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Utilizing Microwave Susceptors to Visualize Hot-Spots in Trinitrotoluene

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ABSTRACT

In recent years, researchers have shown that the inclusion of susceptors in monomolecular explosives increases microwave absorption where the monomolecular explosive alone is nearly transparent to the microwave energy. In this study, graphite particles are used to absorb microwave energy and decompose trinitrotoluene 1,3,5 (TNT). Aluminum particles were also used with TNT but very little energy was absorbed. Thermal and electric properties of the susceptor affect microwave energy conversion to thermal energy in explosives. The temperature gradients across the broad face of a cylindrical sample of TNT with and without susceptors were spatially observed using an infrared camera. Hot-spots were observed during 120 seconds exposure to microwave energy at a frequency of 3.3 GHz and found to directly correlate with susceptor concentration. When using graphite, the TNT heated above its melting temperature and decomposed as a vapor.

KEY WORDS: Microwave heating, susceptors, aluminum, graphite, explosives, TNT.

INTRODUCTION

Energetic materials include explosives, propellants and pyrotechnics (and thermites). The interaction of electromagnetic energy with energetic materials is a relatively new area of research. Understanding some key aspects to what has been learned on this topic provides direction for future work. For this reason, a limited review of the influence of susceptors in explosives exposed to microwave energy is presented here, with key conclusions highlighted.

Microwave heating of high explosives was originally reported in a patent that showed mixtures of high explosives with materials that readily absorb microwave energy (i.e., susceptors) ignite more readily than the corresponding neat explosives [Perry et al., 2006].

In fact, most neat explosives are rather transparent to microwave energy [Perry et al., 2006, Hasue et al., 1990]. Specifically, HMX (0.5 g) mixed with carbon nanotubes (1 wt%) ignited with 7.5 J at an average rate of 750 W for 10 ms. To raise the same mass of neat HMX to an autoignition temperature of 473 K requires significantly more energy (about 110 J) for a longer duration (~ 150 ms). They suggested that materials that can be used to sensitize explosives to microwave radiation include: carbon nanotubes, finely divided metallic particles, and semiconductor particles, which are examples of materials that strongly absorb microwave energy [Perry et al., 2006].

Similarly with explosives, propellants have been doped with susceptors to sensitize the composite to microwave energy absorption [Baker and Lee, 2000]. An interesting finding from [Hasue et al., 1990] is that when susceptors were introduced, the energetic material exposed to microwave energy generally melted before initiation.

This study expands our understanding of the interaction of microwave energy by exposing an explosive (i.e., 1,3,5-Trinitrotoluene (TNT)) to microwave energy. A unique microwave exposition chamber designed for energetic studies provided 164-168 W of electromagnetic power at a frequency of 3.3 GHz. Flakes of TNT were pressed into pellets, placed in a transparent microwave holder and positioned in the wave guide. In addition to neat TNT, samples were also prepared with 10 wt% susceptors that included either micron scale aluminum powder or micron scale graphite particles. The weak energy fields induced on the sample were monitored and thermal energy build up was visually recorded with the IR camera.

EXPERIMENTAL PROCEDURE

Flakes of trinitrotoluene (TNT) were uniaxially pressed, either with or without the susceptor additives, into cylindrical pellets with a diameter of 1.27 cm (Table I). Specifics of the general pressing procedure can be found in [Pantoya et al., 2009]. The mass of the pressed pellets and physical dimensions are also shown in Table I. Caution is advised when working with explosives and explosive safety experts were consulted prior to the pressing procedure to ensure safety.

Table I. Physical properties for the three types of samples evaluated in this study. Bulk density is reported in terms of %TMD (i.e., percent theoretical maximum density).

Material	Mass of TNT	Thickness	Mass of Additive	% TMD
TNT	345.8 mg	1.77 mm	0.00 mg	93.2%
TNT w/Al	320.8 mg	1.65 mm	33.3 mg	91.0%
TNT w/ Graphite	305.3 mg	1.63 mm	34.8 mg	88.6 %

While previous explosive studies reportedly used 1 wt% susceptor [Perry et al., 2006], prior work with thermite powders showed 10 wt% susceptor enabled heightened microwave energy absorption using the same microwave frequency and power as described here [Crane et al., 2014]. For this reason, susceptors were added at 10 wt% of the TNT. The susceptors were dry mixed with the TNT flakes and the dispersion quality was poor with significant susceptor concentration gradients. Two susceptors were examined, aluminum powder and graphite powder. The aluminum powder was procured from Alfa Aesar (Item No. 41000, Lot No. H12X017) with an average particle size of 3.3 μm. Flake graphite powder was procured from Alfa Aesar (Item No. 43209, Lot No. F01X009) has a particle size nominally less than 44 microns with a median particle size of 9.5 μm. Photos illustrating the final samples are shown in the results section.

A detailed description of the Electromagnetic Exposure Chamber (EMEC) can be found elsewhere [Crane et al., 2013], but is summarized here and shown for illustration in Figure 1. The samples were placed into a polytetrafluoroethylene (PTFE) sample holder and positioned inside a WR229 waveguide.

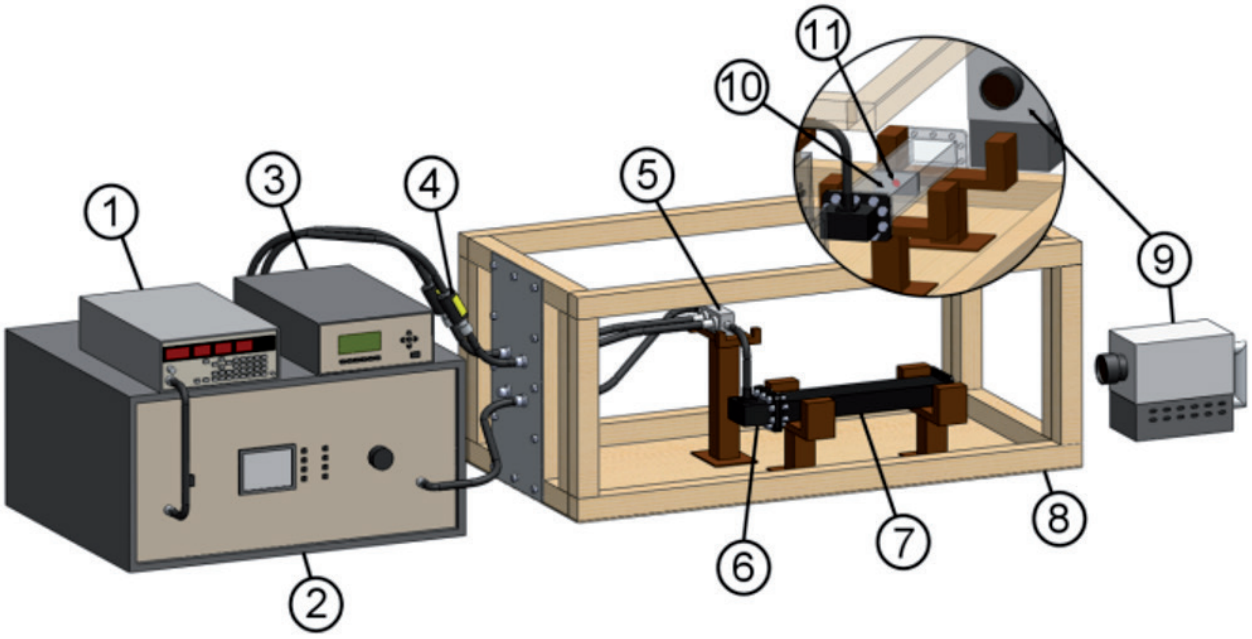

Figure 1. The Electromagnetic Exposure Chamber (EMEC) consists of a 1) signal generator, 2) microwave amplifier, 3) power meter, 4) power sensors, 5) dual-directional coupler, 6) coaxial to waveguide adapter, 7) waveguide, 8) faraday cage (aluminum meshing not shown), 9) Infrared camera, 10) PTFE sample holder, and 11) sample.

The sample holder places the radial face of the sample perpendicular to the axial direction of electromagnetic propagation. The microwave amplifier was set to 100% gain, with a measured power output varying between 164 to 168 W of electromagnetic power at a frequency of 3.3 GHz.

The design of the EMEC allows for unobstructed observation of the broad face of the cylindrical sample using a FLIR Indigo Phoenix 9803 infrared camera. The IR camera was calibrated for measuring temperatures in the range of 15 to 250 ºC using an Omega Engineering (Model No. BB701) blackbody calibration source and linear extrapolation between 150 to 250 ºC. The emissivity was estimated by taking ambient radiance measurements of the samples and comparing them to a reference sample with a known emissivity in the same measurement window. Since both samples are at the same equilibrium room temperature, the emissivity of the unknown sample can be determined using the camera software. The emissivity of the samples at room temperature (22.8 ºC) was approximately 0.95, and was assumed to be constant as for the narrow temperature ranges investigated.

RESULTS

The three samples shown in Table I were exposed to microwave energy for 120 seconds and the results for the maximum measured temperature are shown in Figure 2.

Figure 3 shows photographs of the samples prior to microwave exposure while Figure 4 shows the same samples during microwave exposure. All sample images are oriented in the same direction, such that higher temperatures observed in Figure 4 correspond to regions of more sparsely concentrated susceptors as seen in Figure 3. Figures 4a and 4b correspond to 120 seconds but Figure 4c was captured prior to 120 seconds. Transient temperature profiles for corresponding to the samples in Figures 4a and b show steady state temperatures have been reached. Figures 5a-c show the TNT sample with graphite at various times of microwave exposure up to the point where the sample melts. Figure 5c shows the hot melt left on the edges of the sample holder

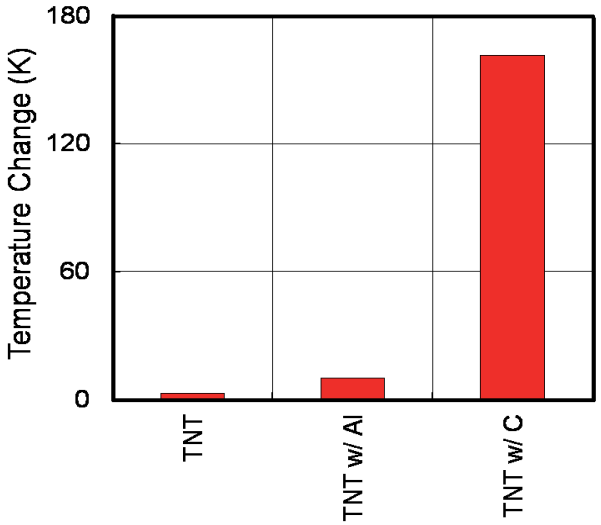

Figure 2. Maximum measured temperature on the back surface of the sample due to microwave heating up to 120 seconds. The TNT/graphite sample ignited prior to 120 seconds of microwave exposure, but did not detonate. The measurement shown here corresponds to a time just prior to ignition when the pellet was intact.

and a drop in temperature in the middle of the sample holder where the TNT/graphite was located prior to melting. Figures 6a and b show the aftermath of the melted TNT/graphite sample.

DISCUSSION

An interesting observation in this study is that the susceptors do not appear to manifest the absorbed microwave energy as thermal energy because the regions of high susceptor concentration do not show significant thermal buildup as seen from the IR imaging. Instead, regions of sparsely concentrated susceptors correspond to hot spot build up. The explanation for this observation lies in the fact that the penetration depth of the electromagnetic waves into the sample is dependent on the sample's electrical conductivity. The higher the conductivity, the smaller is the penetration depth. For highly conductive materials, this depth is very small (a few microns). Most of the incident microwave energy is reflected back rather than getting dissipated as heat in the sample, that is why bulk conductor materials, like aluminum, do

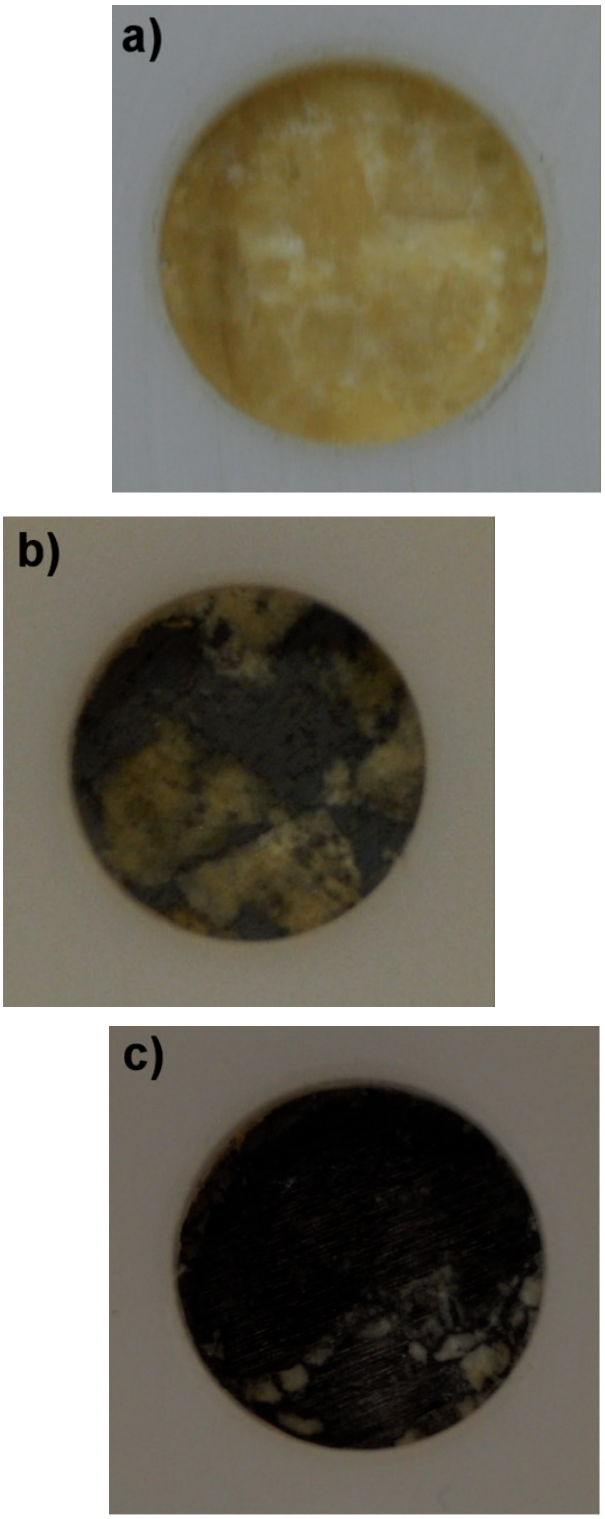

Figure 3. Photographs of a) TNT, b) TNT with aluminum, and c) TNT with graphite. TNT appears as the lighter yellowish orange areas, aluminum is the darker grey, and graphite is the darker black in the image to the right.

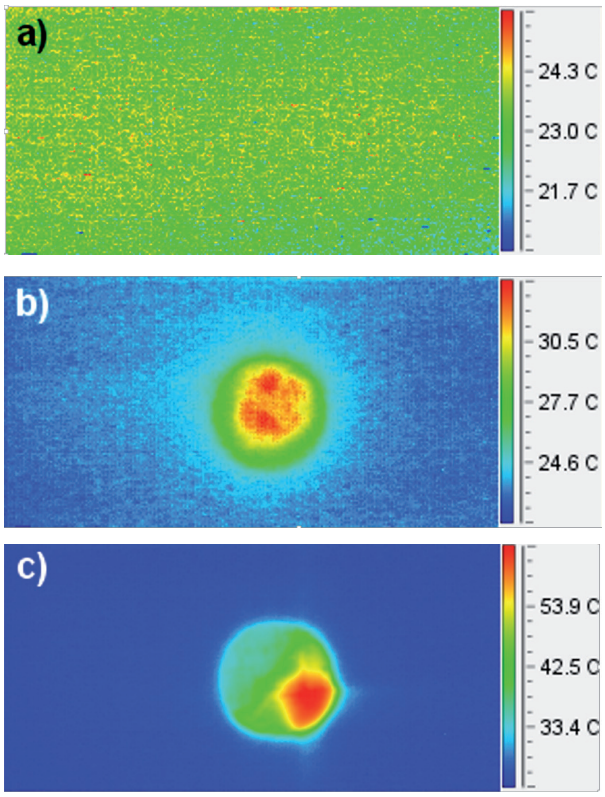

Figure 4. Infrared images of a) TNT and b) TNT with aluminum when microwaved at 3.3 GHz for 120 seconds, and c) TNT with graphite, igniting prior to the 120 second mark.

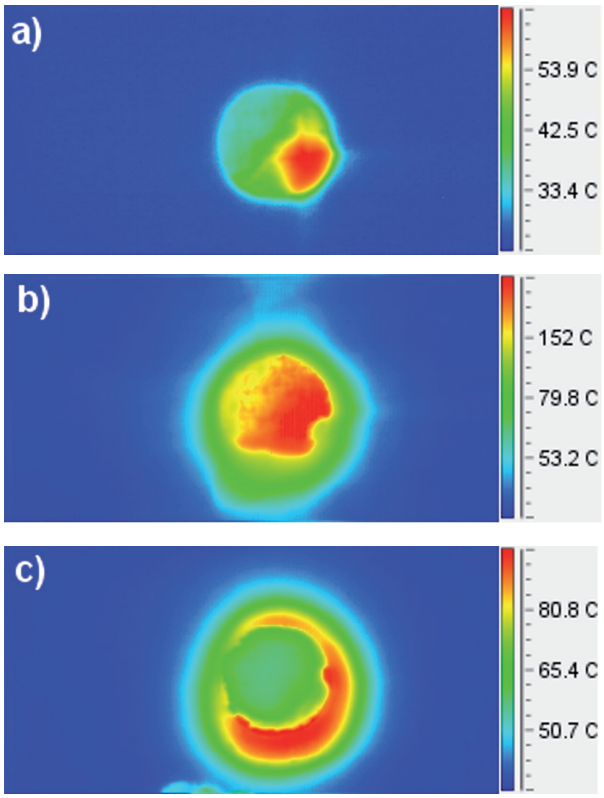

Figure 5. A series of sequential infrared images taken of the TNT/graphite sample at the onset of sample melting and phase change, all prior to 120 seconds. Hot spots correspond to vapor phase just above the sample surface.

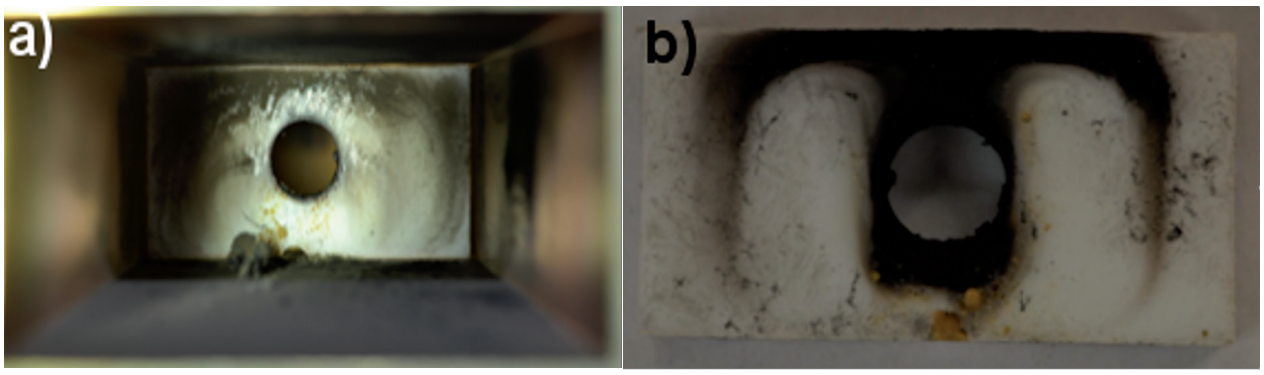

Figure 6. Aftermath of microwave heating the TNT/graphite sample before the 120 second mark for data acquisition.

not exhibit significant heating when exposed to microwaves. Regions of high susceptor concentrations have higher effective electrical conductivities than regions where the susceptors are well dispersed. Consequently, electromagnetic waves in areas of higher susceptor concentrations do not penetrate the sample as deep as areas with sparse concentrations, and therefore, absorb less microwave energy resulting in lower temperatures. On the other hand, having very low well dispersed concentration of susceptor material will cause the electromagnetic waves to pass

through the sample without losing much of its energy, producing less heating. An optimal concentration of well dispersed susceptor materials will produce the most heating response.

To understand the coupled effects of thermal and electrical properties on microwave heating, COMSOL 4.3 Multiphysics FEA software was used to model the temperature distribution in a representative heterogeneous material simulating the susceptor loaded samples. Property values used in the simulations are shown in Table II. Figure 7 shows the model and identifies which sections are modeled as either TNT or graphite.

This parametric study examines limiting cases associated with elevated electrical vs thermal properties. As a baseline, the reported values (i.e., Table II) were used in the simulation shown in Figure 8a. Heating is concentrated in regions where the graphite susceptor is located. In Figure 8b, the thermal properties of graphite were used for both materials and the electrical properties differed as shown in Table II. In this simulation, heating is more uniform. In Figure 8c, the electrical properties of graphite were used for both materials and the thermal properties were representative of the constituents. Heating is concentrated in regions where the TNT is located and correlates well with the heating observed in Figures 3c and 4c. Finally, in Figure 8d, the properties were returned to their original values, but the electrical conductivity of TNT was increased nine orders of magnitude to 1.115 S/m. The heightened electrical conductivity of the TNT region simulates TNT with low concentrations of graphite. As in Figure 8c, heating is concentrated in regions of TNT. These simulations illustrate the coupling of thermal and electrical properties. The most representative simulation of the experimental observation is Figure 8d and shows that increasing the electrical conductivity of the TNT region without altering its thermal properties significantly influences localized heating within that region and corresponds with experimental observations of regions where TNT is sparsely concentrated with graphite. Experimentally this was achieved in regions of lower concentrations of graphite susceptors that demonstrated hot spots.

When examining the experimental results, the TNT/graphite sample exceeds the melting temperature of TNT (80 °C) and boils as indicated by the vapor above the sample as seen in the second image of Figure 5. Though the temperature was not measured directly, this evidence would

Table II. Approximate thermal properties for trinitrotoluene, aluminum, and graphite. (a) properties from [Gibbs and Popolato,1980]; (b) [Crane et al., 2013; Gibbs and Popolato,1980]; (c) real and imaginary parts of permittivity [26]; (d) real and imaginary parts of permittivity [Hotta et al., 2011].

	TNT [a]	Aluminium [b]	Graphite [b]
Thermal Conductivity [$kW \cdot m^{-1} \cdot K^{-1}$]	0.000246	0.210	0.024
Specific Heat at Constant Presure [$kJ \cdot kg^{-1} \cdot K^{-1}$]	1.27	0.900	0.708
Density [$Kg \cdot m^{-3}$]	1590	2699	2250
Thermal Diffusivity [$m^2 \cdot s^{-1}$]	1.22×10^{-7}	8.646×10^{-5}	1.507×10^{-5}
Electrical Conductivity [$S \cdot m^{-1}$]	1.115×10^{-9}	-	1.667×10^{4}
Relative Permittivity	$2.0\text{-}j*2.4 \times 10^{-4}$ [c]	-	$22.5\text{-}j*12.5$ [d]

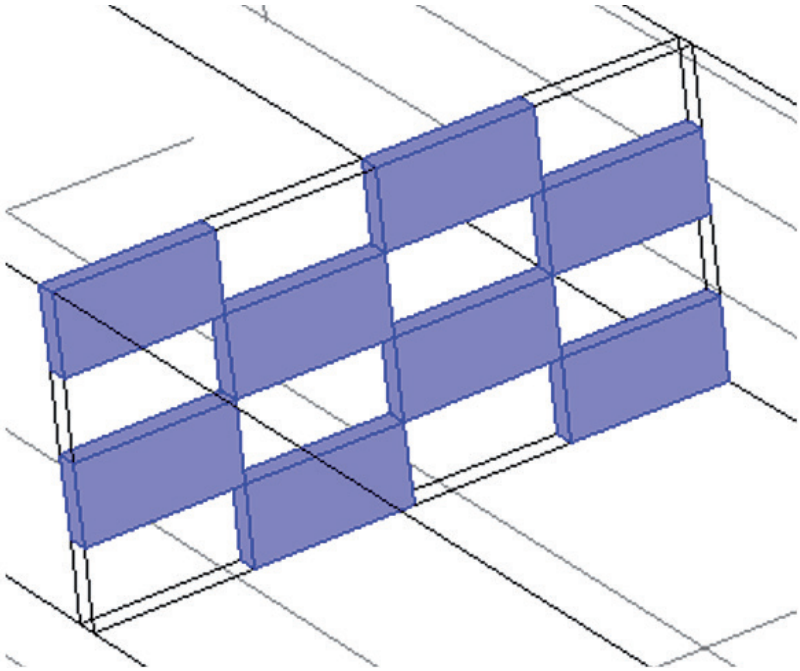

Figure 7. The darker blue sections highlighted in the model represent TNT while the remaining sections represent graphite. The sample was 2 mm thick and spanned the cross-sectional area of the WR229 waveguide.

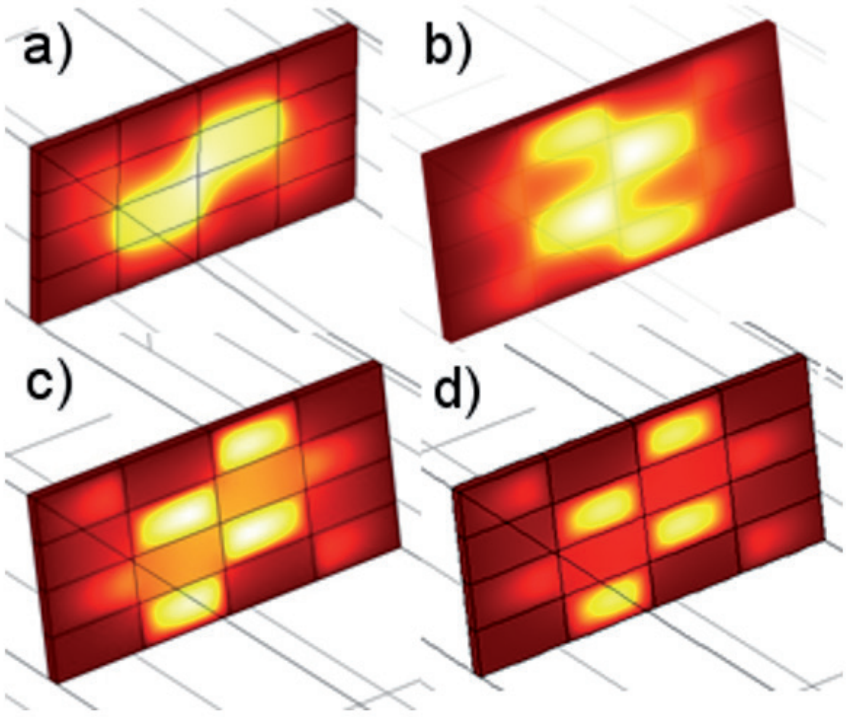

Figure 8. Analysis of microwave heating for 120 seconds at a frequency of 3.3 GHz for a baseline TNT/graphite sample. (a) Baseline simulation using properties from Table II; (b) Thermal properties are equivalent, electrical properties from Table II; (c) Electrical properties are equivalent, thermal properties from Table II; and, (d) All properties from Table II, except graphite electrical conductivity, increased by nine orders of magnitude.

indicate a temperature around the boiling (decomposition) of TNT in the range of 240 °C. Figures 6 a and b also show evidence of melting and decomposition of the TNT. Carbon soot can be seen on both the waveguide and the sample holder. The pattern of the carbon soot on the sample holder indicates a convective flow transported the soot through the waveguide also evidence of the vapor generated during heating and a temperature gradient in the wave guide inducing flow. It is also noted that the melted samples did not jeopardize the integrity of the waveguide or the sample holder. The TNT residue can also be seen as yellowish orange accumulations near the bottom of the sample holder in Figure 6. This is consistent with results from Hasue et al. [1990] that also observed TNT melting and then boiling with exposure to microwave energy.

CONCLUSION

Samples of TNT and TNT with susceptors of aluminum or graphite were exposed to weak microwave energy fields with power output varying between 164 to 168 watts at a frequency of 3.3 GHz for up to 120 seconds. Aluminum particles did not work well absorbing electromagnetic energy and showed very little temperature increase. Graphite showed significant microwave coupling, thermal response and hot spot development. The addition of susceptors to neat TNT flakes showed hot spot development in locations sparsely concentrated with susceptors and less temperature rise in regions of highly concentrated susceptors. These results were modeled using COMSOL to show that increasing the electrical conductivity of the TNT without significantly altering the thermal properties influences localized heating and best simulates the experimental results. The ability to spatially visualize the thermal distribution on the surface of an energetic material is a new approach for understanding the thermal energy evolution in-situ to exposure to microwave energy. These results suggest there is an optimum dispersion and concentration of susceptor that most effectively couples microwave energy.

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