably, irradiating the material, such as carbon nanotubes, with microwave radiation also generates a plasma about the nanotubes. The plasma includes ions of elements that are found in and/or on nanotubes, such as carbon ions as well as impurity ions that may be present in the nanotubes, such as oxygen, hydrogen, iron, nitrogen and/or silicon ions.

[0022] Any suitable microwave emitting device may be used. For example, a magnetron, a klystron or a backward wave oscillator microwave emitting device may be used. If a magnetron microwave source is used, then a microwave emitter, such as an antenna, of the magnetron is positioned as close as possible to the carbon nanotubes. For example, the emitter is preferably positioned 4 mm or less from the nanotubes in a near field configuration to deliver an electric field of at least 10,000 V/cm in the nanotubes.

[0023] *PROCESS PARAMETERS.* Without wishing to be bound by a particular theory, the present inventors unexpectedly found that by controlling some or all of the process parameters described above, they were able to obtain an energy gain or release from the material being irradiated with long wavelength radiation. These parameters include the electric field component of microwave radiation, size of the nanoscale size space in the material (such as the inner diameter of carbon nanotubes) and absence of oxygen. Other parameters include the density of carbon nanotubes, the purity of carbon nanotubes, the generation of the plasma and physical stability of the sample in the microwaves.



**[0024]** If some of these process parameters are not provided, then less thermal energy may be released from the material than microwave energy has been put into the material. In other words a certain number of joules of microwave energy will be supplied and a lower number of joules of thermal energy will be released. However, some measure of thermal energy may still be obtained from the material and this may still be a very efficient method of converting microwave energy to thermal energy.

**[0025]** *AMBIENT.* The first process parameter that should be controlled for energy gain is the ambient. For carbon nanotube material, the absence of oxygen contributes to the energy gain. If oxygen is present, then purified and unpurified nanotubes will rapidly oxidize or burn (i.e., will be destroyed). For example, unpurified nanotubes exposed to microwave fields in air will result in two substances, one of which will be orange in appearance, and the other will be black. It is believed that the orange substance will be hematite, or iron oxide, such as from the catalyst used to prepare the nanotubes, and that the black substance will be highly purified nanotubes, which will lack the extra carbon material and iron catalyst. However, if the nanotubes are maintained in a non-oxidizing ambient, such as in a vacuum, in an inert ambient, such as argon, helium or nitrogen, or in a reducing ambient, such as in hydrogen or forming gas, then the oxidation and burning of the nanotubes can be avoided. The nanotubes remain intact and release thermal energy in excess of the microwave energy. An ultra high, high or even a low vacuum (such as in vacuum of levels  $10^{-3}$  torr and lower) ambient may be used to generate an energy gain.

**[0026]** *ELECTRIC FIELD.* The second process parameter is that the electric field in the material being irradiated with the microwaves should be above a certain threshold value. The threshold value may vary for different materials and may also vary based on the other process parameters. A preferred threshold value of the electric field is about 10,000 V/cm. However, the threshold value may be higher for some process parameter combinations, such as between 11,000 and 15,000 V/cm, for example, or lower for other parameters, such as between 700 and 9,000 V/cm. For example, for irradiation with high power microwaves, such as microwaves having a power of 2 kW, it is believed that an electric field of only 667 V/cm may produce an

energy gain of about one (i.e., the input energy is about the same as the output energy). Thus, for 2 kW microwave power, an electric field of greater than 700 V/cm is expected to generate an energy gain of greater than one. In general, for high microwave power of several thousand Watts, such as 2 kW or greater, an electric field of several hundred to a few thousand V/cm may generate an energy gain of greater than one. In contrast, for low microwave power, such as 1 kW or lower microwave power, an electric field of several thousand V/cm, such as at least 10,000 V/cm may generate an energy gain of greater than one.

**[0027]** The electric field of the microwaves should be higher than that in a home microwave oven due to a typical difference in source to sample distance. In a home microwave oven, the object exposed to the microwave field is in a far field configuration. This is an area of the field in which the electric and magnetic components of the field are completely coupled and the field is fully formed. For magnetron type microwave source, it is preferred that the material being irradiated with microwaves be in a near field with respect to the microwave emitter, such as when the material is located within 4 mm from the emitter. In the near field, a large electric component of the field is present due to the field or wave not being completely formed. Furthermore, the electric and magnetic components are essentially independent entities due to the lack of a poynting vector in the near field. In the case of the near field, the electric field can reach 10,000 Volts per centimeter and above.

**[0028]** The high electric field can also be obtained by using various resonant devices such as a waveguide or resonant cavity rather than using a near field configuration. The resonant devices take in a microwave from a suitable microwave source, such as a microwave magnetron, klystron, backward wave oscillator or some other device. The resonant devices then separate the microwave into its component electric and magnetic parts and either cause that wave to travel down the waveguide or be in some resonant condition in a cavity device. In such resonant devices, the electric and magnetic components will be at a maximum and in theory can become very high. In practice, it is believed that the highest observed electric field to date is

about 1.2 million Volts per centimeter. The resonant devices can be used to expose the carbon nanotubes to microwaves having very high electric field values and thereby causing an increase in the energy gain.

[0029] *PLASMA*. It is believed that when carbon nanotubes are exposed to microwaves having a high electric field value in a non-oxidizing ambient, then a bright plasma will ignite. The specific examples illustrate that the thermal energy release is more efficient with the presence of the plasma. In contrast, if no plasma is formed, then thermal energy release may still be seen but its efficiency will be much lower and the excess energy gain or release may be absent. When the electric field is below 5000 V/cm, it is believed that no plasma will be formed.

[0030] The plasma's spectra exhibits plasma lines from any element or impurity in the nanotube sample. For instance, most nanotube samples contain carbon, hydrogen, nitrogen, iron, etc. Thus, ionized states of each of these elements will be observed, with the plasma lines being more ionized with a decrease in the diameter nanotubes or an increase in the electric field. Without wishing to be bound by a particular theory, it is believed that these two factors lead to a higher surface charge density on the nanotubes (i.e., due to the smaller surface area of narrower nanotubes) which will in turn lead to a higher ionized plasma state. The ionized state of the plasma may be controlled by controlling the nanotube dimensions and purity. For example, a higher ionized state may be obtained by decreasing the nanotube diameter and/or length. Furthermore, the particular ion species in the plasma may be provided by providing or doping the species into or onto the nanotubes.

[0031] Furthermore, without wishing to be bound by a specific theory, it is believed that an electron plasma may also be formed. It is believed that the ion and/or the electron plasma does not necessarily have the same pulse frequency as the pulse frequency of the pulsed microwave source. Thus, a continuous plasma or a plasma that has a different frequency than that of the microwave source may be generated. Thus, the nanotubes may exhibit a capacitive effect with regards to plasma generation because the microwave pulse frequency may be decoupled from the plasma frequency. Without wishing to be bound by a specific theory, it is believed that the

nanotubes act as an initial energy pathway between the microwave energy and the plasma. It is possible that the ion plasma may be maintained in the presence of the microwaves even after the nanotubes are removed.

**[0032]**        *NANOTUBE DIAMETER.* It is believed that at least for SWNTs and possibly for MWNTs, the nanotube diameter is inversely proportional to the energy gain (i.e., excess thermal energy release). Thus, as the inner diameter of the single walled (and possibly the multi-walled nanotubes) is decreased, the thermal output is increased. Preferably, the inner diameter of the nanotubes, such as single walled nanotubes is 1.1 nm or less, such as 0.7 to 1.1 nm. Slightly larger diameter nanotubes may also be used if the other process conditions are optimized. However, for multi-walled nanotubes, the inner diameter may be larger than 1.1 nm, such as 1.1 nm to 5 nm for example. However, multi-walled nanotubes contain spaces between the nanotube walls which have a width of less than 1.1 nm. When the nanotubes are housed in a glass container, for a decreasing nanotube inner SWNT diameter, a larger amount of glass melts for a predetermined amount of microwave irradiation time or the same amount of glass melts but in a much shorter time.

**[0033]**        *NANOTUBE DENSITY.* The energy gain also increases with increasing nanotube density and purity. For example, BuckyPearl® brand HiPCo type of nanotubes are about 40 times denser than conventional single wall nanotubes. The use of this brand of nanotubes results in very intense plasmas consisting of more highly ionized states than plasmas generated with the less dense nanotubes of a similar diameter. This increase in density also causes a more energetic reaction to be seen (i.e. more glass will melt in a smaller period of time). Without wishing to be bound by any particular theory, the present inventors believe that the increase in thermal energy gain or release with increasing nanotube density may be due to an exchange of phonons from one nanotube to another in a more efficient manner and/or due to a more efficient interaction of overlapping EMF generated by the nanotubes to conduct a current that they pick up from the microwave field.

**[0034]**        *NANOTUBE PURITY.* Furthermore, a higher energy gain results from increasing nanotube purity. Without wishing to be bound by any particular theory,

the present inventors believe that the increase may be due to decreased amount of amorphous carbon which does not provide an energy gain in the purified samples.

[0035] *SAMPLE STABILITY.* The physical stability of the nanotubes is important to the energy gain to the extent that the nanotubes are maintained in the sufficient electric field of the microwaves for a sufficient amount of time. The stability may be achieved by any suitable method that keeps the nanotubes from flying around inside their container during microwave irradiation. If the nanotubes are not kept still, upon application of the microwave field, the nanotubes will fly out of the field. If the microwaves are applied to the nanotube sample in such a way as to maintain the high electric field in moving nanotubes, then the nanotubes do not have to be fixed or kept still. Therefore either fixing the nanotubes in one location or spreading the high electric field over the area where the nanotubes can become mobile can be used to generate the energy gain.

[0036] *Specific Examples*

[0037] The present inventors conducted a number of independent experiments, in which milligram quantities of nanotubes were exposed to continuous microwave fluxes for several seconds of irradiation and produced a blinding light emission, comparable in intensity to a welding arc. Many of these experiments were repeat runs which produced very similar results. Some experiments, as detailed below, produced an energy gain of at least a factor of ten in the form of thermal energy emission from the SWNTs.

[0038] The SWNTs are observed to retain their overall structural integrity after irradiation and thus are not consumed by chemical reactions. However, after a certain duration of irradiation, a sufficient percent of the nanotubes in the sample experienced diameter doubling and/or a chirality change which led to termination of the energy gain and/or of the energy transfer (though not necessarily at the same time) when a majority of the nanotubes experienced this change. Furthermore, in some experiments the tube containing the nanotubes was broken which lead to oxidation of the nanotubes and termination of the energy gain and light emission.

[0039] A variety of nanotubes of different purification levels and diameters were used from different sources: 1.5 nm average diameter, purified SWNTs (laser oven purified SWNTs from Carbolex Inc., Lexington, KY and other custom made SWNTs); 1.5 and 1.1 nm average diameter, raw and purified SWNTs made by the HiPCo process (CNI, Houston, TX); 2-20 nm diameter x 5-20  $\mu$ m length multiwall nanotubes (MWNTs from Sigma Aldrich); and 0.9 nm average inner diameter double walled MWNTs from Rossetter Holdings. The energy gain was only observed for the dense BuckyPearl® 1.1 nm HiPCo SWNT samples and the Rossetter Holdings MWNT samples. A small quantity of 0.7 and 1.3 nm SWNTs from NEC were also used in a very limited number of experiments. However, the amount of NEC SWNTs that were obtained were not sufficient to conduct a sufficient number of repeatable experiments.

[0040] The microwaves were generated using a number of magnetrons, such as Goldstar® model 2M223 magnetrons operating at a frequency of 2.45 GHz (12.2 cm wavelength) radiating into free space. Different power magnetrons were used, such as about 300 watt, about 450 watts and about 1000 watts. The typical output power measured for a new 450 watt magnetron was about 420 to 450 watts. Some magnetrons were subsequently internally modified by inducing a slight shorting condition in the tube such that the output power was about 50 watts.

[0041] The maximum microwave power output of each modified magnetron in the absence of SWNT samples was calibrated by thermal calorimetry using the heating of a water medium and/or current-voltage (I-V) monitoring of the magnetron unit. Both methods give agreement within the errors of the measurements (typically  $\pm 2-3\%$ ). In addition, cross checks on the maximum radiation power possible were done by monitoring the (I-V) characteristics of the line voltage during operation.

[0042] In each experiment, a weighed amount of a SWNT sample ranging from 5 to 500 mg was placed in a laboratory tube. Most runs used about 25 mg of nanotubes. The tubes in most experiments were closed end, clear tubes of about 4 mm inner diameter, about 6 mm outer diameter and having a length of about 15 cm. The tubes were believed to be made of quartz, but could have been made of a similar

clear glass type substance, such as Pyrex. In most experiments, the tubes were sequentially connected to a stainless steel vacuum system through a glass to metal seal. A valve was situated between the sample and the main chamber, which was pumped to a pressure lower than  $10^{-8}$  Torr with the sample valve open. Pressures as high as about  $10^{-3}$  Torr, however, did not result in significant differences in the experimental results.

**[0043]** In a large number of the experimental runs, the nanotubes were packed in a thermal absorbing packing material to fix the nanotubes to a predetermined location in the tubes because the nanotubes without the packing material tend to fly around the container. Specifically, crushed  $\text{SiO}_2$  was placed in the bottom of the tubes, the nanotubes were placed on top of the crushed  $\text{SiO}_2$  and then additional crushed  $\text{SiO}_2$  was placed on top of the nanotubes. 1 to 50 grams of crushed  $\text{SiO}_2$  was placed below and above the nanotubes. In most runs, 1 gram of  $\text{SiO}_2$  was used with 4 mm diameter tubes. Larger amounts of  $\text{SiO}_2$  were used for runs conducted in larger diameter tubes. However, the increase in tube diameter increases the likelihood that the nanotubes will move around the tube during irradiation. For a 30 gram  $\text{SiO}_2$  experimental run, the  $\text{SiO}_2$  extends about 1 cm above and below the nanotubes in the tube. Crushed quartz is preferred to silica powder as the crushed  $\text{SiO}_2$  since silica powder tends to be thrown around the vacuum system. Other microwave transparent materials, such as alumina, can also be used as a packing material. A ceramic spacer was placed on the bottom of the tubes for two experiments, but was found not to provide any appreciable benefit and was not used in the remainder of the experiments.

**[0044]** The sample tubes were sequentially placed in the near-field region at a distance of 1-4 mm from the front surface of the magnetron (i.e., from the emitter of the magnetron). The long edge of the tubes was placed running at about a 5 to 15 degree angle off parallel to the front face of the magnetron, with the edge of the tube containing the nanotubes located 1-4 mm from the front face of the magnetron. The angular positioning of the tubes is believed to be one way to increase the electric field generated in the nanotubes. A rectangular reflector with a cone angle of about 105 degrees was used in a number of runs to increase the electric field generated in the

nanotubes. Figure 8A schematically illustrates a side cross sectional view of the reflector 10 attached to the front, flat surface of the magnetron 12 containing the broadcast antennae 14. Figure 8A also shows the electrical wires 16 connected to the magnetron 12. The reflector 10 comprises an assembly of six pieces A to F, shown in Figure 8B. The top piece A is shown in Figure 8C, left piece B is shown in Figure 8D, right piece C is shown in Figure 8E, front and back pieces D and E, respectively, are shown in Figure 8F, and bottom piece F is shown in Figure 8G. The dimensions of each piece A-F are shown in Figures 8C to 8G.

[0045] Light emission from the nanotubes was detected using an optical fiber with a focusing lens placed about 5 cm from the sample. The fiber was connected to an optical spectrometer (Ocean Optics model USB 2000) operating over the range of 180-880 nm at 0.28 nm resolution with a minimum 10 ms full spectrum acquisition time.

[0046] In every example which used SWNTs with average diameter of 1.1 nm or smaller, regardless of the synthesis method, source of material or purity level, a brilliant light, similar to a welding arc, was emitted from the sample upon the start of microwave irradiation, continued for 3-5 s, in some cases up to 15-20 s, and typically ended abruptly (usually due to the melting of the glass or quartz tube), as shown in Figure 1. It should be noted that the quality of the tubes varies. Thus, there was a difference in how long it took a particular tube to melt (i.e., 5 seconds or less to 15 seconds or more depending on the quality of the tube). Examination immediately after the termination of the emission in experiments with an energy gain and in some experiments without an energy gain showed that local melting of the tube holding the sample had occurred in the vicinity of the SWNT sample, as shown in Figure 2. Thus, the melting of the tube and the exposure of the nanotubes to air could have been responsible for the termination of the light emission. Furthermore, the microwave irradiation causes any adsorbed or absorbed species on the microwaves, such as hydrogen atoms, to be desorbed or desorped from the nanotubes.

[0047] The brightness of the light emission was greatest for the smallest diameter, the highest purity and the most dense forms of the nanotubes, all other



variables being constant for each experiment. In particular, for SWNT samples with average diameters of 1.5 nm, the light emission was very dim and produced only minor warming of the tube. MWNT samples with large inner diameter nanotubes similarly showed only a minor temperature rise but in this case the only light emission was a barely detectable output in the near IR region. Control experiments with purified graphite powder, standard carbon particles and inert dielectric substances such as powdered SiO<sub>2</sub> showed no effects whatsoever, with even minutes of irradiation, resulting in only the expected slight warming of the sample.

**[0048]** Visual examination of post-irradiation samples heated in vacuum typically showed the samples remained as a black material, even for runs with extremely bright light emission and heating. In the case of 1.1 nm SWNTs, for which intense heating and light emission had occurred, removal of the samples, which required breaking the surrounding melted quartz, and reloading into a vacuum system followed by microwave irradiation resulted in some light emission activity, though reduced in intensity from the original irradiation event.

**[0049]** Several 1.1 nm SWNT samples were characterized by scanning and transmission electron microscopy (SEM and TEM) and by Raman spectroscopy after being irradiated with microwaves. The results of the microscopy examination showed that after microwave exposure, these samples still consisted of nanotubes, although with some subtle variations in the physical structures. Figures 3A and B show SEM images of a 1.1 nm average diameter SWNT sample before (Figure 3A) and after (Figure 3B) 6 second irradiation of 2.45 GHz microwaves from a magnetron source having the above described reflector and operating with a 50 W total output power. The samples were about 25 mg BuckyPearl® HiPCo SWNT samples packed with about 1 gram of crushed quartz. The microwave emitter was positioned about 1 to 4 mm from the nanotubes at an angle of about 5 to 15 degrees. The image clearly shows the presence of nanotube ropes after the irradiation process. During this irradiation, brilliant light emission and intense heating were observed. The length scale shown is the same for both images. The nanotubes tend to fuse or weld to adjacent nanotubes and to form looped structures after being irradiated with

microwaves. Furthermore, the nanotubes tend to expand from their original volume during the microwave irradiation and then contract to a volume that is about the same or greater than the original volume after the irradiation is completed. Still further, in a sample of mixed metallic and semiconducting SWNTs, a chirality shift is observed. The metallic SWNTs in the sample are restructured into mostly semiconducting SWNTs between about 4 and 7 seconds of irradiation (peaking at about 6 seconds of microwave irradiation) and then revert back to mostly metallic SWNTs after more than 7 seconds, such as 20 seconds of microwave irradiation. Without wishing to be bound by a particular theory, it is believed that the chirality shift may occur due to a partially completed coalescence of the nanotubes which leads to diameter increase, such as diameter doubling, of the nanotubes during irradiation.

**[0050]** Figures 4A and B show the typical Raman spectra (514.5 nm excitation) of single wall nanotubes before (Figure 4A) and after (Figure 4B) 6 second microwave irradiation. Both spectra show a main, intense asymmetric peak at about  $1592\text{ cm}^{-1}$  (presumed to be  $E_{15}$ ,  $E_{2g}$ ,  $A_{1g}$ , C-C stretching modes) and a weak peak at about  $1340\text{ cm}^{-1}$  (presumed to be a disorder or defect peak), which are uniquely distinctive for the nanotube form of carbon. Figure 4B also shows sharp features in the  $140\text{-}200\text{ cm}^{-1}$  region (presumed to be  $A_{1g}$  breathing modes), which are indicative of a transformation of diameters of nanotubes to larger diameters. Figure 4C shows the  $1250\text{ to }1850\text{ cm}^{-1}$  Raman spectra for SWNTs irradiated for 0, 6 and 20 seconds. The nanotube peak for the sample irradiated for 20 seconds is in about the same position as the peak for the sample that was not irradiated. However, the nanotube peak for the sample irradiated for 6 seconds shifted to a higher wavenumber. The height of the defect peak also increased with increasing irradiation time. For samples that have been irradiated for minutes, the diameters of the nanotubes double and then double again.

**[0051]** An example of the microwave irradiated SWNT light emission spectra is shown in Figure 5. This Figure shows a time averaged spectrum of a 1.1 nm average diameter, purified SWNT sample exposed to a 2.45 GHz magnetron source measured to have a  $50 (\pm 3)$  W total output power with no sample. The sample was an

about 25 mg BuckyPearl® HiPCo SWNT sample packed with about 1 gram of crushed quartz. The microwave emitter was positioned about 1 to 4 mm from the nanotubes at an angle of about 5 to 15 degrees. The spectrum integration was done over a 100 ms acquisition starting after 2 seconds of magnetron on time to ensure the attainment of a pulsed microwave flux. The sharp peaks are assigned to diagnostic optical transitions for elements, such as C, H and Fe, present in the nanotube sample, as indicated for C and H. As light emission progresses, the H line intensity consistently decreases rapidly, indicating outgassing of hydrogen impurity, which is confirmed by parallel quadruple mass spectrometer measurements. Despite extensive examination, no oxygen spectra were found, consistent with the absence of combustion. The inset shows the envelope of the broad spectral feature stretching from about 400 to 700 nm. The estimated peak of this curve is at about 480 nm, as marked by the arrow.

**[0052]** The presence of line spectra indicates the presence of a charged plasma within the region of the nanotube sample. The broad intensity envelope under the line features is reminiscent of a blackbody curve and shows a maximum in the about 500 nm region. During the course of any of the above described experiments, the peak typically shifted. For example, in Figure 5, the peak shifted from about 535 to 477 nm and the peak shown in the inset appears at about 480 nm. This is typical of non-equilibrium systems such as this.

**[0053]** Figure 6 shows the time evolution of average system temperatures over a parallel run as calculated from the envelope maximum frequency with Wien's law of black body radiation. Specifically, Figure 6 shows an estimated blackbody temperature versus total irradiation time for a parallel experiment to the one shown in Figure 5. The temperatures were calculated from the spectral data by assigning all the atomic transitions, subtracting curve-fitted line spectra for each of these transitions from the overall curve to leave a broad, nearly featureless envelope, assigning the maximum of the resulting broad envelope, and applying Wien's law,  $T = 0.0029/\lambda_{\max}$ . The correlation between the peak maxima and temperatures were verified by calibration using a NIST traceable light source as shown in Figure 7. The longest

time point shown occurred just prior to the cessation of the light emission for this sample. The estimated total errors in the temperature values are about +/- 5%.

[0054] The average temperature in Figure 6 stays between about 5400 to about 6000 K. In view of the theoretically estimated  $4 \times 10^3$  K disintegration temperature threshold for carbon nanotubes, the ability of the nanotubes to maintain significantly higher electron plasma temperatures without disintegration suggests extensive decoupling of the phonon and electron plasma excitation manifolds. The presence of gamma and X-ray emission was checked using calibrated NaI detectors. No emission was observed above normal background radiation in the 4 - 70 keV and 70 KeV- 8 MeV regions, with an upper limit of  $\sim 2 \times 10^5$  counts set for gamma rays at 2 MeV.

[0055] *ENERGY GAIN CALCULATIONS.* Quantitative lower limit measurements of the thermal heat output were done in a series of experiments with crushed quartz surrounding the SWNT samples. In each experiment, in addition to typical bright light emission and accompanying local melting of the containment tube, the added SiO<sub>2</sub> was visually observed to have fully melted.

[0056] A typical thermal balance is illustrated in an experiment in which about 1 g of crushed quartz surrounding the 1.1 nm diameter BuckyPearl® SWNTs was exposed to an about 3 second microwave flux from a magnetron with a calibrated total output power of 50 ( $\pm 3$ ). The microwave source was positioned about 1 mm away from the tube containing the SWNTs. Post-irradiation examination clearly showed the added SiO<sub>2</sub> had fully fused, along with some local melting of the containment tube. The melted portion of the tube was cut away and weighted. The weight of this portion of the tube was about 0.3 grams. Thus, a total of 1.3 grams (0.022 mol) of SiO<sub>2</sub> was melted (1 gram from the crushed quartz and 0.3 grams from the tube). 10 repeat runs all gave substantially identical results. Given the melting point for SiO<sub>2</sub> of about 2000 K, the energy required to bring the SiO<sub>2</sub> from ambient temperature to just below the melting point (about 1900 K) at constant pressure is given by the standard enthalpy difference  $H^\circ(1900) - H^\circ(298) = 111.5$  kJ/mol. Adding the fusion enthalpy of 9.6 kJ/mol gives a minimum of about 2.6 kJ required to melt

the 0.022 mol of SiO<sub>2</sub>. Heating to higher temperatures would only increase the enthalpy demand. The maximum amount of energy that possibly could be delivered from the microwave source to the sample would be  $P_{\text{total}} * t$ , where  $P_{\text{total}}$  = the calibrated total magnetron power output, which is about 50 J/s in this case, and  $t$  = total irradiation time. Setting  $t = 5$  seconds (conservatively including the 2 second warm up time before maximum flux), a conservative estimate of  $P_{\text{total}}$  is about 0.25 kJ. Thus, the ratio of the minimum energy required to heat and melt the SiO<sub>2</sub> to the maximum possible microwave energy delivered to the sample represents a very conservative energy gain factor of about  $(2.6 \text{ kJ}) / (0.25 \text{ kJ}) = 10.4$ . A cross check on the heat content of the glowing tube was estimated calorimetrically by accurately measuring the temperature change after plunging the vessel into a weighed quantity of water (thermal balance connected for evaporative losses) gave a value of 2.1 kJ, about 80% of above number.

**[0057]** The tables below illustrates the results of various experiments in which 50, 300 or 1000 watt microwave sources irradiated a sample having about 25 mg SWNTs packed in 1 gram of crushed quartz, and located in vacuum, with 1-4 mm emitter to nanotube distance and 5 to 15 degree emitter angle used with the reflector described above. It is believed that an electric field of at least 10,000 V/cm was generated in the nanotubes by the microwave irradiation. The SWNT samples were either 1.5 nm diameter custom made SWNTs or 1.1 nm diameter BuckyPearl® SWNTs. The irradiation duration was either 5 or 15 seconds. The thermal energy release for the experimental runs with the 1.1 nm samples was estimated using the above described method. The thermal energy release for the experimental runs with the 1.5 nm samples was estimated using a water dunk calorimetry test.

**[0058]** Table 1 shows the results for 50 W microwave irradiation for 5 seconds for custom made 1.5 nm diameter SWNTs (comparative examples 1-5) and 1.1 nm BuckyPearl® SWNTs (examples 1-5). It should be noted that the term “comparative examples” as used herein does not mean “prior art examples” and should not be considered to be an admission that the subject matter of the comparative examples is found in the prior art. Instead, comparative examples are examples in which no

energy gain was observed. However, the subject matter of the comparative examples may still be part of certain embodiments of the present invention.

Table 1

EXAMPLE NUMBER	AMOUNT OF MICROWAVE ENERGY IN	AMOUNT OF THERMAL ENERGY RELEASE
<b>1.5 nm nanotubes</b>		
C1	250 Joules	220 Joules
C2	250 Joules	210 Joules
C3	250 Joules	250 Joules
C4	250 Joules	140 Joules
C5	250 Joules	200 Joules
<b>1.1 nm nanotubes</b>		
1	250 Joules	2610 Joules
2	250 Joules	2700 Joules
3	250 Joules	2630 Joules
4	250 Joules	2540 Joules
5	250 Joules	2580 Joules

[0059] Table 2 shows the results for 300 W microwave irradiation for 5 seconds for custom made 1.5 nm diameter SWNTs (comparative examples 6-10) and 1.1 nm BuckyPearl® SWNTs (examples 6-10).

Table 2

EXAMPLE NUMBER	AMOUNT OF MICROWAVE ENERGY IN	AMOUNT OF THERMAL ENERGY RELEASE
<b>1.5 nm nanotubes</b>		
C6	1500 Joules	1110 Joules
C7	1500 Joules	1220 Joules
C8	1500 Joules	1210 Joules
C9	1500 Joules	1260 Joules
C10	1500 Joules	1310 Joules
<b>1.1 nm nanotubes</b>		

6	1500 Joules	6010 Joules
7	1500 Joules	6040 Joules
8	1500 Joules	5910 Joules
9	1500 Joules	6080 Joules
10	1500 Joules	6020 Joules

[0060] Table 3 shows the results for 1000 W microwave irradiation for 5 seconds for custom made 1.5 nm diameter SWNTs (comparative examples 11-15) and 1.1 nm BuckyPearl® SWNTs (examples 11-15).

Table 3

EXAMPLE NUMBER	AMOUNT OF MICROWAVE ENERGY IN	AMOUNT OF THERMAL ENERGY RELEASE
<b>1.5 nm nanotubes</b>		
C11	5000 Joules	1270 Joules
C12	5000 Joules	1160 Joules
C13	5000 Joules	1220 Joules
C14	5000 Joules	1200 Joules
C15	5000 Joules	1220 Joules
<b>1.1 nm nanotubes</b>		
11	5000 Joules	5980 Joules
12	5000 Joules	6030 Joules
13	5000 Joules	6010 Joules
14	5000 Joules	6040 Joules
15	5000 Joules	5990 Joules

[0061] Table 4 shows the results for 50 W microwave irradiation for 15 seconds for custom made 1.5 nm diameter SWNTs (comparative examples 16-20) and 1.1 nm BuckyPearl® SWNTs (examples 16-20).

Table 4

EXAMPLE	AMOUNT OF MICROWAVE	AMOUNT OF THERMAL
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NUMBER	ENERGY IN	ENERGY RELEASE
<b>1.5 nm nanotubes</b>		
C16	750 Joules	470 Joules
C17	750 Joules	500 Joules
C18	750 Joules	510 Joules
C19	750 Joules	510 Joules
C20	750 Joules	490 Joules
<b>1.1 nm nanotubes</b>		
16	750 Joules	6900 Joules
17	750 Joules	6920 Joules
18	750 Joules	7080 Joules
19	750 Joules	7140 Joules
20	750 Joules	7010 Joules

[0062] Table 5 shows the results for 300 W microwave irradiation for 15 seconds for custom made 1.5 nm diameter SWNTs (comparative examples 21-25) and 1.1 nm BuckyPearl® SWNTs (examples 21-25).

Table 5

EXAMPLE NUMBER	AMOUNT OF MICROWAVE ENERGY IN	AMOUNT OF THERMAL ENERGY RELEASE
<b>1.5 nm nanotubes</b>		
C21	4500 Joules	2790 Joules
C22	4500 Joules	2830 Joules
C23	4500 Joules	2750 Joules
C24	4500 Joules	2840 Joules
C25	4500 Joules	2810 Joules
<b>1.1 nm nanotubes</b>		
21	4500 Joules	8470 Joules
22	4500 Joules	8590 Joules
23	4500 Joules	8410 Joules
24	4500 Joules	8440 Joules





[0065] Table 7 summarizes the results of comparative examples 1-30 for 1.5 nm nanotube samples, while 8 summarizes the results of examples 1-30 for 1.1 nm nanotube samples from tables 1-6.

Table 7

Microwave power (in Watts)	Duration of exposure (in seconds)	Energy of microwave radiation (in Joules)	Thermal energy released from nanotubes (in Joules,+/-100J)
50	5	250	200
300	5	1500	1200
1000	5	5000	1200
50	15	750	500
300	15	4500	2800
1000	15	15000	2700

Table 8

Microwave power (in Watts)	Duration of exposure (in seconds)	Energy of microwave radiation (in Joules)	Thermal energy released from nanotubes (in Joules,+/-100J)
50	5	250	2600
300	5	1500	6000
1000	5	5000	6000
50	15	750	7000
300	15	4500	8500
1000	15	15000	9200

[0066] It should be noted that from Table 8, it can be inferred that there may be an upper limit to the microwave energy as which an energy gain is observed. For example, the thermal energy output by the nanotubes for the 300 watt and 1000 watt microwave irradiation is very similar. For the 1000 watt irradiation for 15 seconds,

the amount of energy put in was actually less than the amount of energy released. However, for shorter duration experiments (i.e., 5 second experiments) and for longer duration experiments (i.e., 15 second experiments) at lower microwave power (50 or 300 W), the amount of energy released is greater than the amount of energy put in. For the lowest energy experiments (50 W), the amount of energy released was at least about 10 times greater than the energy put in. However, as seen from Table 7, for large diameter nanotubes (1.5 nm for example), the amount of energy released was less than the amount of energy put in.

**[0067]** Additional comparative examples were performed by varying other parameters of experiments 1-30. In a first set of comparative examples, low density purified and unpurified SWNT samples (i.e., SWNTs having a density that is about 40 times lower than BuckyPearl® SWNTs) were exposed to about 420 W microwave irradiation in air. The unpurified SWNT samples which contained a large amount of amorphous carbon and amorphous iron catalyst material ignited and burned within a few seconds of being irradiated. After the completion of the irradiation, it was determined that the remainder of the burned material included hematite ( $\text{FeO}_3$ ) and purified nanotubes (i.e., more purified than the starting nanotube sample). The purified samples containing a low amount of amorphous carbon and catalyst material did not burn, but instead displayed random scintillation of white light. No discernable changes were observed by electron microscopy and Raman spectroscopy in the purified samples after the microwave irradiation.

**[0068]** In a second set of comparative examples, the experiments of the first set of comparative examples were repeated in a vacuum ambient. The vacuum system was constructed from stainless steel with the nanotubes placed in a glass vessel or tube that was attached to the vacuum system through a glass to vacuum seal. The glass vessel was about 2 cm in diameter and several centimeters in length. There was no crushed quartz or powdered silica on top of the nanotubes. The nanotubes were merely placed in the bottom of the glass vacuum vessel. The magnetron was placed directly underneath the glass vacuum vessel approximately  $2/3$  wavelengths away (8cm). It is believed that the magnetron in this configuration provided an electric

field that is less than 10,000 V/cm in the nanotubes because the magnetron microwave source was not sufficiently close (i.e., farther than 4 mm away) from the nanotubes.

[0069] The unpurified nanotubes expanded in volume immediately upon application of the microwave field and then later contracted back to their original volume. During this time they emitted light. The purified single walled nanotubes of the original diameter produced light spectra very similar to that of unpurified nanotubes. Furthermore, hydrogen was desorbed from the nanotube samples upon application of the microwave field. This experiment was repeated multiple times to show repeatability and reproducibility of the light spectra which repeated identically (within experimental error limitations) from run to run. However, no energy gain was observed at the low electric field configuration, although it was noted that the nanotubes would become very hot as a result of the application of the microwave field. Thus, even without an energy gain, nanotubes can rapidly and efficiently convert microwave energy to thermal energy. The nanotubes expanded and contracted repeatedly and flew about the vacuum vessel, which allowed some of them to escape the microwave field.

[0070] In a third set of comparative examples, the large vacuum vessel described above was replaced with a smaller one and small amount of SiO<sub>2</sub> powder placed above the nanotubes to attempt to hold the nanotubes in place during microwave irradiation and to prevent the nanotubes from flying around the sample vessel or tube. The smaller vacuum vessel was a tube having a 4 mm inner and a 6 mm outer diameter. 25 mg plus or minus a very small mass (2%) due to experimental error of 1.1 nm SWNTs was placed into the vacuum vessel. The samples had small amounts 25mg of powdered SiO<sub>2</sub> on top of them. The vacuum level of at least 10<sup>-5</sup> torr but not lower than 10<sup>-9</sup> torr was used. This level of vacuum is more than sufficient to remove enough of the oxygen from the atmosphere that oxidation of the sample could not take place. Five of the runs were performed with purified nanotubes and five were performed with unpurified nanotubes in the low electric field configuration.

[0071] However, the smaller diameter vacuum vessel and the small amount of SiO<sub>2</sub> powder were not sufficient to hold the nanotubes in place. The nanotubes were still

blown around the vacuum system very quickly as the hydrogen in the samples was desorbed, which was verified by a residual gas analyzer or RGA. It is believed that the narrow vacuum vessel did not allow expansion of the nanotube sample. Thus, the nanotubes became essentially micro thrusters. Thus, small amounts of nanotubes in this configuration can produce a small amount of thrust for airborne terrestrial and space vehicles, devices or payloads through release of stored gases. No energy gain was observed in the low electric field configuration.

[0072] In a fourth set of comparative examples, the amount of SiO<sub>2</sub> was increased. 1 gram of crushed Pyrex was used for a 25 mg SWNT sample. It was found that in some of the runs the nanotubes would remain stable and not move, but in other runs the nanotubes still moved around the vacuum system and out of the range of the microwave source. No energy gain was observed in the low electric field configuration even when the nanotubes remained in the microwave field and did not move about the vacuum vessel.

[0073] In a fifth set of comparative examples, the microwave source to sample distance was reduced to just about 0.5 mm plus the thickness of the glass. This is a higher electric field configuration for a magnetron microwave source. However, it is believed that the electric field induced in the nanotubes by the microwaves was less than 10,000 V/cm because the magnetron microwave source lacked a reflector which is adapted to increase the electric field induced by the magnetron to 10,000 V/cm and above. Low density 1.1 nm SWNTs were used in five runs. In these runs, slightly faster (several tens of ms) desorption of gasses from the samples were observed than in the other comparative example. Furthermore, through post irradiation sample analysis, it was determined that in short exposure times (4-7 seconds) the majority of the nanotubes would take on a semiconducting form rather than a mixture of conductors and semiconductors. No energy gain was observed.

[0074] In a sixth set of comparative examples, the low density SWNTs were replaced with purified BuckyPearl® SWNTs which are approximately 40 times more dense than the nanotubes used in the other comparative examples. The blackbody temperatures started to elevate beyond the about 2000 to 3000 Kelvin to temperatures

more closely approximating the surface of the sun and beyond (about 5500-6500 Kelvin). Furthermore, a plasma was observed in these runs. The plasma was singly ionized during most of the runs and reached a doubly ionized state near the end of the 25-35 second long runs. The plasma may reach higher ionized states with a higher electric field. A higher electric field may be obtained by any suitable microwave source adjustment, such as by placing a flat piece of metal that completely covers the reflector 10 shown in Figure 8A, except for an aperture (about 1/4 wavelength in size) on the front of the reflector. The aperture is located directly over the top of the antennae post 14 of the magnetron 12. Other suitable methods of increasing the electric field may also be used. Thus, the high density, low diameter SWNTs irradiated in a relatively high electric field produced a plasma. However, no energy gain was observed. Without wishing to be bound by a particular theory, it is believed that the electric field induced in the nanotubes was not sufficiently high to produce the energy gain. In contrast, it is believed that the electric field in examples 1-25 illustrated in Tables 1-5 and 8 above was above 10,000 V/cm due to the microwave source and sample configuration.

**[0075]** Regarding experiments with Rossetter Holdings MWNTs with very narrow inner diameters (i.e., dual wall MWNTs with about 0.9 nm inner diameter), the energy release was so rapid (i.e., about 1/10 the time for SWNTs) that it results in a thermal shock which shatters the tube containing the nanotubes. The shattering of the nanotube and the splatter of the SiO<sub>2</sub> packing material prevented an accurate estimation of the energy release. It is believed that a net energy gain (positive net energy release) is also present with narrow inner diameter MWNTs, but it could not be quantified due to shattering of the tubes.

**[0076]** The factor of 10 or less energy gain described with respect to examples 1-25 above and no energy gain for the larger diameter nanotubes of comparative examples 1-30 are an extremely conservative lower limit because it neglects heat removed from the tube by air convection, heat losses from the intense broadband photon radiation and the assumptions that all of the radiated microwave power, actually broadcast over a hemisphere, is totally directed at the small SWNT volume (<1 cm<sup>3</sup>) and absorbed

with 100% efficiency. Independent resonant cavity measurements of the same SWNT materials that give the anomalous heat emission show, in fact, that only about 15% absorption of the power directly incident on the sample at 2.45 GHz. Applying this absorption factor changes, the minimum energy gain increases from a factor of about 10 to a factor of about 60. Furthermore, if the wide area radiation pattern is factored in, then the energy gain would increase even higher than a factor of 60, such as a factor of about 100 to about 1000.

[0077] An energy gain of a factor of about 1,000 can be estimated from the following calculation. The input microwave power for a 50W microwave source at sample volume of about  $3 \text{ cm}^3$  provides a total input microwave power, corrected for solid angle, of about 40W. Thus, the power absorbed by the nanotubes is about 40W after the about 0.3 absorption cross section is taken into account. Thus, for about a 3 second microwave irradiation, the input microwave energy,  $Q_{IN}$ , equals to  $(40\text{J/s})(3\text{s}) = 120\text{J} = 0.12 \text{ kJ}$ .

[0078] The output energy may be estimated as follows. From the first law of thermodynamics,  $Q_{OUT} = Q_{IN}$ . Thus,  $Q_{OUT} = Q_{HEAT TUBE} + Q_{MELT SiO_2} + E_{RADIATION} + Q_{CHEM REACTIONS} + Q_?$ . The values of  $Q_{HEAT TUBE}$ ,  $E_{RADIATION}$ ,  $Q_{CHEM REACTIONS}$  and  $Q_?$  are assumed to be greater than zero (i.e., for only endothermic reactions, if any, occurring). The heat absorbed to raise the temperature of the  $SiO_2$  to the melting point and the heat required to complete melting is provided by  $Q_{MELT SiO_2} = Q(\text{heat to mp}) + Q(\text{melt})$ .

$$Q(\text{heat to mp}) = \Delta H^\circ (298 \rightarrow \sim 1900) =$$

$$1900$$

$$\int_{298} C_p(T) dT = H^\circ_{1900} - H^\circ_{298} = 111.5 \text{ kJ/mol.}$$

$$298$$

[0079] Thus,  $Q(\text{heat to mp}) = 111.5 \text{ kJ/mol}$  (for  $SiO_2$  crystal based on data from NIST Standard Reference Database 69, 03/2003 Release: NIST Chemistry WebBook).  $Q(\text{melt}) = \Delta H^\circ_{MELT} (SiO_2, \text{ crystal}) = 9.6 \text{ kJ/mol}$  (based on data from

Handbook of Chemistry and Physics, 81<sup>st</sup> ED, David R. Lide, Ed, CRC Press, NY, 2000). Thus,  $Q_{\text{MELT}}=Q(\text{heat to mp})+Q(\text{melt})=111.5 + 9.6 = 121.1 \text{ kJ/mol}$ .

[0080] The thermal balance of the input microwave energy and the minimum output energy may be provided as measured by the melting of the  $\text{SiO}_2$  and the radiation. The value of the latter quantity is not given but clearly it is positive since intense optical and ultra-violet radiation is observed to come from the sample. The result of the balance is that insufficient energy is inputted from the microwave source to account for all the energy flowing out of the nanotube sample.

[0081] Thus, energy  $Q_{\text{OUT}} = \text{heating of surroundings} + \text{heat of phase transitions} + \text{radiation} + \text{energy associated with chemical reactions (+ or -)}$ . In other words,  $Q_{\text{OUT}} = Q_{\text{HEAT SYSTEM}} + Q_{\text{MELT SiO}_2} + E_{\text{RADIATION}} + Q_{\text{CHEM REACTIONS}} > 111.5 + 9.6 \text{ kJ/mol} > 0 + > 0$ . Thus,  $Q_{\text{OUT}} > 121 \text{ kJ/mol}$ . For 60 g of  $\text{SiO}_2$  (=1.0 mol),  $Q_{\text{OUT}} > 121 \text{ kJ}$ . Thus, the ratio of input energy to output energy exceeds 1000:  $Q_{\text{OUT}}/Q_{\text{IN}} > (121 \text{ kJ})/(0.12 \text{ kJ}) \sim 10^3$ . Thus, an energy gain factor of 10 to 1,000 may be achieved by irradiating the nanotubes with microwaves.

[0082] The above specific examples establish that microwave irradiation of single wall carbon nanotubes under vacuum conditions can cause intense emission of light, ranging from UV to near IR wavelengths, and heat output that significantly exceeds the total input power of the microwave fields by at least an order of magnitude. However, the underlying mechanisms for this effect have not been conclusively established.

[0083] Without wishing to be bound by a particular theory, the present inventors believe that the energy gain may occur because the microwave irradiation of nanotubes may excite phonon and electron resonances. Furthermore, potential quantum effects, such as quantum fluctuations of the EM field trapped in the nanotubes, triggered by the quasi-1D geometries of the small diameter nanotubes may also be responsible for the energy gain. Furthermore, it is possible that carbon tips act as pin point electron field emitters, which contribute to the effect. Low energy 2.45 GHz radiation can induce excitation of the system to produce electron temperatures of



about 5000-6000 K with accompanying photon emission at UV frequencies, an upconversion of  $>10^5$ . The nanotube structures unexpectedly remain intact at high electron temperatures given estimates of about 4000 K for the disintegration threshold. Thus, the conversion of microwave energy to thermal energy (as well as to UV, visible and IR radiation emission) may be generated continuously, such as for at least 10 minutes to 1.5 hours, for example between 10 minutes and 10 hours, without destroying a structural integrity of the nanotubes. The energy conversion continues until the nanotubes are destroyed or until the container holding nanotubes is breached by the heat emitted by the nanotubes. Furthermore, if the nanotubes are heated at a temperature of 1500 °C or above, the diameters of the nanotubes double and then quadruple which eventually terminate the energy conversion. Thus, nanotubes may be heated at a temperature of below 1500 °C, such as about 1300 °C for long term, continuous energy conversion. In order to provide long term energy gain from microwave irradiation of nanotubes, the nanotubes may be continuously replaced or cycled in the zone of microwave irradiation. Thus, the nanotubes may be placed in a fluid, such as a liquid or a gas, which cycles nanotubes through the microwave irradiation zone. As the nanotubes increase in diameter after a period of microwave irradiation, they are replaced with other nanotubes of a sufficiently small diameter to continue to convert microwave energy to thermal energy, preferably with an energy gain.

**[0084]**        *APPLICATIONS.* The energy generated by a first material, such as the nanotubes, which is irradiated by the long wavelength radiation, is provided to a second material. The second material is different from the first material and the second material may be located adjacent to the first material. The second material may comprise a portion of any suitable device or article of manufacture. Exemplary devices and articles which utilize the energy gain described below. It should be noted that other devices and articles may also use the energy gain. Other exemplary devices and articles which utilize the energy generation are described in U.S. Published Application 2004/0180244 incorporated herein by reference in its entirety

[0085] When carbon nanotubes are placed in an EM field in the microwave and radio frequency region, carbon nanotubes can sustain charged particle plasmas that can reach temperatures of at least as high as 5000 Kelvin. Furthermore, these temperatures are attained within a second. These plasmas generate intense or hyper bright light that can be as bright as a typical welding arc, generally estimated to be equivalent to a temperature of approximately 6000 Kelvin. The production of hyper bright plasmas from carbon nanotubes exposed to electromagnetic irradiation provide a method for providing remotely controlled extremely bright light produced instantaneously at the location where the carbon nanotubes are placed. By placing the carbon nanotubes in a non-oxidizing ambient at specific locations (very high E-field points) within the volume of an object that can be penetrated by a remotely controlled EM field, the plasmas will be created at those locations and subsequent irradiation of bright light will bathe and radiate from those regions. Further, by selecting the amount of carbon nanotubes to be placed at a desired location, the intensity of the desired effect can be precisely regulated. By selecting the power of the applied EM field, the intensity of the desired effect can be precisely regulated.

[0086] The placement of carbon nanotubes at specific locations followed by controlled irradiation by selected frequencies of electromagnetic fields advantageously allows the delivery of remotely controlled plasma, light and heat instantaneously to regions within an object. This feature can be used to initiate chemical reactions or physical processes at that exact location. Since a single carbon nanotube should be able to sustain a plasma, the size or volume of a single location to receive the light from a carbon nanotube may be on the order of the size of the carbon nanotube and thus at the scale of a nanometer in diameter and up to the length of the carbon nanotube.

[0087] This process of releasing thermal energy and generating a plasma, and the apparatus used to implement it, facilitates numerous improvements of existing applications, such as for use in engines and power production plants, and gives rise to novel methods of medical treatments as disclosed herein.

[0088] *POWER PLANT* For example, these above described behavior of nanotubes in EM fields may be useful in a power plant for example. In this case, the nanotubes and the EM field source are provided in conjunction with an apparatus which can convert the heat and/or plasma generated in the nanotubes to electrical energy or power. When one batch of nanotubes is exhausted (i.e., no longer provides an energy gain and plasma due to diameter doubling or other physical change), it is replaced with a fresh batch of nanotubes, similar to the way coal or other fuel is replenished in a conventional power plant. Since MWNTs are fairly inexpensive and provide megawatts of power, they can be used as fuel in a power plant instead of coal or other hydrocarbon fuels, using the above described energy gain or generation methods. Alternatively, other material, such as a gas, for example, may be provided into the plasma on a continuous or batch basis, in order to sustain the plasma.

[0089] *ENGINES*. Although there are a wide variety of engines, one characteristic of engines remains constant, that is they require some type of release of energy. The embodiment of the present invention comprises a novel process and apparatus for energy release that may be useful in several types of existing engines. The embodiment of the present invention advantageously improves efficiency in engines. The present process and apparatus of this invention is described with reference to several types of engines. Regardless of the fuel used, the fundamental operation of any internal combustion engine is essentially the same, if fuel is introduced to an enclosed space and ignited, energy will be released in the form of an expanding gas. The expanding gas will, in turn, push some type of piston which will drive a series of mechanical devices. These mechanical devices will convert the release of energy into some type of work. In the automobile, the work results in some type of motion for the automobile and its contents.

[0090] As described herein, the embodiment of the present invention can be adapted for use as a catalyst for a rapid combustion reaction not entirely dissimilar to current fuel types such as unleaded gasoline or diesel fuel. In other words, nanotubes may be added to a liquid or even a solid fuel, such as hydrocarbon fuel, for example gasoline or diesel fuel. A microwave source is then used to ignite or heat the nanotubes in the

fuel. The ignited or heated nanotubes then ignite the fuel. In a motor vehicle engine, the microwave source can be used as a substitute for spark plugs, since the microwaves are used to create the spark in the nanotubes. Alternatively, the nanotubes may be in a vessel which is located adjacent to the fuel. The vessel is maintained in a non-oxidizing ambient, such as a vacuum or inert ambient. The nanotubes in the vessel are heated by microwaves and the heat from the nanotubes is then transferred to the fuel through the container walls and/or through a heat transfer medium, such as a heat pipe. This configuration may be used in a steam engine.

[0091] Regardless of the type of engine, to generate power, some sort of fuel is necessary and some sort of reaction must take place. In internal combustion engines, a spark that ignites fuel that has been mixed with oxygen is used to start the reaction. Power in excess of that put in will come out of the reaction, the excess coming from the reaction, be it electrochemical, combustion, or electromagnetic. Thus, as described above, local heating by carbon nanotubes exposed to EM radiation can be used for driving mechanical motion such as occurs in internal combustion or steam engines, turbines and other devices with mechanically moving parts whose operation depends upon local heating and thermal gradients. It should be noted that the nanotubes do not necessarily have to be in a non-oxidizing ambient to provide additional thermal energy to an engine. For example, the nanotubes may comprise a fuel additive or be dispersed in an aerosol in the fuel containing areas of an engine to provide additional thermal energy to the fuel in the engine. The nanotubes provide an efficient energy conversion and/or an energy gain from microwave energy to thermal energy for use in the fuel in the engine.

[0092] Furthermore, the nanotubes irradiated by microwaves can act as micro thrusters by expelling adsorbed or absorbed gas and/or by moving in response to microwave irradiation. Thus, small amounts of nanotubes in this configuration can produce a small amount of thrust through release of stored gases and/or through microwave induced movement of the nanotubes.

[0093] *FUEL CELLS.* Further, an embodiment of the present invention includes the use of carbon nanotubes as a fuel storage material for various fuel cells. The

scientific literature is replete with articles dealing with hydrogen storage in nanotubes. However, even if a sufficient amount of hydrogen is stored in the nanotubes, it is desirable to increase the speed of desorption or desorbition of hydrogen from the nanotubes. The microwave irradiation of nanotubes containing adsorbed or absorbed hydrogen causes a rapid desorption or desorbition of hydrogen from the nanotubes as described above. Thus, microwave irradiation may be used to provide hydrogen stored in carbon nanotube hydrogen storage media into the fuel cell. Thus, a fuel cell, such as a PEM or other hydrogen using fuel cell, contains an electrolyte, an anode electrode, a cathode electrode, a carbon nanotube hydrogen storage material and a source of microwave radiation. When it is desired to provide hydrogen into the fuel cell, the nanotube storage material is irradiated with microwaves to remove the hydrogen stored in the nanotubes and to provide it into the fuel cell to generate electricity.

[0094] In another aspect of the fuel cell embodiment, EM fields, such as microwave fields, are used to both remove the hydrogen from its nanotube storage matrix and ionize it in one step. This could be used to increase the efficiency of hydrogen fuel cells. Gases other than hydrogen, including inert gases, can also be used for this effect and are included within the scope of this embodiment of the present inventions. Such devices serve as rapid delivery devices for ionized fuel. These ionized atoms could be separated through a membrane or magnetic field into positive and negatively charged particles which could be made to generate a potential or voltage which could be used by electrical devices. In other words, the ions may be used as fuel in membrane electrolyte type fuel cells and in magnetic field separation type electrical energy generation devices. The devices of this embodiment of the present invention can be scaled down to the nanoscale at which carbon nanotubes exist or higher for such devices as plasma-driven engines or power production plants.

[0095] When carbon nanotubes are heated, first any gases that are present will separate. However if the carbon nanotubes are caused to superheat, a plasma will be formed when the gas is stripped of its electron, as described above. The characteristic of the embodiment of the present invention can be used in a variety of applications,

including but not limited to, fuel cell applications and plasma applications. Fuel cells do not require the plasma to be lighted, just that it is possible to remove the electron from the proton. This embodiment of the present invention could then be used to pass the proton through a fuel cell membrane (i.e., electrolyte) and make use of the electrical charge on the electron to generate electricity. The hydrogen can be extracted and the hydrogen ionized all in the same step improving the efficiency of energy production. The reaction can be controlled in a variety of methods depending on the total heat production, efficiency, and overall reaction speed desired.

[0096] *COMPOSITES*. This process of the embodiment of the present invention can be adapted to rapidly heat whatever structure with which the carbon nanotubes are blended. The carbon nanotubes will be selectively superheated and radiate heat into the matrix in which they are blended, which will in turn cause a rapid increase in temperature in the storage matrix.

[0097] *MEDICAL APPLICATIONS*. In medical applications, the embodiment of the present invention allows the delivery of remotely controlled light and heat instantaneously to regions within the human or an animal body where carbon nanotubes can be placed. This will permit precise methods for microsurgery and microtherapy. For example, cancerous tissues and cells can be selectively removed by use of external electromagnetic fields that activate intense radiation from the carbon nanotubes but are otherwise harmless to the body. The carbon nanotubes can be placed within the body by a variety of means, for example, by chemically attaching to nanoscale objects such as nanoparticles or proteins, that move readily through the body and have specific tags which cause attachment to the target cancer cells. In other words, the carbon nanotubes are attached to nanoscale objects, such as specific proteins or nanoparticles which selectively bind or attach to areas of disease in the human body, such as cancerous cells or other diseased cells, tissue or organ parts. The diseased area of the body is then irradiated by microwaves, preferably from outside the body. The microwaves cause the nanotubes attached to the nanoscale objects to emit intense but localized heat. The intense heat destroys the diseased cells or tissue where the nanotubes are located, but does not significantly effect healthy

cells or tissue. Furthermore, the carbon nanotubes may be used as a diagnostic marker. In one aspect, the nanotubes are bound to the nanoscale objects, which are in turn selectively bound to diseased cells or tissue of the body. Microwave radiation is then used to cause the nanotubes to emit localized light and/or heat. The light and/or heat is detected to determine if and where the diseased cells or tissue are located. Alternatively, a positron emission tomography (PET) contrast agent, such as a radioactive tracer element, is bound to or stored in the nanotubes which are in turn bound to the nanoscale objects. The nanoscale objects are selectively bound to the diseased cells or tissue, which are then detected by a PET scanner due to the emission of gamma radiation from the contrast agent.

[0098] *UNDERWATER HEAT SOURCE.* The embodiment of the present invention can be adapted to cause a desired reaction under water. Such a reaction would be useful in delivery of immediate intense heat sources in this environment for such things as repair of micro-holes in the hulls of ocean going vessels. A hand held embodiment of the present invention could be fabricated to perform this task.

[0099] *LIGHT SOURCE.* The light emitted from carbon nanotubes irradiated by EM waves as described herein exhibit a range of wavelengths or energies ranging from the soft X-ray region through the ultraviolet into the visible and extending into the infrared regions. The peak intensity of this light, typically in the visible region, can be moved to higher or lower wavelengths by using carbon nanotubes of different diameters and/or changing the materials or impurities that are trapped within the interior of the carbon nanotubes. For example, the peak wavelength of the emitted radiation moves to a longer wavelength with an increased nanotube diameter and with an increase in the amount of the doping element in the nanotubes, such as a hydrogen doping element. Using these methods alone or in combination, the spectral characteristics of the light delivered from the carbon nanotubes to specific locations, as required by the specific application, can be altered as desired for the purposes of enhancing the specific application. Thus, the nanotubes may be used in a solid state light source.

[0100] *HEAT SOURCE.* Further, depending on the parameters of amount and type of carbon nanotubes and the frequency and power of the EM radiation, carbon nanotubes can heat the local regions surrounding them to temperatures of at least 2000 K, which can, for example, melt silicon dioxide, a typical component of glass. The production of these high temperatures can be used as a light replacement as the causal agent for the effect by heat and temperature rise. The production of heat to local areas can be used to instantaneously induce local chemical and physical processes such as degradation, initiation of explosions, melting, vaporization, and boiling, among other things.

[0101] In order to vary the heat or control the other effects of the reactions, a variety of parameters can be varied. One such parameter is the total volume of sample involved in the reaction. The volume can vary from microscopic range for small battery or chip power production up as large as that useful for power plant energy production. In order to control the reaction one may also alter the power level delivered to the sample at a particular microwave frequency. The controllability of microwave sources permit small changes in the amount of microwave power delivered in experimental data runs. The small power changes have exponential differences in the amount of heat production or efficiency of the power production. Advantageously, this applications may be implemented in size or scale from the microscopic power requirement range all the way to the macroscopic range.

[0102] Another way to control the reaction process is by exposing the carbon nanotubes to various frequencies of microwaves and in different resonant conditions. Different frequencies in the microwave range are absorbed by carbon nanotubes with different efficiencies. By changing the frequency, the overall efficiency, size and speed of the reaction can be controlled.

[0103] *ENERGY DELIVERY SYSTEM.* The microwave irradiation of nanotubes and other materials can be used as an energy conversion and deliver mechanism, where energy is delivered to a remote object by transmitting a microwave beam to the remote object.



[0104] U.S. application serial number 10/764,092 filed 1/23/04 and U.S. application serial number 10/846,045 filed 5/14/04 are incorporated herein by reference in their entirety. The foregoing description of the invention has been presented for purposes of illustration and description. It is not intended to be exhaustive or to limit the invention to the precise form disclosed, and modifications and variations are possible in light of the above teachings or may be acquired from practice of the invention. The description was chosen in order to explain the principles of the invention and its practical application. It is intended that the scope of the invention be defined by the claims appended hereto, and their equivalents.

What is claimed is:

1. An energy generation method, comprising irradiating a first material containing nanoscale sized space therein with microwave or radio frequency radiation having a first energy to generate a second energy greater than the first energy.
2. An energy generation method, comprising irradiating nanotubes with microwave radiation having a first energy to generate a second energy greater than the first energy from about the nanotubes.
3. An energy generation method, comprising:  
providing carbon nanotubes located in a non-oxidizing ambient in a container;  
irradiating the carbon nanotubes with pulsed microwave radiation to provide an electric field of at least 10,000 V/cm in the carbon nanotubes for a sufficient time to generate thermal energy from the carbon nanotubes,  
wherein a magnitude of the thermal energy is greater than a magnitude of an energy of the pulsed microwave radiation.
4. The method of claim 1, wherein the nanoscale sized space comprises a filled or an unfilled cavity in the first material having a smallest dimension of less than 1.2 nanometers.
5. The method of claim 1, wherein the nanoscale sized space comprises a substantially cylindrical enclosed space in the first material having a diameter of less than 1.2 nanometers.
6. The method of claim 1, wherein the first material comprises a carbon material.
7. The method of claim 1, wherein the first material comprises carbon nanotubes.

8. The method of any one of claims 1 to 7, wherein the first material or the nanotubes comprise single walled carbon nanotubes having an internal diameter of 1.1 nm or less.
9. The method of any one of claims 1 to 7, wherein the first material or the nanotubes comprise multi-walled carbon nanotubes.
10. The method of any one of claims 7 to 9, wherein the carbon nanotubes comprise purified carbon nanotubes.
11. The method of any one of claims 7 to 10, wherein the carbon nanotubes comprise highly dense carbon nanotubes.
12. The method of any one of claims 1 to 11, wherein the step of irradiating comprises irradiating the first material or the nanotubes with microwaves.
13. The method of any one of claims 1 to 12, wherein the step of irradiating comprises irradiating the first material or the nanotubes with microwaves to provide an electric field of at least about 10,000 V/cm in the first material or the nanotubes.
14. The method of any one of claims 7 to 13, wherein the step of irradiating comprises irradiating carbon nanotubes having an internal diameter of 1.1 nm or less and located in a non-oxidizing ambient with microwave pulses such that an electric field of at least about 10,000 V/cm is provided in the carbon nanotubes for a sufficient time to generate the second energy having a magnitude that is greater than the first energy.
15. The method of claim 14, wherein the carbon nanotubes are restrained from moving during the irradiation with the microwaves pulses.
16. The method of claim 14, further comprising:

placing the carbon nanotubes in a container;  
fixing the carbon nanotubes to a predetermined location in the container;  
generating a vacuum in the container; and  
irradiating the carbon nanotubes with the microwave radiation while the carbon nanotubes are fixed and while the vacuum is maintained in the container.

17. The method of claim 14, further comprising:  
placing the carbon nanotubes in a container;  
fixing the carbon nanotubes to a predetermined location in the container;  
providing an inert ambient in the container; and  
irradiating the carbon nanotubes with the microwave radiation while the carbon nanotubes are fixed and while the inert ambient is maintained in the container.
18. The method of any one of claims 7 to 17, wherein the carbon nanotubes comprise purified, highly dense carbon nanotubes.
19. The method of any one of claims 1 to 18, wherein the second energy comprises a thermal energy having a magnitude greater than the first energy.
20. The method of any one of claims 1 to 19, wherein a magnitude of the second energy is at least 10 times greater than the first energy.
21. The method of any one of claims 1 to 20, wherein the irradiated first material or the nanotubes emit at least one of infrared, visible and ultraviolet radiation.
22. The method of any one of claims 1 to 21, further comprising irradiating the first material or the nanotubes to generate a plasma about the first material.
23. The method of any one of claims 1 to 22, wherein the step of irradiating comprises irradiating the first material or the nanotubes with microwave radiation from a magnetron microwave source having a microwave emitter positioned 4 mm or

less from the first material or the nanotubes in a near field configuration to generate an electric field of at least 10,000 V/cm in the first material or the nanotubes.

24. The method of any one of claims 1 to 23, wherein the step of irradiating comprises irradiating the first material or the nanotubes with microwave radiation from a microwave source selected from a group consisting of a resonant device coupled to one of a klystron and backward wave oscillator to generate an electric field of at least 10,000 V/cm in the first material or the nanotubes.

25. The method of any one of claims 1 to 24, wherein the step of irradiating comprises irradiating the first material or the nanotubes with microwave radiation having a frequency between 2 GHz and 90 GHz and power between 30W and 1 kW.

26. The method of any one of claims 1 to 25, further comprising irradiating the first material or the nanotubes with the microwave or radio frequency radiation to generate the second energy without destroying a structural integrity of the first material or the nanotubes.

27. The method of any one of claims 1 to 26, further comprising providing the second energy generated by the first material or the nanotubes to a second material, wherein the second material is different from the first material or the nanotubes and the second material is located adjacent to the first material or the nanotubes.

28. A method of generating a plasma, comprising:  
providing a first material;  
irradiating the first material with pulsed microwave radiation having a first frequency; and  
generating a plasma about the first material having a second frequency different from the first frequency.

29. The method of claim 28, wherein:

the first material comprises carbon nanotubes;  
the nanotubes have an internal diameter of 1.1 nm or less;  
the nanotubes are maintained in a non-oxidizing ambient; and  
an electric field of at least 10,000 V/cm in the nanotubes is generated by the microwave radiation.

30. An energy generation apparatus, comprising:  
a first material containing nanoscale sized space therein; and  
a microwave or radio frequency radiation emitting device positioned with respect to the first material to irradiate the first material with microwave or radio frequency radiation having a first energy to generate a second energy greater than the first energy.
31. An energy generation apparatus, comprising:  
nanotubes located in a container adapted to provide a non-oxidizing ambient to the nanotubes; and  
a microwave emitting device positioned with respect to the first material to irradiate the first material with pulsed microwave radiation having a first energy to generate a second energy from the about nanotubes greater than the first energy.
32. An energy generation apparatus, comprising:  
a first material containing nanoscale sized space therein; and  
a first means for irradiating the first material with microwave or radio frequency radiation having a first energy to generate a second energy from the first material greater than the first energy.
33. An energy generation apparatus, comprising:  
carbon nanotubes having an internal diameter of 1.1 nm or less located in a container adapted to provide a non-oxidizing ambient to the nanotubes; and  
a first means for irradiating the carbon nanotubes with pulsed microwave radiation having a first energy to provide an electric field of at least 10,000 V/cm in

the carbon nanotubes and to generate a second energy from the carbon nanotubes greater than the first energy.

34. The apparatus of claims 30 or 32, wherein the nanoscale sized space comprises a filled or an unfilled cavity in the first material having a smallest dimension of less than 1.2 nanometers.

35. The apparatus of any one of claims 30, 32 or 34, wherein the nanoscale sized space comprises a substantially cylindrical enclosed space in the first material having a diameter of less than 1.2 nanometers.

36. The apparatus of any one of claims 30, 32, 34 or 35, wherein the first material comprises a carbon material.

37. The apparatus of any one of claims 30, 32, 34, 35 or 36, wherein the first material comprises carbon nanotubes.

38. The apparatus of any one of claims 30 to 37, wherein the first material or the nanotubes comprise single walled carbon nanotubes having an internal diameter of 1.1 nm or smaller.

39. The apparatus of any one of claims 30 to 37, wherein the first material or the nanotubes comprise multi-walled carbon nanotubes.

40. The apparatus of any one of claims 30 to 39, wherein the first material or the nanotubes comprise purified carbon nanotubes.

41. The apparatus of any one of claims 30 to 40, wherein the first material or the nanotubes comprise highly dense carbon nanotubes.

42. The apparatus of any one of claims 30 to 41, wherein the device or first means is adapted to irradiate the first material with microwaves.
43. The apparatus of any one of claims 30 to 42, wherein device or first means is adapted to irradiate the first material or the nanotubes with microwaves to provide an electric field of at least about 10,000 V/cm in the first material or the nanotubes.
44. The apparatus of any one of claims 37 to 43, wherein the device or first means is adapted to irradiate carbon nanotubes having an internal diameter of 1.1 nm or less and located in a non-oxidizing ambient with microwave pulses such that an electric field of at least about 10,000 V/cm is provided in the carbon nanotubes for a sufficient time to generate the second energy having a magnitude that is greater than the first energy.
45. The apparatus of claim 44, wherein the carbon nanotubes are restrained from moving.
46. The apparatus of claim 44, further comprising a vacuum source connected to a container containing the carbon nanotubes.
47. The apparatus of claim 44, further comprising a non-oxidizing gas source connected to a container containing the carbon nanotubes.
48. The apparatus of any one of claims 30 to 47, wherein the first material or the nanotubes comprise purified, highly dense carbon nanotubes.
49. The apparatus of any one of claims 30 to 48, wherein the second energy comprises a thermal energy having a magnitude greater than the first energy.
50. The apparatus of any one of claims 30 to 49, wherein a magnitude of the second energy is at least 10 times greater than the first energy.



51. The apparatus of any one of claims 30 to 50, wherein the irradiated first material or the nanotubes emit at least one of infrared, visible and ultraviolet radiation.
52. The apparatus of any one of claims 30 to 51, wherein the first material or the nanotubes are adapted to generate a plasma about the first material or the nanotubes when irradiated with microwaves.
53. The apparatus of any one of claims 30 to 52, wherein the device or first means comprises a magnetron microwave source having a microwave emitter positioned 4 mm or less from the first material or the nanotubes in a near field configuration to generate an electric field of at least 10,000 V/cm in the first material or the nanotubes.
54. The apparatus of any one of claims 30 to 52, wherein the device or first means comprises a resonant device coupled to one of a klystron and backward wave oscillator to generate an electric field of at least 10,000 V/cm in the first material or the nanotubes.
55. The apparatus of any one of claims 30 to 54, wherein the device or first means is adapted to emit microwave radiation having a frequency between 2 GHz and 90 GHz and power between 30W and 1 kW.
56. The apparatus of any one of claims 30 to 55, further comprising providing a second material which is different from the first material or the nanotubes, wherein the second material is located adjacent to the first material or the nanotubes.
57. A method of generating a plasma, comprising irradiating nanotubes with microwaves in a non-oxidizing ambient to generate the plasma.
58. The method of claim 57, wherein the plasma comprises an ionic plasma.

59. The method of claim 58, wherein the ionic plasma comprises at least one of a carbon plasma and a plasma of species present about the nanotubes.
60. The method of claim 59, wherein the species present about the nanotubes comprises hydrogen adsorbed or absorbed to the nanotubes.
61. The method of any one of claims 57 to 60, wherein the nanotubes comprise carbon nanotubes having an inner diameter of 1.1 nm or less.
62. The method of claim 61, wherein the nanotubes comprise SWNTs or MWNTs and the microwaves comprise pulsed microwaves.
63. The method of claim 57, wherein an electric field of at least 5,000 V/cm is induced by the microwaves in the nanotubes.
64. A method of generating light or heat comprising irradiating nanotubes with microwaves in a non-oxidizing ambient to generate at least one of light and heat.
65. A method of desorbing hydrogen from nanotubes comprising irradiating the nanotubes containing stored hydrogen with microwaves in a non-oxidizing ambient to desorb hydrogen from the nanotubes.

FIGURE 1





FIGURE 2

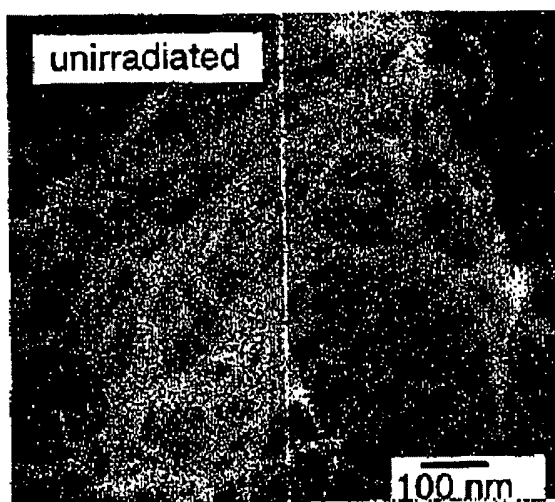


FIGURE 3A

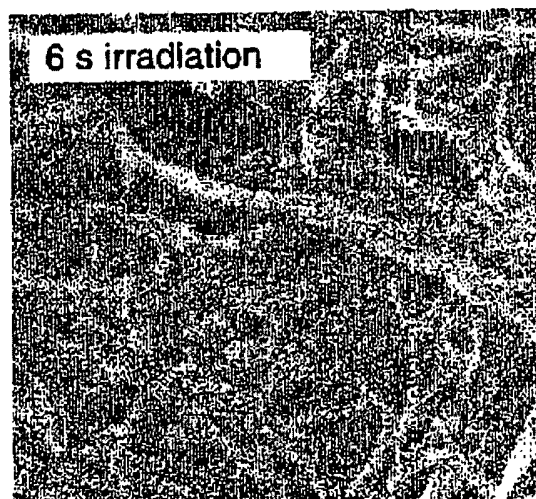


FIGURE 3B

FIGURE 4A

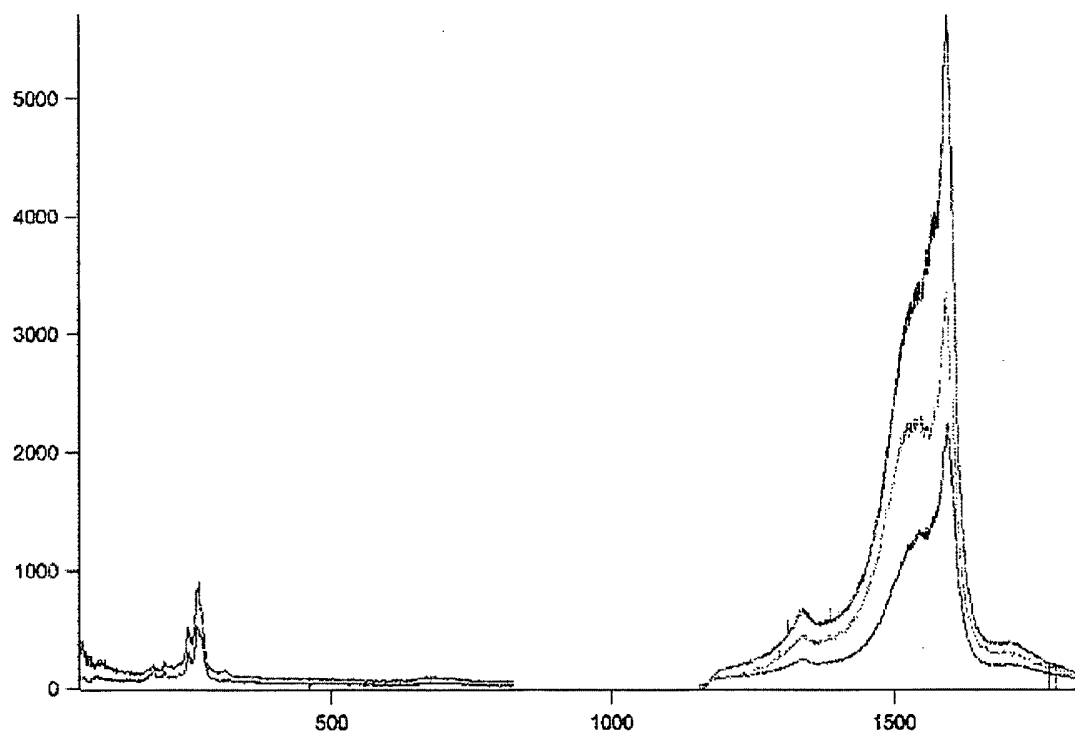
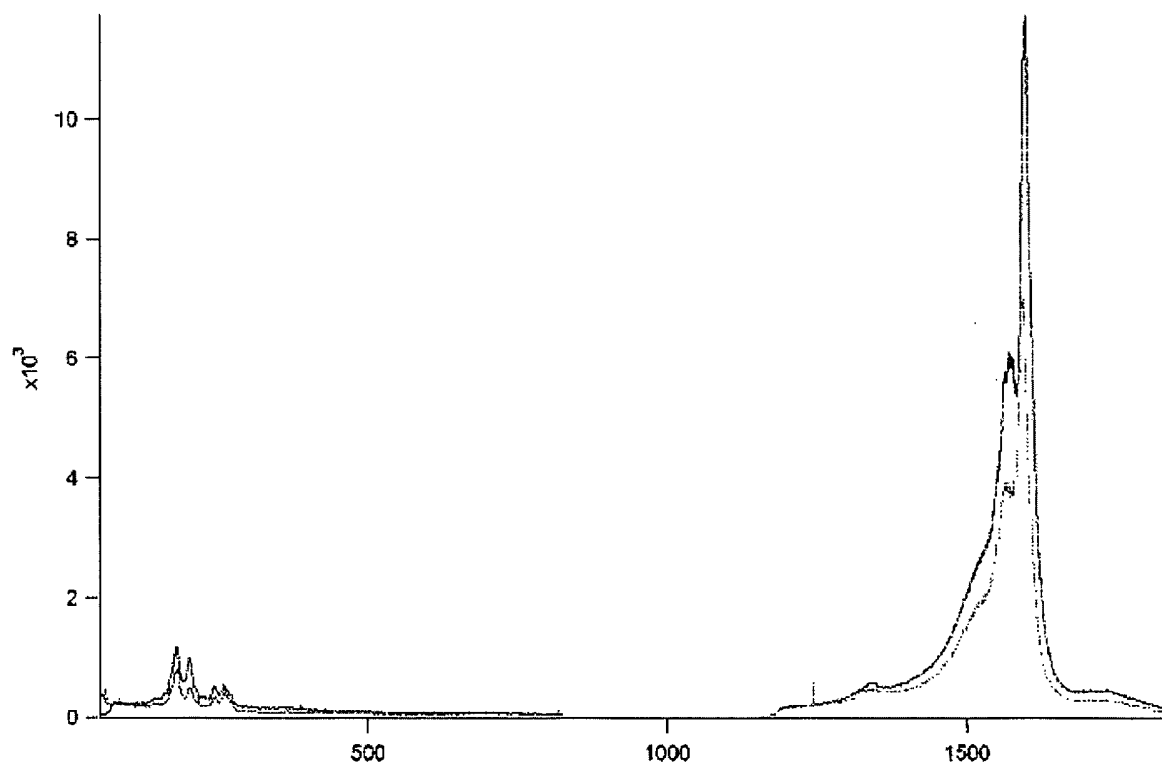


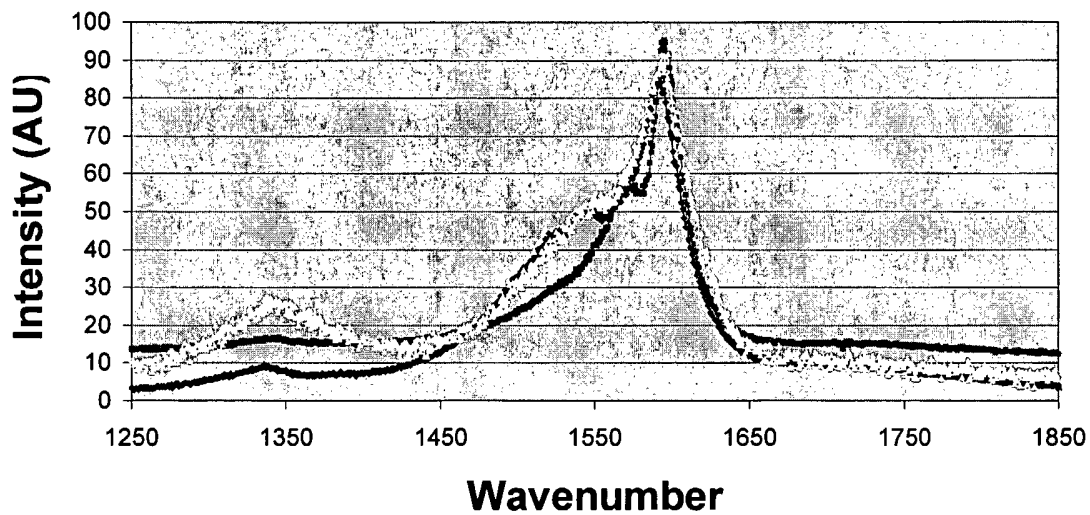
FIGURE 4B



6/11

FIGURE 4C

# RAMAN Spectra Argon Ion Laser



→ No Exposure to Microwaves -- Short Exposure to Microwaves ··· Long Exposure to Microwaves



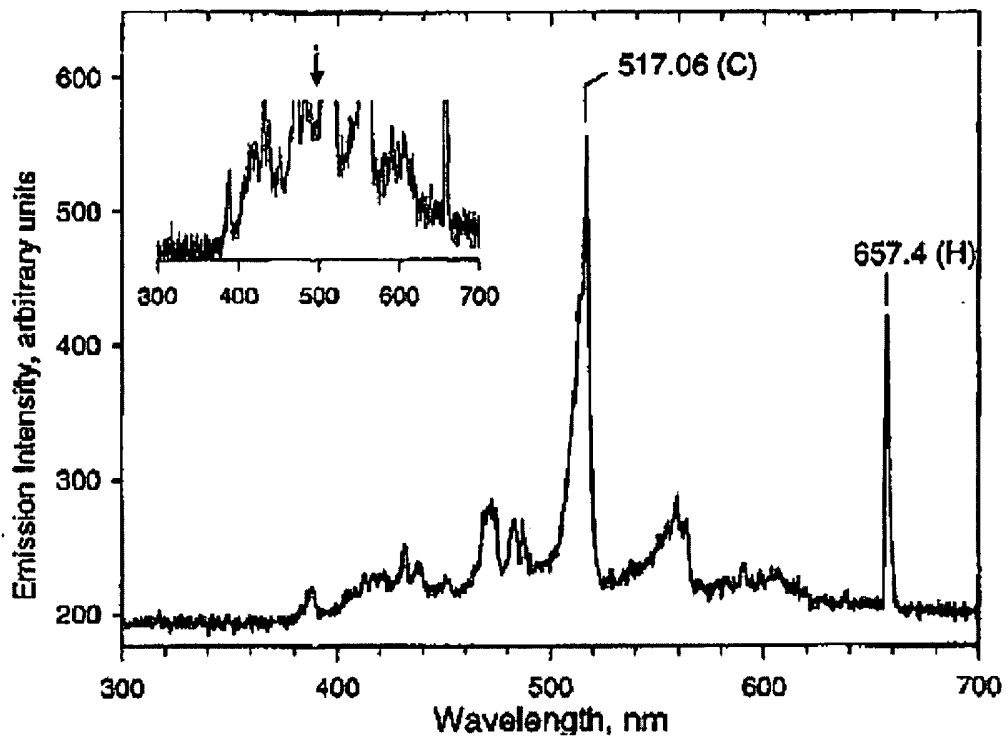


FIGURE 5

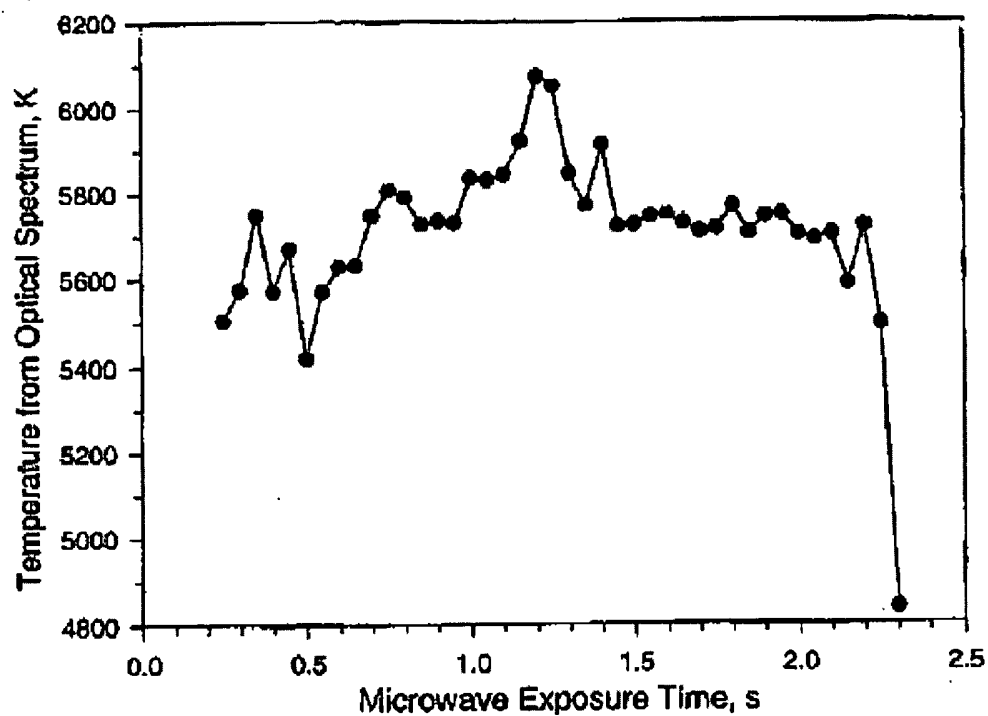


FIGURE 6

FIGURE 7

Nanotube Sample with Hydrogen Removed Previous to Data Acquisition vs. Known Source

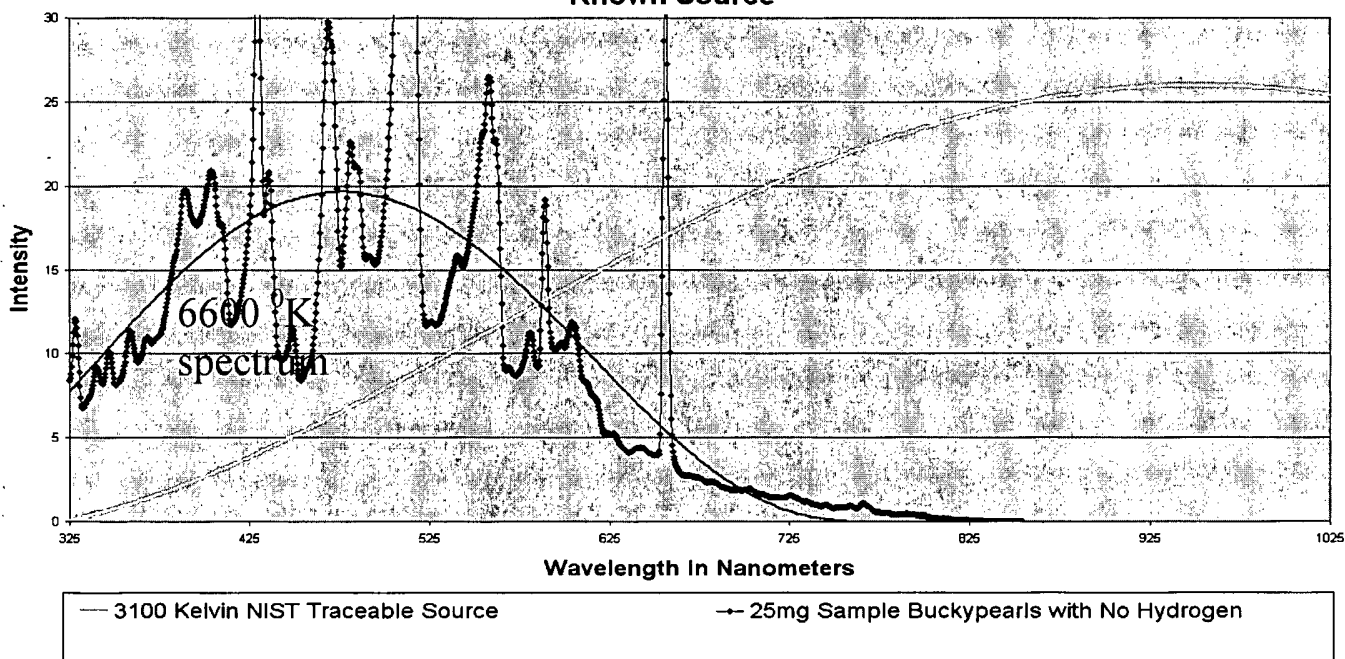


FIGURE 8G

Bottom piece  
E

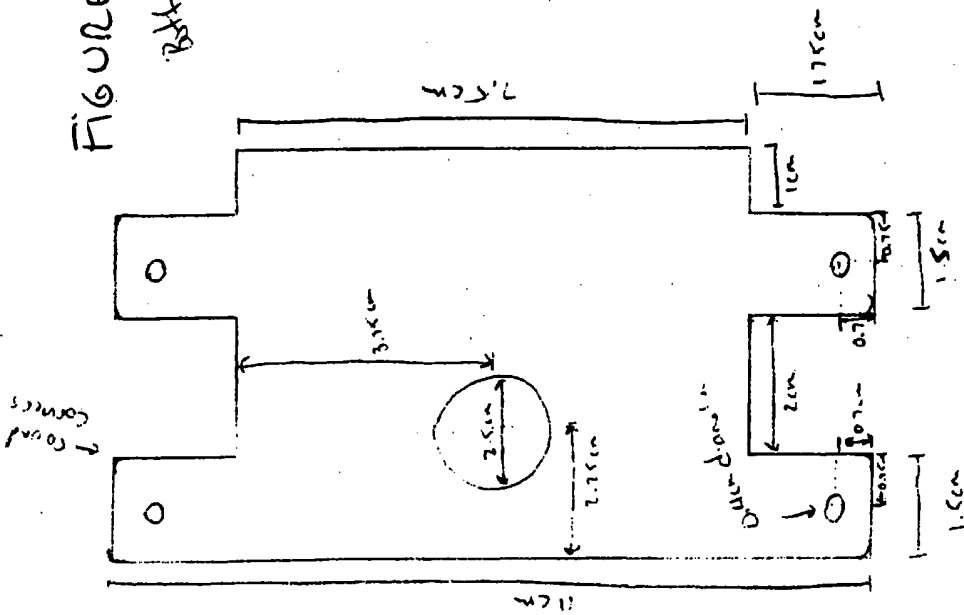


FIGURE 8B  
Assembly

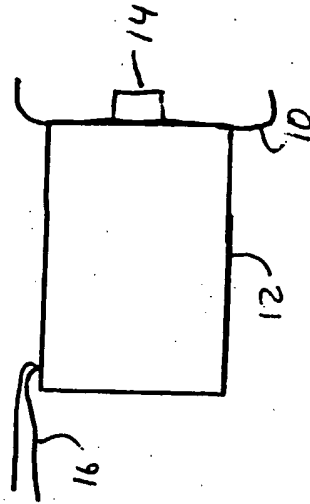
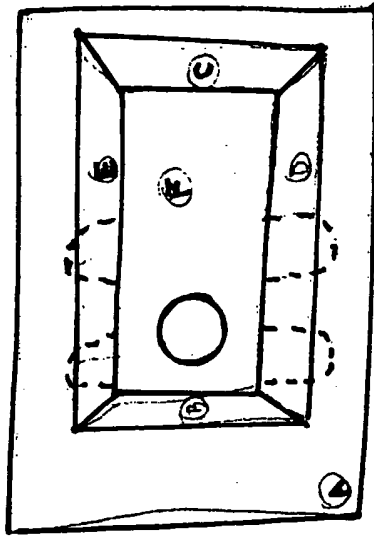


FIGURE 8A

D  
E  
Front & Back piece

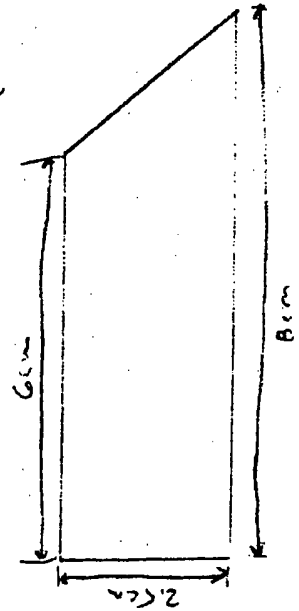
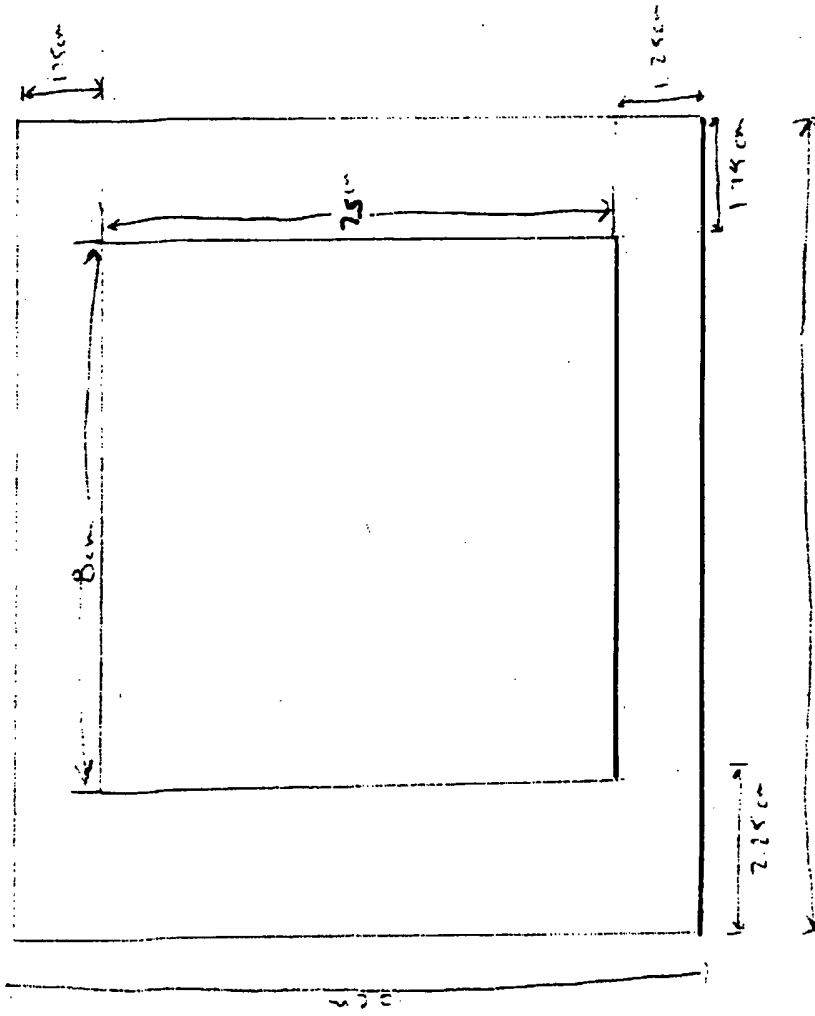


FIGURE 8F

aluminum 1/32" thick

FIGURE 8C

top piece (A)



(B)

Right

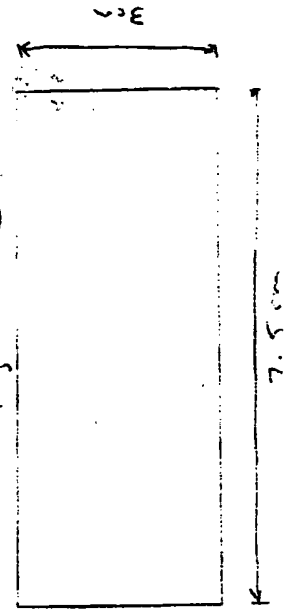


FIGURE 8E

left piece (C)

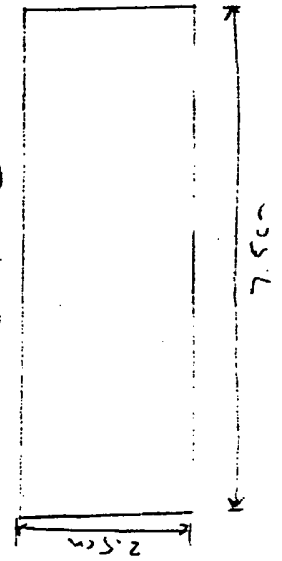


FIGURE 8D