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The effects of particle size on microwave heating of metal and metal oxide powders

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ABSTRACT

To understand the effect of particle size on microwave absorption, it is important to separate absorption in good bulk conductors (like aluminum or copper) from dielectrics, like iron oxide (Fe_2O_3). This study experimentally examined coupling microwaves to powder compacts of discretely different materials such as aluminum and iron oxide as a function of particle size. An electromagnetic chamber exposed compacted powder samples of each material to microwaves at a frequency of 3.3 GHz and in-situ 2-D spatial temperature measurements of the sample surface were captured to quantify microwave heating. Results show that for the non-conductive oxidizer (Fe_2O_3), decreasing the particle size increased the microwave absorption because of the increase in effective surface area and effective conductivity. In contrast, decreasing the conductive metal (Al) particle size resulted in decreased microwave absorption because the ratio of particle size to the skin depth was shown to be a critical parameter controlling energy absorption. This research contributes to new understandings of how microwave energy interacts with metal and metal oxide compacted pellets as a function of particle size.

1. Introduction

Industrial microwave heating of different types of materials has been thoroughly studied with an emphasis on dielectric materials [1]. Microwave heating is dependent on many factors like materials properties, physical characteristics, boundary conditions, and the types of fields the material is being exposed [2]. Dependent on the electrical/magnetic properties, the electromagnetic energy is converted to heat for most materials via conductive, magnetic, and dielectric losses when exposed to electromagnetic radiation [3]. Using microwaves in heating powders has recently become a focus in sintering applications where it was noticed that metal powders do a greater job of absorbing microwave power than their bulk counterparts which reflect microwaves. Walkiewicz et al. studied different metals heated at different rates when exposed to microwave radiation and showed that powered metals coupled well with microwaves, better than some dielectric metal oxides [4].

In using microwaves to heat energetic materials, research has been conducted on microwave ignition of monomolecular explosives with mixed results [5]. Recently, Batanov et al. [6] and Meir et al. [7] used microwaves to ignite thermite compositions. Batanov et al. ignited compacts of titanium–boron and aluminum–iron (III) oxide reaction using 75 GHz microwave beams that ignited the sample via microdischarges on the sample's surface. Meir et al. [7] ignited micron sized aluminum–iron oxide powder mixtures using a microwave drill they developed which consisted of a coaxial waveguide and electrode. In Meir's study [7], hot spots were developed locally near the electrode where thermal runaway lead to combustion of the composite. The potential was shown that microwave heating of energetic composites could be developed further as a means to provide rapid heating rates [1,3] and provide volumetric heating [8].

The goal of this study is to resolve the unique interactions between microwave energy and individual reactants typical of composite energetic materials that represent good bulk conductors (such as aluminum) and dielectrics (such as iron oxide) as a function of particle size. This is accomplished by microwave heating two different sizes of the fuel (aluminum powder) and oxidizer (iron (III) oxide powder), separately, for 60 seconds. Results from this study will broaden understanding on how particle size and aluminum's passivation shell affect heat generation and the parameters to consider when coupling microwaves with energetic composites for future ignition studies.

2. Materials and methods

Aluminum (Al) and iron (III) oxide (Fe_2O_3) powders were pressed individually as separate cylindrical pellets measuring approximately 12.6 mm in diameter with less than a 1.7 mm thickness. Two different regimes of particle sizes were studied, one set having Al and Fe_2O_3 average particle size in the micron range, and one set having Al and Fe_2O_3 average particle size in the submicron range. The powders were

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weighed and uniaxially cold pressed using a carbon steel piston and die to relative bulk densities ranging from 50 to 73% of the theoretical maximum density. The thickness was kept thin (i.e. ~2 mm) so that surface measurements of temperature would be better representative for the entire sample. The iron (III) oxide powder did not adhere well as a pellet at these relative densities therefore a polytetrafluoroethylene (PTFE) binder was added with a mass of 10% of that of the iron (III) oxide powder. Information and approximate physical characteristics of the samples used in this study such as purity, spherical particle size, bulk electrical conductivity (σ), the dielectric constant (ε_r'), the dielectric loss factor (ε_r''), the magnetic permeability (μ_r'), the bulk thermal conductivity (k), the specific heat capacity at constant pressure (Cp), the particle density including impurities (ρ), the pellet mass (m), the pellet diameter (d), the pellet thickness (t), and the relative density of the pellet (ρ_r) are detailed in Table 1.

The electromagnetic exposure chamber (Fig. 1) developed at Texas Tech University was used to evaluate the 2-D spatial temperature distribution on the back surface of a thin cylindrical sample. Microwaves produced from the microwave amplifier are transmitted to a waveguide. The waveguide acts as a conduit for the electromagnetic radiation where the samples were exposed. A sample was placed inside of a PTFE sample holder which was positioned 12.22 cm from the end of a waveguide where both the electric and magnetic fields were present. Fig. 1 shows the sample relative to the sample holder (10) and the placement of the sample inside the waveguide (7) and (9) housed in the electromagnetic exposure chamber.

A brass WR229 waveguide was used in these experiments. The signal generator was set to 3.3 GHz which is the lowest recommended frequency of the waveguide. The gain of the amplifier was set to 100% which equated to approximately 166 W of forward power with an empty sample holder in the waveguide. The end of the waveguide was located 15.24 cm from the aluminum mesh siding of the Faraday cage. The forward and reverse power was monitored at the amplifier and at a directional coupler prior to entering the waveguide.

To monitor in-situ temperatures on the surface of the sample, an infrared camera was used to make real-time spatial measurements. A FLIR Indigo Phoenix 9803 infrared camera was aligned to view axially down the waveguide towards the sample and sample holder within a 256 by 128 pixel window size and an integration time of 0.5 ms. The infrared camera operates in the mid-wavelength region spanning a band of wavelengths from 3 to 5 μm. The surface emissivity of the samples was estimated at 0.95 by comparing the ambient temperatures of the samples to a known reference sample at the same temperature.

The infrared camera was calibrated at eight different temperatures spanning from 288.15 to 393.15 K using an infrared blackbody. The radiance emitted was calibrated against the expected thermal radiation according to Planck's Law using the RTools software package that accompanies the infrared camera. The software uses curve fits to convert the raw camera counts (C_{ad}) to a radiance (R, $W \cdot sr^{-1} \cdot cm^{-2}$), and then uses a 6th order curve fit of Plank's equation to convert radiance to temperature (T, °C) at a set emissivity. To mimic the conditions of the experimental measurements, the calibration procedure included an aluminum mesh with the same distance from the blackbody emitter as it would be from the cylindrical sample. The infrared camera's distance to the mesh in both the calibration and experimental setup was also kept constant. A tabulation of the coefficients ($C_{\#}$) for the curve-fits is shown in Table 2 and equations using these coefficients for calculating the thermal radiance and temperature are shown in Eqs. (1) and (2).

$$R(C_{AD}) = C_1 C_{AD} + C_0 \tag{1}$$

$$T(R) = C_6 R^6 + C_5 R^5 + C_4 R^4 + C_3 R^3 + C_2 R^2 + C_1 R^1 + C_0 \tag{2}$$

Measurements were taken prior to heating (ambient conditions) and at 60 s of microwave exposure to determine the temperature increase of the samples over this time span. Each sample of Al and Fe_2O_3 was heated once and new samples were used to examine the reproducibility of the results.

3. Results

Table 3 details the forward (P_f) and reverse power (P_r) measurements at the amplifier (subscript *amp*) and the dual directional coupler (subscript *ddc*) when the infrared measurements were recorded. Also included in Table 3 is the amount of time between triggering the microwaves and triggering the infrared camera. Fig. 2 plots the temperature change for each sample at 60 s of 3.3 GHz microwave exposure.

4. Discussion

The results in Table 3 show that the forward power from the amplifier is nearly the same for every test and the reverse power is nearly the same for the different sized fuels and oxidizers but differs between the fuel and the oxidizer. This result follows the knowledge of microwave interaction with solid metals [3] where a percentage of microwave energy is reflected because of a short penetration distance. Penetration depth, or skin depth, describes the distance from the surface exposed to electromagnetic waves and to the point where the electric field strength

Table 1
Representative thermal, electrical, and physical properties of the reactants exposed to microwaves at room temperature and 2.45 GHz.

	Submicron Al	Micron Al	Submicron Fe_2O_3	Micron Fe_2O_3	PTFE	Aluminum oxide
Manufacturer	Nanotechnologies	Alfa Aesar	Sigma Aldrich	Alfa Aesar	Sigma Aldrich	
Purity	81%	99.8%	100%	99%	100%	…
Average particle size	120 nm	3.3 μm	28 nm	<45 μm	35 μm	…
σ(S/m)	$3.75 * 10^7$	$3.75 * 10^7$	$1.00 * 10^{-7}$	$1.00 * 10^{-7}$	$1.00 * 10^{-16}$	$1.00 * 10^{-14}$
ε_r'	1	1	~8[a]	~8[a]	2.1	9.6
ε_r''	0	0	0.012[a]	0.012[a]	0.00021	0.0006
μ_r'	~1.00	~1.00	~1[a]	~1[a]	1.00	~1.00
k(W/m·K)	237	237	20	20	0.25	36
Cp(J/kg·K)	900	900	650	650	1200	765
ρ(kg/m^3)	2941	2703	4818	4935	2160	3970
m(mg)	289.4	304.1	554.3 (10% mass PTFE)	530.0 (10% mass PTFE)	…	…
d(mm)	12.60	12.66	12.62	12.62	…	…
t(mm)	1.18	1.49	1.60	1.67	…	…
ρ_r	72.8%	55.1%	56.1%	52.6%	…	…

[a] Electrical properties measured [9] for Fe_2O_3 compressed samples of a relative density of 53.2% at a frequency of 2.45 GHz.