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Perry et al.

(54) **MICROWAVE HEATING OF ENERGETIC MATERIALS**

(76) Inventors: **William L. Perry**, Jemez Springs, NM (US); **Steven F. Son**, Los Alamos, NM (US); **Blaine W. Asay**, Los Alamos, NM (US)

Correspondence Address:
UNIVERSITY OF CALIFORNIA
LOS ALAMOS NATIONAL LABORATORY
P.O. BOX 1663, MS A187
LOS ALAMOS, NM 87545 (US)

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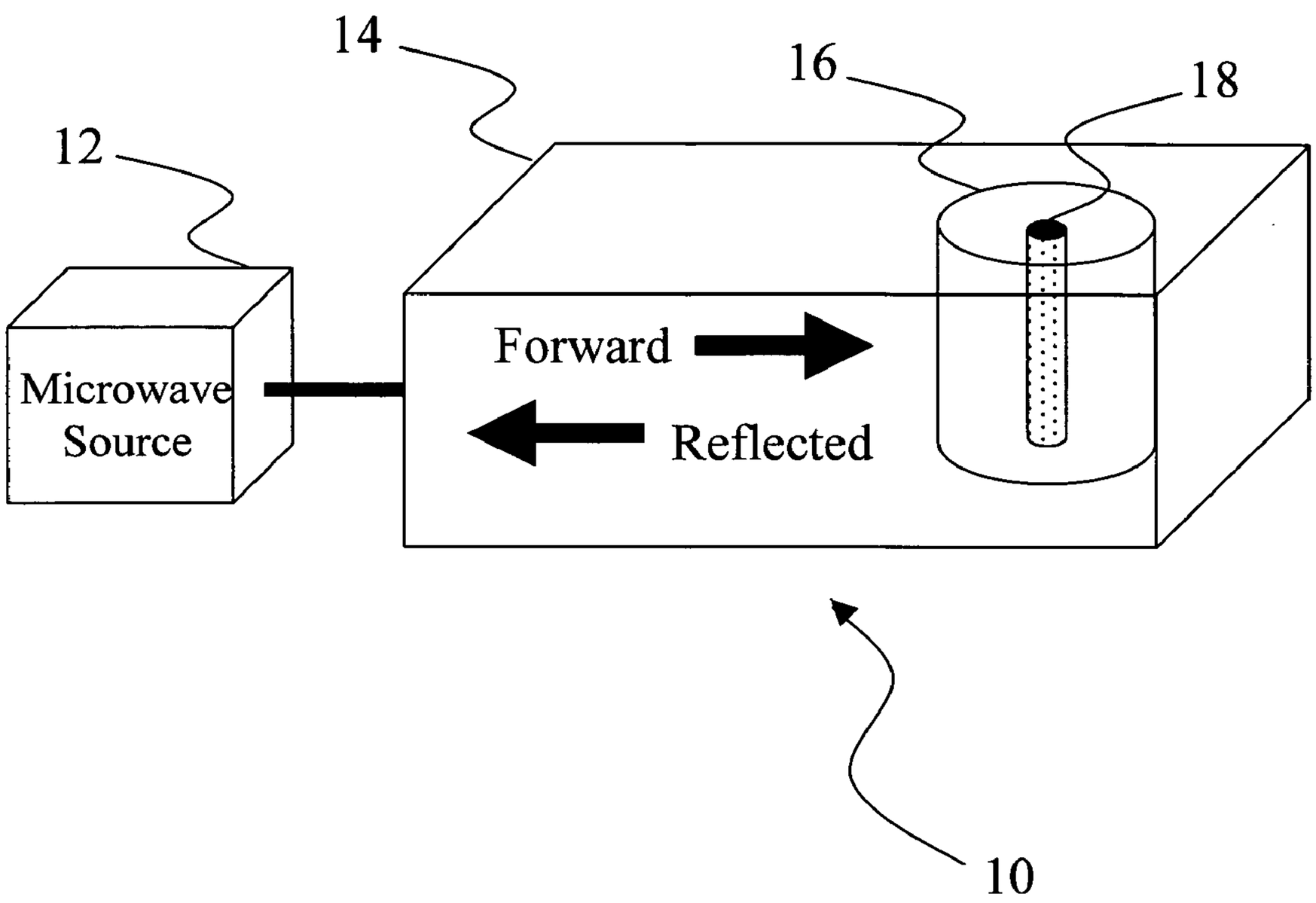
(57) **ABSTRACT**

Mixtures of high explosives with materials that readily absorb microwaves ignite more readily when exposed to microwave energy than the corresponding neat explosives. A charge of HMX (0.5 gram) mixed with carbon nanotubes (1 percent by mass) ignited with 7.5 joules at an average rate of 750 W for 10 milliseconds. To raise a charge of the same mass of neat HMX to an autoignition temperature of 200 degrees Celsius would require much more energy (about 110 joules) for a longer duration (about 150 milliseconds).



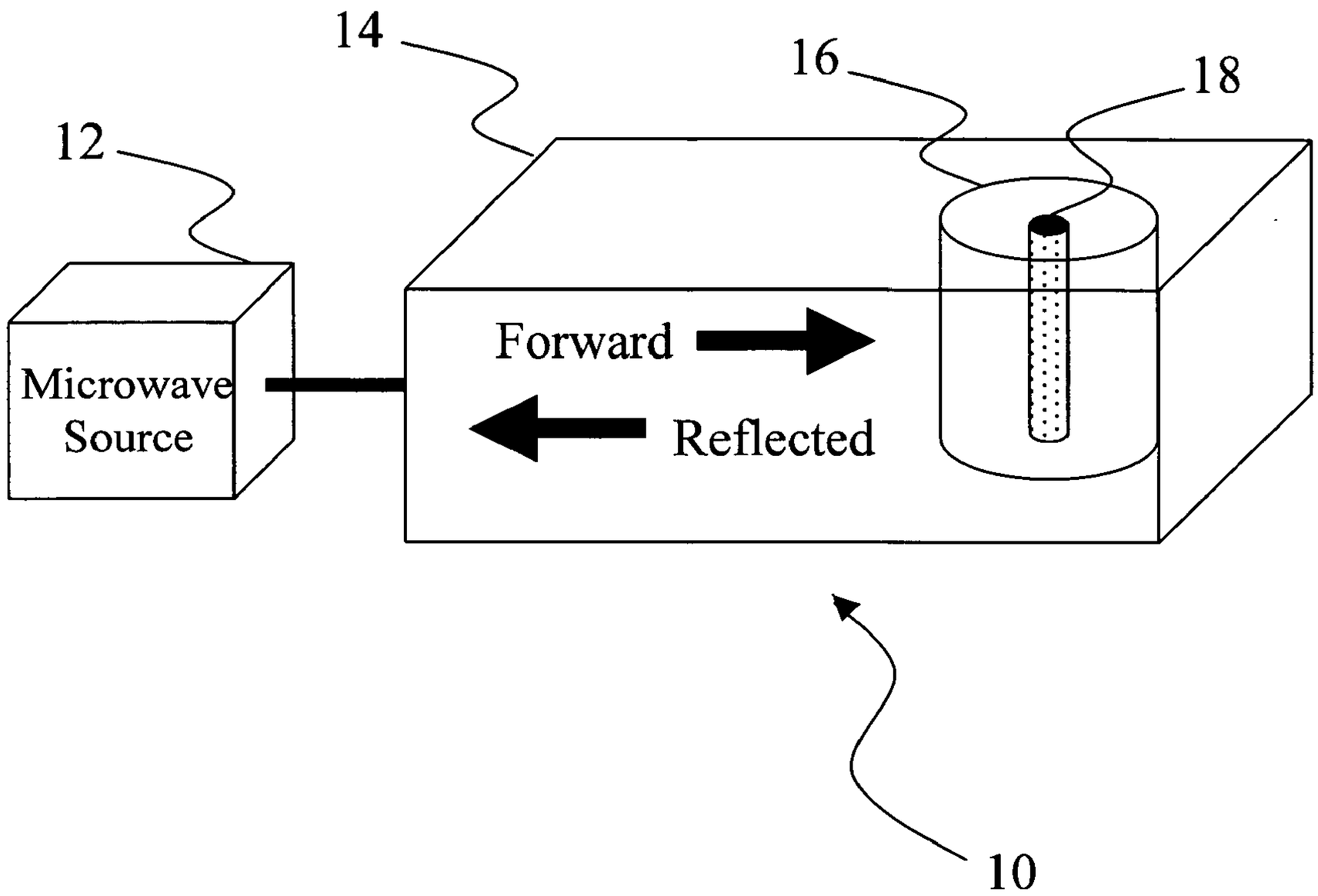


Fig. 1

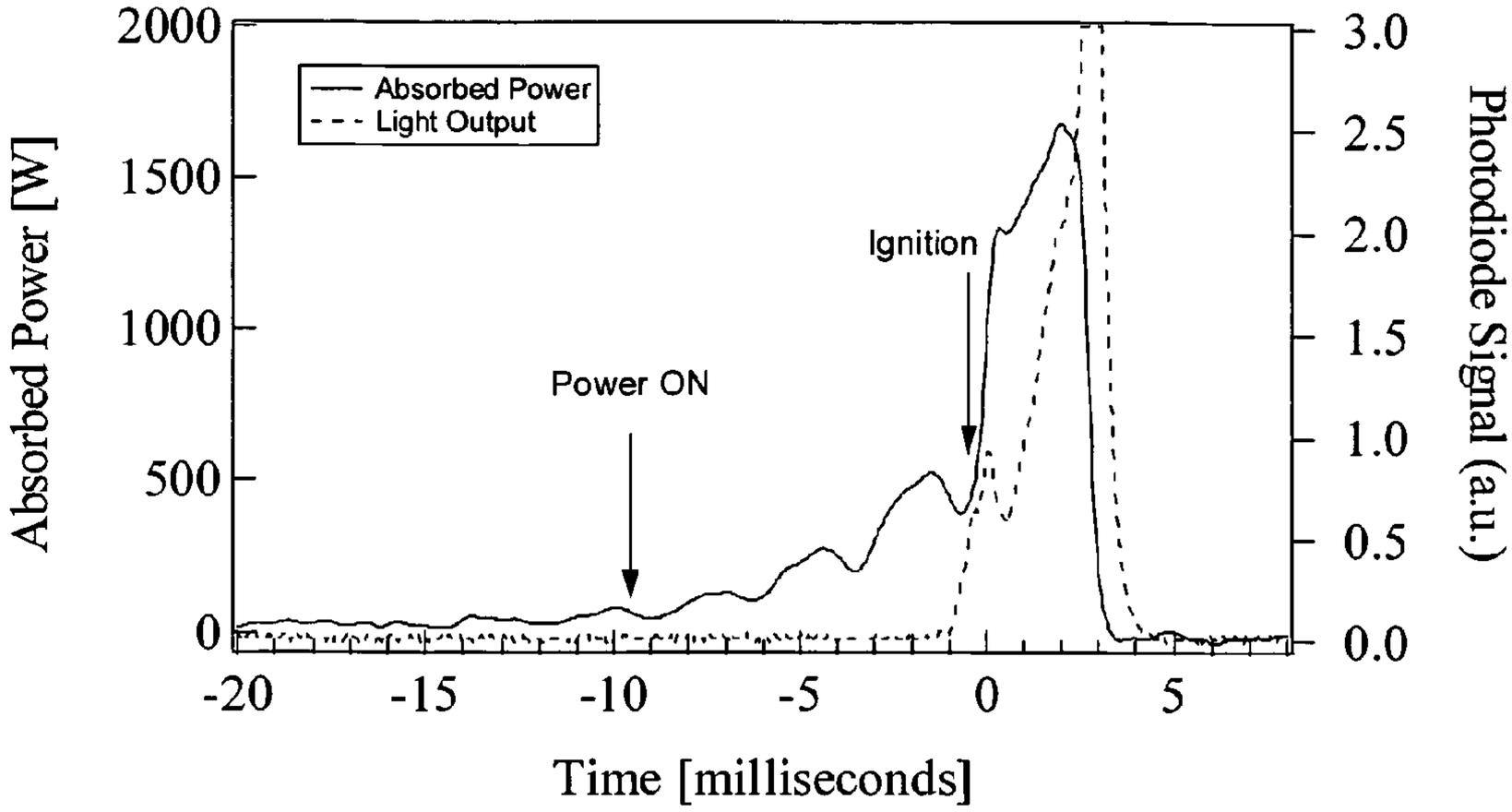

Absorbed Power
Light Output
Ignition
Power ON
Absorbed Power [W]
Photodiode Signal (a.u.)
2000
1500
1000
500
0
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-20
-15
-10
-5
0
5
Time [milliseconds]

Fig. 2

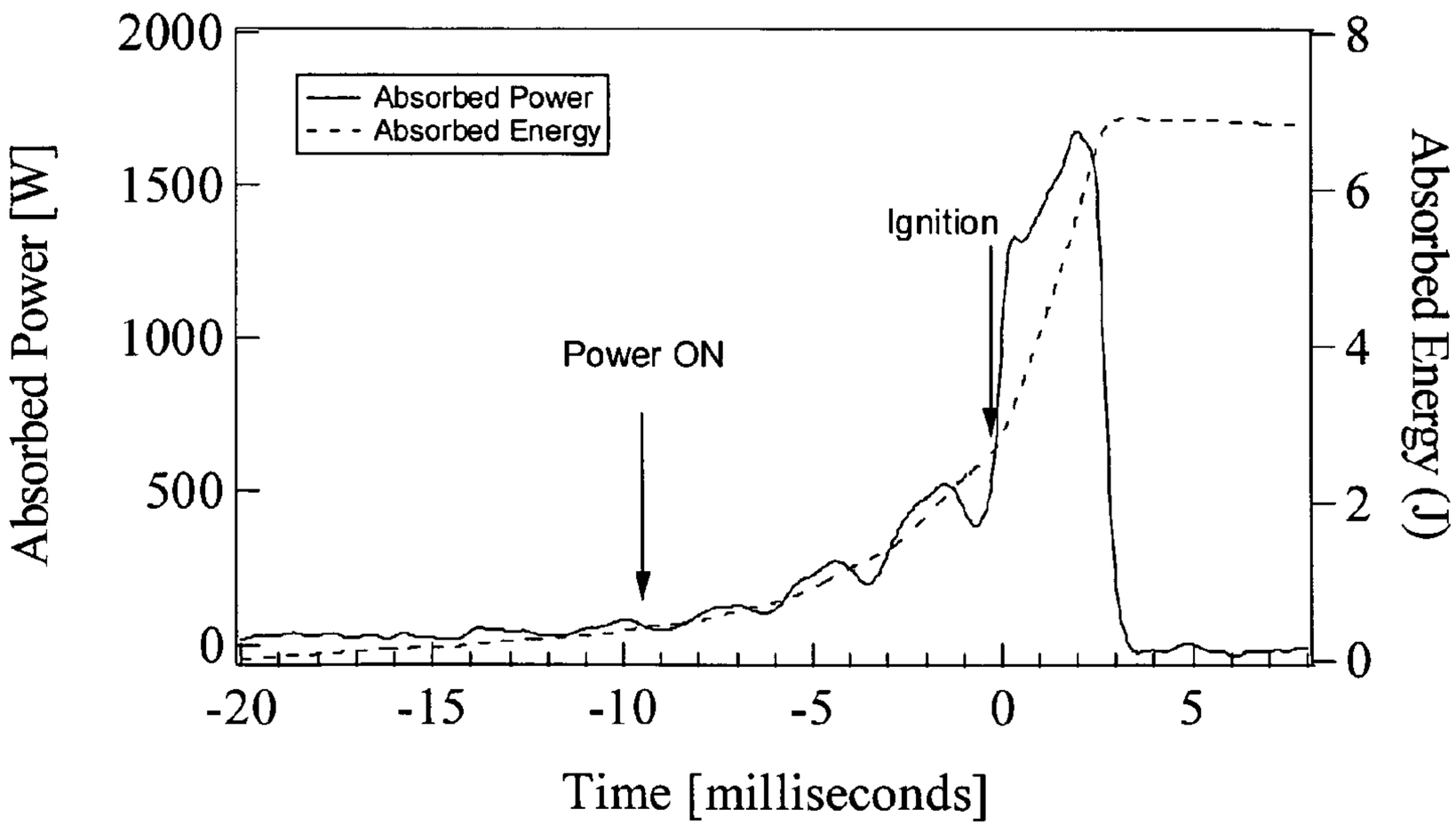

Absorbed Power
Absorbed Energy
Ignition
Power ON
Absorbed Power [W]
Absorbed Energy (J)
2000
1500
1000
500
0
8
6
4
2
0
-20
-15
-10
-5
0
5
Time [milliseconds]

Fig. 3

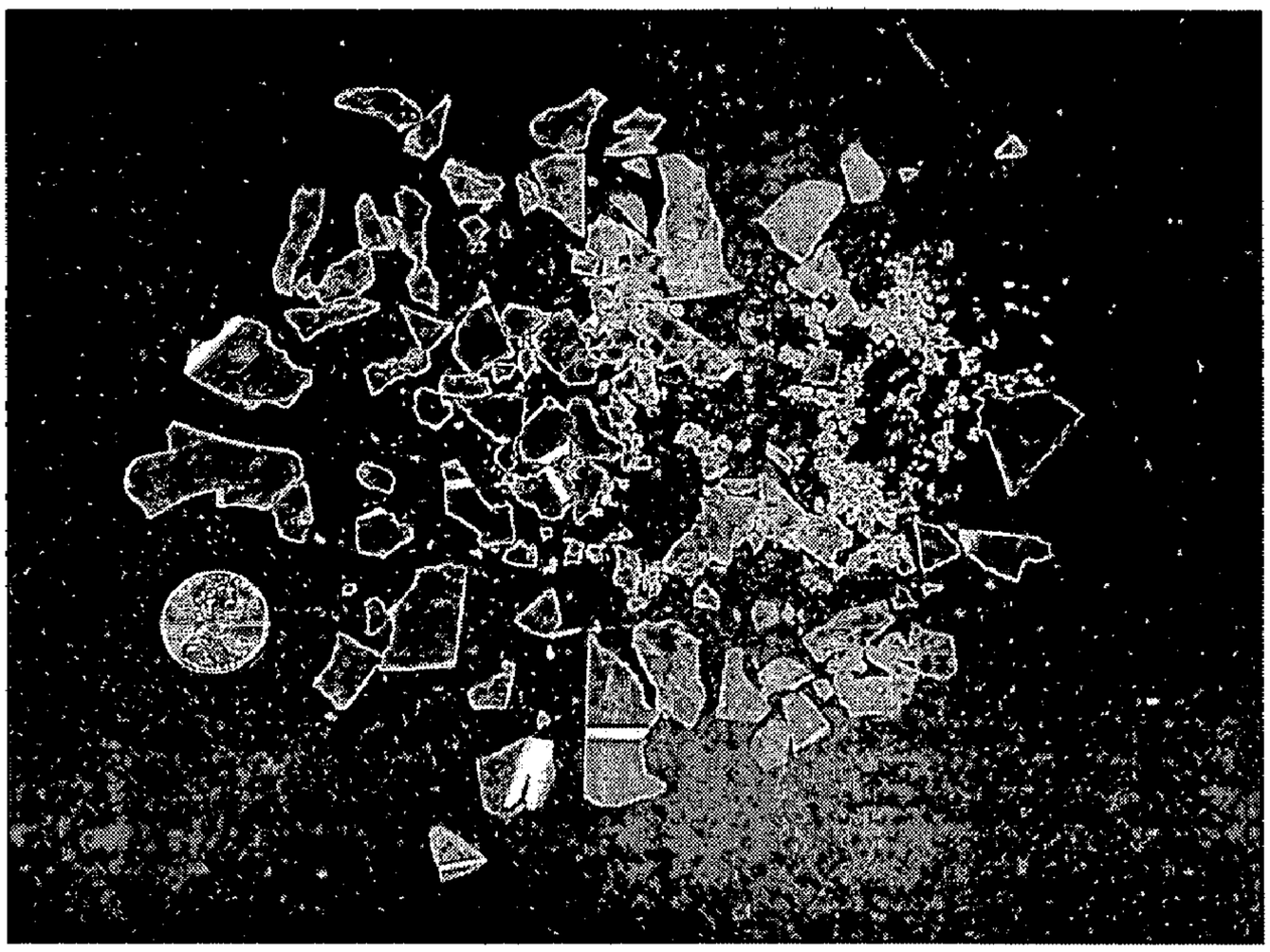


Fig. 4

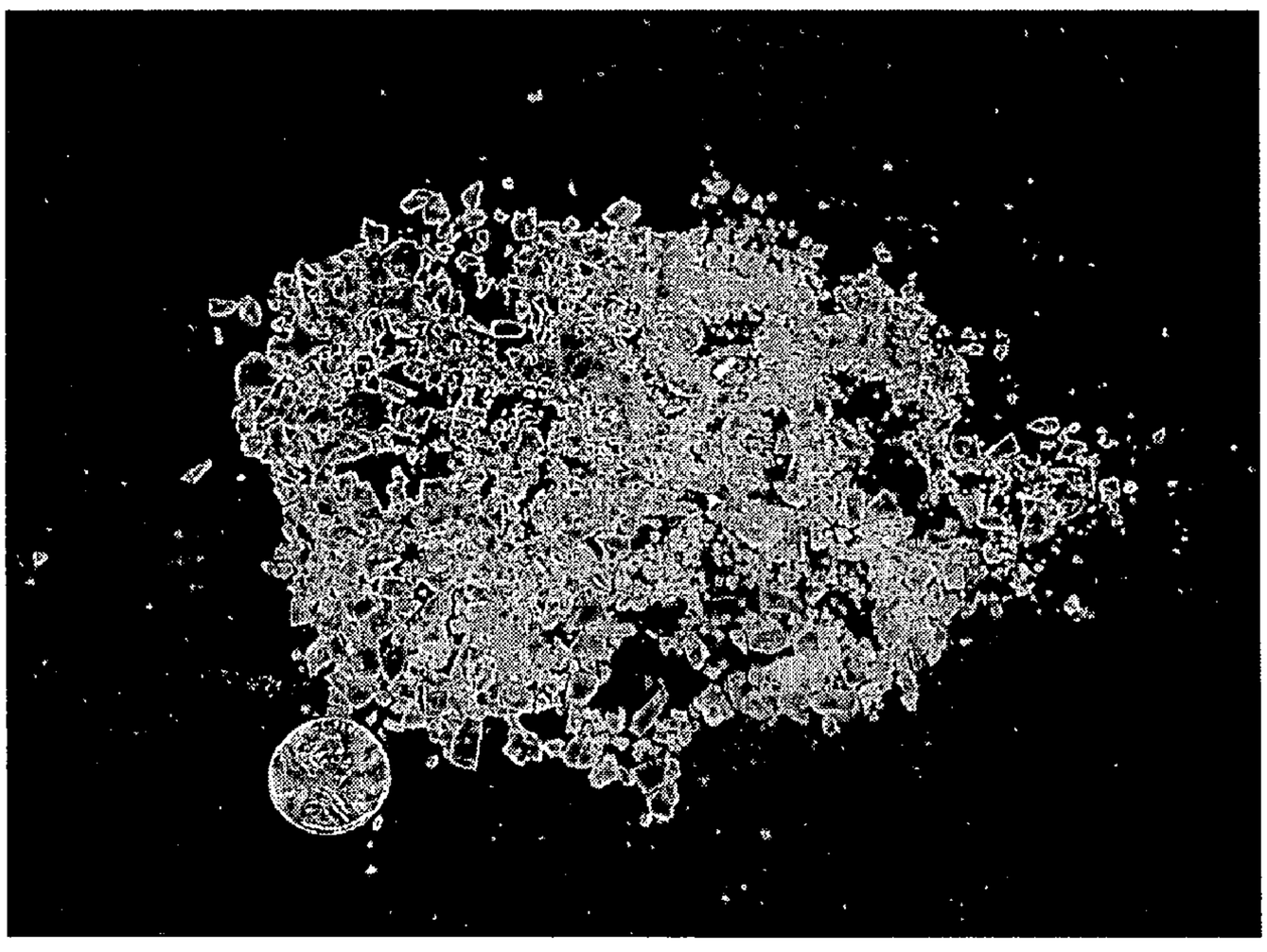


Fig. 5

MICROWAVE HEATING OF ENERGETIC MATERIALS

STATEMENT REGARDING FEDERAL RIGHTS

[0001] This invention was made with government support under Contract No. W-7405-ENG-36 awarded by the U.S. Department of Energy. The government has certain rights in the invention.

FIELD OF THE INVENTION

[0002] The present invention relates generally to devices employing energetic materials (i.e. explosives) and more particularly to microwave heating of a charge of explosive.

BACKGROUND OF THE INVENTION

[0003] Microwave radiation is electromagnetic radiation with a frequency in the range from about 1,000 MHz to about 30,000 MHz. Microwave techniques have been employed for a wide variety of applications that include radio astronomy, long distance communication, navigation, microwave ovens, and the study of physical and chemical properties of matter.

[0004] Recently, the ignition of several important high explosives by microwave irradiation has been reported (see Kazuo Hasue, Masami Tanabe, Nobutune Watanabe, Shoji Nakahara, and Fumiaki Okada, "Initiation of Some Energetic Materials by Microwave Heating," Propellants, Explosives, Pyrotechnics, vol. 15, pp. 181-186 (1990), incorporated by reference herein). Samples of high explosive (PETN, RDX, HMX, and TNT, for example) were confined in tubes and ignited by microwave radiation having a frequency of 2450 MHz±50 MHz. The TABLE below the next paragraph summarizes some of the properties of these explosives.

[0005] The uniformity of heating by microwave radiation is related to the dielectric properties of the material being heated. The absorbed power, which results in microwave dielectric heating, is given by equation (1)

$$P=(5/9)\times f\times E^2\times\epsilon\times\tan\delta\times10^{-10}[W/m^3] \quad (1)$$

[0006] where f is the frequency in Hertz (Hz), ϵ' is the real part of the relative dielectric constant, tan δ is the dielectric loss, and E is the electric field intensity in volts/cm. Values for ϵ', tan δ, and ϵ'' are given in the TABLE for the high explosives PETN, RDX, HMX, and TNT. If the frequency f and the electric field E are constant, then the absorbed power P depends on $\epsilon'\times\tan\delta$, which is equal to ϵ'', which is the imaginary part of the relative dielectric constant; ϵ' and tan δ", however, may change with changes in temperature and frequency.

TABLE

material	ϵ'	tanδ	ϵ''	Melting point (° C.)	Ignition temperature
PETN	2.1	3.0×10^{-3}	6.3×10^{-3}	141.3	200
RDX	2.5	6.7×10^{-4}	1.7×10^{-3}	204.1	200
HMX	2.4	2.9×10^{-5}	7.0×10^{-5}	276–277	241
TNT	2.0	1.2×10^{-4}	2.4×10^{-4}	80.75	330

[0007] The power reflectivity is given by equation (2)

$$|\Gamma|^2=(P_o'/P_o)\times100[\%] \quad (2)$$

where $|\Gamma|^2$ is the power reflectivity, P_o (kW) is the incident power and P_o' (kW) is the reflected power. Hasue et al. calculated an initiation energy, E_i, using equation (3)

$$E_i=(P_o-P_o')\times t[kJ] \quad (3)$$

where t is the initiation delay time in seconds.

[0008] As the TABLE shows, TNT, PETN, RDX, and HMX exhibit low dielectric loss, i.e. they have a small value for tan δ. For these materials, the absorption of microwave radiation was low, indicated by the small value of ϵ''.

[0009] When these energetic materials are exposed to microwave energy, their temperatures increase as they absorb the microwaves until the exothermic reaction takes over and they ignite. It is believed that as microwaves are directed at a location of the explosive charge, the temperature rises exponentially at that location, ignition occurs and the reaction spreads out until it consumes the entire charge. The reaction can range in intensity from a non-violent burning to very violent thermal explosion; this depends largely on the level of confinement of the explosive; an initiated explosive contained in a strong, gas tight vessel will explode violently while in most circumstances, an unconfined explosive will simply burn.

[0010] According to Kazuo Hasue et al. "Initiation of Some Energetic Materials by Microwave Heating," Propellants, Explosives, Pyrotechnics, vol. 15, pp. 181-186 (1990), for all of the explosives tested, a delay in ignition using microwaves was observed. The delays observed are as follows: PETN 71 seconds, RDX 88 seconds, HMX 176 seconds, and TNT 100 seconds. These ignition times are too long for a practical use using microwave ignition.

[0011] If the delay time for microwave ignition for high explosives were significantly reduced, ignition of high explosives using microwave energy may be employed as an initiation mechanism for practical devices. Therefore, there remains a need for reducing the delay time for ignition of high explosives for microwave energy to be useful as an initiation mechanism.

[0012] Therefore, an object of the invention is a method for microwave ignition of high explosives with a reduced delay time.

[0013] Another object of the invention is a high explosive mixture sensitized to ignition by microwave energy.

[0014] Additional objects, advantages and novel features of the invention will be set forth in part in the description which follows, and in part will become apparent to those skilled in the art upon examination of the following or may be learned by practice of the invention. The objects and advantages of the invention may be realized and attained by means of the instrumentalities and combinations particularly pointed out in the appended claims.

SUMMARY OF THE INVENTION

[0015] In accordance with the purposes of the present invention, as embodied and broadly described herein, the present invention includes a method for igniting high explosive. The method includes preparing a mixture of high explosive having a first dielectric loss and a material having a second dielectric loss that is higher than the first dielectric loss of the high explosive, and thereafter exposing the mixture to microwave energy.

[0016] The invention also includes a mixture of high explosive having a first dielectric loss and about 1 percent by mass or less of a material having a second dielectric loss that is higher than the first dielectric loss of the high explosive.

BRIEF DESCRIPTION OF THE DRAWINGS

[0017] The accompanying drawings, which are incorporated in and form a part of the specification, illustrate the embodiment(s) of the present invention and, together with the description, serve to explain the principles of the invention. In the drawings:

[0018] **FIG. 1** shows a schematic representation of an apparatus that was used to demonstrate microwave ignition according to the present invention.

[0019] **FIG. 2** shows a graph of the absorbed power, and intensity of light output, as a function of time for a sample of HMX (0.5 gram) mixed with 1 percent by mass of carbon nanotubes.

[0020] **FIG. 3** shows a graph of the absorbed microwave power, and absorbed energy, as a function of time for the sample of **FIG. 2**.

[0021] **FIG. 4** shows a photomacrograph of fragments generated by microwave initiation of a sample of HMX and carbon nanotubes; and

[0022] **FIG. 5** shows a photo macrograph of fragments generated by microwave initiation of another sample of HMX and carbon nanotubes.

DETAILED DESCRIPTION

[0023] The invention relates to the ignition of explosives using microwave energy. For the purposes of this invention, the terms "energetic material" and "explosive" are interchangeable. One aspect of the invention involves preparing mixtures of high explosive that can be ignited using much less energy than what would is required for the neat high explosive. Another aspect of the invention relates to reducing the delay for ignition. High explosives contemplated with this invention include well-known high explosives such as PETN, RDX, HMX, TNT and TATB, and mixtures of these explosives. These high explosive materials do not absorb microwave radiation readily and do not ignite promptly, as previously described (see K. Hasue et al. in "Initiation of Some Energetic Materials by Microwave Heating," Propellants, Explosives, Pyrotechnics, vol. 15, pp. 181-186 (1990)). They are low loss dielectric materials and require ignition times that, according to K. House, vary from 71 seconds to 176 seconds. For this reason, devices and methods employing microwave ignition of these types of materials are not practical.

[0024] According to the invention, high explosive is mixed with materials that absorb microwave radiation strongly. These mixtures can be ignited using microwave energy, do not require as much microwave energy as the neat explosive does for ignition, and have a much shorter ignition time than the neat explosive does.

[0025] The materials that can be mixed with high explosive to sensitize the explosive to microwave radiation include carbon nanotubes, finely divided metallic particles, and semiconductor particles. These are examples of materials that absorb microwave energy strongly. It is expected that any solid metal could be used. Only a very small percentage of the mixture, one percent by total mass and even less, is needed to sensitize an explosive charge to microwave radiation. Example of metals may be useful with the invention include, but are not limited to, aluminum, iron, and tungsten.

[0026] The preparation of mixtures of high explosive with carbon nanotubes and the ignition of these mixtures using a flash of light has been described in U.S. patent application Publication US2004/0040637. According to the '637 Application, energetic mixtures containing from 3 percent to 20 percent by weight of carbon nanotubes with explosives (ammonium perchlorate, RDX, TNT, and black powder) were prepared and ignited using a light flash. This type of ignition is a surface ignition; the flash promotes ignition on the surface and not throughout the bulk of the material because the light does not penetrate the material. By contrast, the present invention relates to volume ignition using microwaves. The microwaves penetrate the entire volume of the mixture, and microwave radiation is absorbed by carbon nanotubes throughout the mixture. Volume ignition results in rapid consumption of the entire charge, not just ignition and burning from the surface of the mixture.

[0027] The invention was demonstrated by preparing a mixture of HMX and a small amount of carbon nanotubes. Only about 0.1% percent by mass of carbon nanotubes relative to the HMX produced a mixture with an ignition less than one tenth the time required for neat HMX.

[0028] In order to construct a practical explosive device, materials referred to herein as "sensitizers" were mixed with energetic material at low concentrations in order to significantly increase the local dielectric loss. When exposed to microwave energy, the sensitizer creates "hot spots," and ignition occurs a many locations throughout the entire charge of energetic material. Adding the sensitizer to the energetic material also increases the overall dielectric constant.

[0029] Sensitizers of the present invention include, but are not limited to, carbon nanotubes, metallic and semiconductor particles. Lossy liquid, i.e. a liquid having a higher dielectric loss than that of the neat explosive material (lossy liquids such as water and acetone, to name a few) or plastics, can also increase the overall dielectric loss of the mixture.

[0030] It is believed that the sensitizer allows the quasi-uniform absorption of microwaves such that the temperature of the energetic material increases uniformly, and/or that the sensitizer provides many locations in the mixture for localized absorption, with induced ignition at these well-distributed discrete "hot spot" locations. If the absorbing sensitizer causes large thermal gradients in a small region, detonation may occur. In any case, the presence of the sensitizer reduces the ignition delay time to such an extent that microwave ignition in a device employing this type of sensitized charge becomes practical.

[0031] Binders may be used with the invention. Useful binders include organic and/or inorganic materials that have a higher dielectric loss than the dielectric loss of the explosive. Some, or all, of the sensitizer could be the binder.

[0032] This technique allows for a wide range of energy release rates. Traditional detonating explosives release their energy very rapidly, at a steady rate, creating high peak

pressures and a short pressure pulse duration. The characteristic blast from detonating explosives is very effective for destroying some types of structures, but improved coupling occurs for many structures by lowering peak pressures and increasing the duration of the pressure pulse. In practice, it is difficult to accomplish this with a device that has good timing control. The invention described here allows for the rapid release of explosive energy over a wide range of impulse characteristics in a single device.

[**0033**] Another aspect of the invention relates to a system that includes a microwave source capable of producing microwave radiation and any energetic material that is sensitive to microwave ignition. These materials can include classical CHNO explosives, insensitive high explosives, high-nitrogen materials, thermites, metastable intermolecular composites, and the like. The energetic material may have intrinsic high dielectric loss, or may include low dielectric loss explosive in combination with sensitizers such as metallic particles, semiconducting particles, nanoscale and/or microscale fibers, including carbon nanotubes. The sensitizer may be distributed throughout the energetic material uniformly or in such a way such that some desired performance characteristic is achieved.

[**0034**] The energetic material may contain more than one type of sensitizer, where each sensitizer may absorb microwave energy of some particular frequency, or within a particular frequency range, or have a particular microwave power response, such that different frequencies or power levels induce different behaviors.

[**0035**] To facilitate extremely fast energy release rates, the energetic material may be a composite consisting of various energetic materials, some sensitized and some not, again to provide flexibility in performance. An example of this type of material may be a HMX/PETN composite, where the PETN contains the sensitizer (the HMX could be uniformly loaded with 100 micron PETN crystals that are uniformly loaded with 0.1% carbon nanotubes, for example).

[**0036**] The common exploding-wire detonator uses PETN. A particulate mixture of PETN crystals and sensitizer (carbon nanotubes or finely divided metallic or semiconductor particles, for example) would be employed as a microdetonators that could be distributed in a bulk charge of a different explosive, say HMX. The distribution could be uniform, or have characteristics that would lead to some specific, desired performance. Using sensitized PETN in such a way would produce detonation waves from many well-distributed locations throughout the composite.

[**0037**] The following EXAMPLES demonstrate the operability of the invention. The EXAMPLES demonstrate the performance of a mixture of 0.5 g HMX and 1 percent by mass carbon nanotubes when exposed to microwave radiation. For comparison, 0.5 g HMX exposed to the same or higher power microwaves over the same or longer duration did not ignite. The EXAMPLES were performed using a microwave initiation apparatus similar to that described by Kazuo Hasue, Masami Tanabe, Nobutune Watanabe, Shoji Nakahara, and Fumiaki Okada, "Initiation of Some Energetic Materials by Microwave Heating," Propellants, Explosives, Pyrotechnics, vol. 15, pp. 181-186 (1990). The used to demonstrate the invention included a microwave generator, a microwave tuner, a power measurement apparatus and a shorted waveguide that provided a region of standing-wave microwave energy to facilitate the absorption of microwave energy into the energetic material. The energetic material and associated fixtures perturb the impedance of the waveguide, and a three-stub tuner was employed to induce maximum power transfer to the energetic material.

[**0038**] A schematic representation of the apparatus is shown in **FIG. 1**. Apparatus **10** includes microwave generator **12**, which supplied the 2.45 GHz, 6 kWatt microwaves. Microwaves pass from generator **12** through waveguide **14**, also known as the microwave applicator, to cylinder **16** and then to the sample of energetic materials **18**. Cylinder **16** was a 1-inch diameter quartz cylinder that was used to contain the sample of energetic material **18** that was being exposed to the microwaves. Forward and reflected powers are illustrated and were measured using directional couplers. With further regard to sample containment, a Pyrex tube (not shown) having an inner diameter of about 3/16 inch was used to confine the sample. Cylinder **16** surrounded the Pyrex tube, protected the waveguide and other components of the microwave initiation apparatus, and also acted as a "witness" for energy release rate by fragmenting during the explosion event that followed ignition. The term "witness" is a commonly used term that is used herein to describe a passive object that is affected in some way by an explosive event. The witness was used in order to provide some measure of the explosive event. In the EXAMPLE below, the quartz 'witnesses' the explosive event and provides information about it by how it fragments as a result of the explosion.

EXAMPLE 1

[**0039**] Ignition of a mixture of HMX with carbon nanotubes. A mixture of HMX (0.5 g) and carbon nanotubes (1 percent by mass) was prepared and ignited by microwave radiation. **FIG. 2** shows a graph of absorbed power as a function of time (solid line, power in units of Watts, time in units of milliseconds) of the mixture. **FIG. 2** also shows the photodiode signal (i.e. the light output) as a function of time (dashed line) from the mixture, along with the absorbed power. As **FIG. 2** shows, the microwave power was turned on at a time of minus 10 milliseconds, and ignition occurred about 10 milliseconds later.

[**0040**] **FIG. 3** shows a graph of the absorbed microwave power as a function of time (solid line) and a graph of the absorbed energy as a function of time (the dashed line) of the mixture. The absorbed energy is the integral of the absorbed power). The total absorbed energy was 7.5 Joules (J) at an average rate of 750 Watts for a duration of 10 milliseconds. For comparison, raising the same mass of neat HMX to a conservative autoinitiation temperature of 200 degrees Celsius would require about 110 J for a duration of 150 milliseconds. The neat HMX would also require much higher electric field strengths due to a weaker interaction of neat HMX with microwave energy.

EXAMPLE 2

[**0041**] Evidence for variable energy release rate. The size of container fragments generated during an explosive event provides a measure of the energy release rate (see, for example, P. R. Lee, "Hazard Assessment of Explosives and Propellants" in Explosive Effects and Applications, J. A. Zukas and W. P. Walters, Springer-Verlag (New York, 1998)

p. 327, incorporated by reference). For this example, two experiments were performed to examine the fragment size of the pyrex sample container and the quartz containment cylinder. In both experiments, the nominal conditions were the same: incident power, sample size and sample loading were nominally identical. Absorbed power data was not recorded but inconsistencies between the two experiments resulted in a difference in impedance matching such that the absorbed power was different, which is indicated by the difference in sizes of the fragments in **FIG. 4** and **FIG. 5**. **FIG. 5** (experiment 1) shows larger fragments than those of **FIG. 4** (experiment 2). A scale bar was not included in these FIGURES, but the penny provides some indication of the fragment sizes. These qualitative results indicate that the energy release rate was significantly faster in EXAMPLE 1 compared to EXAMPLE 2.

[**0042**] In summary, the present invention relates to igniting energetic materials using microwave radiation. High explosives were rendered more sensitive to microwave heating by the addition of sensitizers. Effective sensitizers have a dielectric loss that ranges from one to several orders of magnitude greater than that of the explosive. The addition of these lossy dielectric materials provides the user with the ability to better control the overall behavior of the subsequent explosive event, i.e. the explosion. The added sensitizers allow tuning of the energy release rate, up to detonation of the explosive. The added sensitizers also allow for prompt ignition of energetic materials.

[**0043**] The foregoing description of the invention has been presented for purposes of illustration and description and is not intended to be exhaustive or to limit the invention to the precise form disclosed, and obviously many modifications and variations are possible in light of the above teaching.

[**0044**] The embodiment(s) were chosen and described in order to best explain the principles of the invention and its practical application to thereby enable others skilled in the art to best utilize the invention in various embodiments and with various modifications as are suited to the particular use contemplated. It is intended that the scope of the invention be defined by the claims appended hereto.

What is claimed is:

1. A method for igniting high explosive comprising preparing a mixture of high explosive having a first dielectric loss and a material having a second dielectric loss that is higher than the first dielectric loss of the high explosive, and thereafter exposing the mixture to microwave energy.

2. The method of claim 1, wherein the explosive is selected from the group consisting of TNT, PETN, RDX, HMX, TATB, and mixtures thereof.

3. The method of claim 1, wherein the material that strongly absorbs microwave energy is selected from the group consisting of carbon nanotubes, metal powder, semiconductor powder, and mixtures thereof.

4. The method of claim 3, wherein the metal powder comprises finely divided powder.

5. The method of claim 3, wherein the semiconductor powder comprises finely divided powder.

6. The method of claim 3, wherein the mixture comprises about 1 percent by mass or less of carbon nanotubes.

7. The method of claim 1, wherein the material that strongly absorbs microwave energy comprises a semiconductor.

8. The method of claim 1, wherein the method further comprises adding a binder to the mixture of high explosive having a first dielectric loss and a material having a second dielectric loss that is higher than the first dielectric loss of the high explosive

9. A mixture of high explosive having a first dielectric loss and about 1 percent by mass or less of a material having a second dielectric loss that is higher than the first dielectric loss of the high explosive.

10. The mixture of claim 9, wherein the high explosive is selected from the group consisting of TNT, PETN, RDX, HMX, TATB, and mixtures thereof.

11. The explosive of claim 9, wherein the material that strongly absorbs microwave energy is selected from the group consisting of carbon nanotubes, metal powder, and semiconductor powder.

12. The explosive of claim 11, wherein the metal powder comprises finely divided powder.

13. The explosive of claim 11, wherein the semiconductor powder comprises finely divided powder.

14. The explosive of claim 9, further comprising a binder.

* * * * *